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A denitrogenative palladium-catalyzed cascade for regioselective synthesis of fluorenes

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Supporting Information

Table of Content

1. General considerations

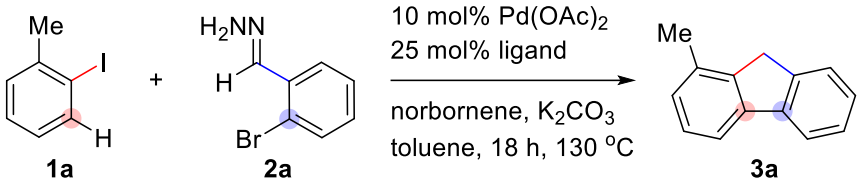
Unless otherwise noted, all reagents were purchased from commercial suppliers and used without purification. All arylation reactions were performed in resealable screw-capped Schlenk tube (approx. 20 mL volume) in the presence of Teflon-coated magnetic stirrer bar (4 mm × 10 mm). 1,4-Dioxane was distilled from sodium benzophenone ketyl under nitrogen. DPPF, $PCyPh_2$, K_2CO_3, Na_2CO_3 and Cs_2CO_3 were purchased from Aldrich. XPhos, cataCXium A, PPh_2-DavePhos and $Pd(OAc)_2$ were purchased from Strem. PPh_3, P(4-OMePh)$_3$ and PCy_3 were purchased from Acros. Hydrazones were prepared using a general procedure as described in the literature.[1] Commercially available aryl halides and benzoic acid derivatives were used as received. Thin layer chromatography was performed on Merck precoated silica gel 60 F_{254} plates. Silica gel (Merck, 70-230 and 230-400 mesh) was used for column chromatography. Melting points were recorded on an uncorrected Büchi Melting Point B-545 instrument. ^{1}H NMR spectra were recorded on a Bruker (500 MHz) spectrometer. Spectra were referenced internally to the residual proton resonance in $CDCl_3$ (δ 7.26 ppm), or with tetramethylsilane (TMS, δ 0.00 ppm) as the internal standard. Chemical shifts (δ) were reported as part per million (ppm) in δ scale downfield from TMS. ^{13}C NMR spectra were referenced to $CDCl_3$ (δ 77.00 ppm, the middle peak). ^{31}P NMR spectra were referenced to 85% H_3PO_4 externally. Coupling constants (*J*) were reported in Hertz (Hz). Mass spectra (EI-MS and ES-MS) were recorded on a HP 5989B Mass Spectrometer. High-resolution mass spectra (HRMS) were obtained on a Brüker APEX 47e FTICR mass spectrometer (ESIMS). GC-MS analysis was conducted on a HP 5973 GCD system using a HP5MS column (30 m × 0.25 mm). The products described in GC yield were accorded to the authentic samples/dodecane calibration standard from HP 6890 GC-FID system. All yields reported refer to isolated yield of compounds estimated to be greater than 95% purity as determined by capillary gas chromatography (GC) or ^{1}H NMR. Compounds described in the literature were characterized by comparison of their ^{1}H, and or ^{13}C NMR spectra to the previously reported data. The procedures in this section are representative, and thus the yields may differ from those reported in tables.

2. General procedures and data for ligand and reaction condition screenings

General procedure for reaction optimization (Table 1, S1-3): (2-Bromobenzylidene)hydrazine (47.8 mg, 0.24 mmol), $Pd(OAc)_2$ (4.5 mg, 0.02 mmol), ligand (0.05 mmol) and base (0.6 mmol) were loaded to a Schlenk tube equipped with a Teflon-coated stir bar and screw cap. The tube was carefully evacuated and backfilled with nitrogen for three cycles. Norbornene (28 mg, 0.3 mmol) was added under a stream of nitrogen purging from the Schlenk line and 2-Iodotoluene (26 μL, 0.2 mmol), solvent (0.1 M, 2.0 mL) were added by syringe. The tube was placed into a preheated oil bath (130 °C) and stirred for 18 h. After completion of reaction, the reaction tube was allowed to reach room temperature. Ethyl acetate (~3 mL), dodecane (45 μL, internal standard), water (~2 mL) were added. The organic layer was subjected to GC analysis. The GC yield was previously calibrated by authentic sample/dodecane calibration curve.

A benchmarking reaction has been shown in Table S1 and we initiated the optimization with a survey of phosphine ligands. $PCyPh_2$ (Table S1, entry 2) was found to have the optimal balance of electron richness and steric hindrance, giving the highest yield in this comparison. More electron rich (Table S1, entry 1, 6) and electron-deficient ligands (Table S1, entry 7, 11) were poor performers in this survey. Bulky (Table S1, entry 4, 12-15) or bidentate (Table S1, entry 16) ligands gave low yields (13-45%) in general. A wild range of strong and mild bases promoted the reaction (Table S2, entry 1, 3-6, 9-10) while K_2CO_3 has been chosen due to its economic attractiveness and ease of handling. Na_2CO_3, Na_3PO_4 and K_2HPO_4 gave low yields (5-10%, Table S2, entry 2, 7-8), which is presumably due to their low solubility in toluene. Several solvents with a high boiling point were screened and DMF proved to be the best solvent (Table S2, entry 12). The stoichiometries of reagents were also optimized as shown in Table S3. 1.5 equiv of norbornene and 1.2 equiv of hydrazone **2a** was found to provide the highest product yield (80%, Table S3, entry 1).

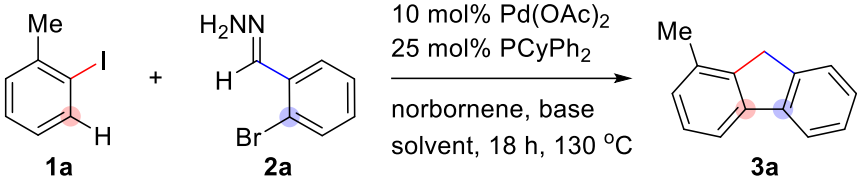
Table S1. Phopsphine ligand screening[a]



Entry	ligand	% yield[b]
1	PCy_3	57
2	$PCyPh_2$	74
3	PPh_3	38
4	$P(t\text{-}Bu)_3{\cdot}HBF_4$	trace
5	P(2-pyridyl)Ph_2	47
6	P(4-OMePh)$_3$	43
7	P(4-CF_3Ph)$_3$	19
8	P(2-OMePh)$_3$	58
9	P(2-MePh)$_3$	41
10	P(2-furyl)$_3$	59
11	P(C_6F_5)$_3$	0
12	cataCXium ABn	13
13	cataCXium A	19
14	XPhos	37
15	PPh_2-DavePhos	45
16	DPPF	31

[a]Reaction conditions: $Pd(OAc)_2$ (10 mol%), ligand (25 mol%), 2-iodotoluene (0.2 mmol), (2-bromobenzylidene)hydrazine (0.24 mmol), norbornene (0.3 mmol), K_2CO_3 (0.6 mmol), toluene (0.1 M, 2 mL), 130 °C for 18 h under N_2. DPPF = 1,1'-bis(diphenylphosphino)ferrocene, cataCXium A = di(1-adamantyl)-n-butylphosphine, XPhos = 2-Dicyclohexylphosphino-2′,4′,6′-triisopropylbiphenyl, PPh_2-DavePhos = 2-dicyclohexylphosphino-2′-(N,N-dimethylamino)biphenyl, cataCXium ABn = Di(1-adamantyl)benzylphosphine. [b]Calibrated GC-FID yields were reported.

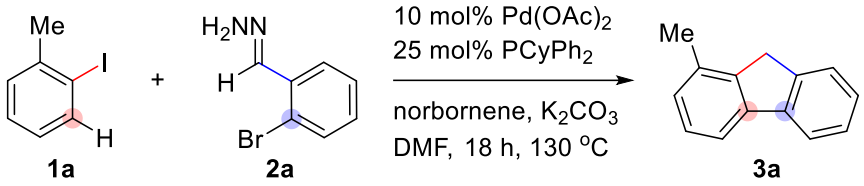
Table S2. Screening of solvent and base[a]



Entry	Base	Solvent	% yield[b]
1	K_2CO_3	toluene	74
2	Na_2CO_3	toluene	5
3	Cs_2CO_3	toluene	73
4	CsOAc	toluene	70
5	KOAc	toluene	31
6	K_3PO_4	toluene	59
7	KH_2PO_4	toluene	9
8	Na_3PO_4	toluene	10
9	KOH	toluene	72
10	NaOH	toluene	73
11	KO*t*-Bu	toluene	trace
12	K_2CO_3	DMF	80
13	K_2CO_3	DMA	74
14	K_2CO_3	NMP	78
15	K_2CO_3	MeCN	39
16	K_2CO_3	diglyme	73
17	K_2CO_3	xylene	76
18	K_2CO_3	mesitylene	77
19	K_2CO_3	1,4-dioxane	76

[a]Reaction conditions: $Pd(OAc)_2$ (10 mol%), $PCyPh_2$ (25 mol%), 2-iodotoluene (0.2 mmol), (2-bromobenzylidene)hydrazine (0.24 mmol), norbornene (0.3 mmol), base (0.6 mmol), solvent (0.1 M, 2 mL), 130 °C for 18 h under N_2. [b]Calibrated GC-FID yields were reported.

Table S3. Stoichiometry screening of reagents[a]



Entry	Equiv of norbornene	Equiv of **2a**	% yield[b]
1	1.5	1.2	80
2	1.0	1.2	78
3	0.5	1.2	75
4	0	1.2	0
5[c]	1.5	1.2	65
6	1.5	1.5	79

[a]Reaction conditions: $Pd(OAc)_2$ (10 mol%), $PCyPh_2$ (25 mol%), 2-iodotoluene (0.2 mmol), (2-bromobenzylidene)hydrazine, norbornene, K_2CO_3 (0.6 mmol), DMF (0.1 M, 2 mL), 130 °C for 18 h under N_2. [b]Calibrated GC-FID yields were reported. [c]Reaction at 110 °C.

3. General procedures for regioselective synthesis of fluorenes

General procedure for regioselective synthesis of fluorenes: Norbornene (141 mg, 1.5 mmol), $Pd(OAc)_2$ (22.5 mg, 0.1 mmol) and $PCyPh_2$ (67.1 mg, 0.25 mmol) were dissolved in DMF (10.0 mL, 5.0 mol% Pd per 2.0 mL stock solution) under N_2 and stirred for 5 minutes to give a light yellow milky solution. 2-Bromoaryl aldehyde hydrazone derivatives (0.24 mmol), aryl halide (if solid, 0.2 mmol) and K_2CO_3 (82.8 mg, 0.6 mmol) were loaded to a Schlenk tube equipped with a Teflon-coated stir bar and screw cap. The tube was carefully evacuated and backfilled with nitrogen for three cycles. Aryl halide (if liquid, 0.2 mmol) and stock solution (0.1 M, 2.0 mL) were added by syringe and the tube was placed into a preheated oil bath (130 °C) and stirred for 18 h. After completion of reaction, the reaction tube was allowed to reach room temperature. Ethyl acetate (~4 mL) and water (~3 mL) were added. The organic layer was separated and the aqueous layer was washed with ethyl acetate. The filtrate was concentrated under reduced pressure. The crude products were purified by flash column chromatography on silica gel (230-400 mesh) to afford the desired product.

4. Procedure for gram-scale reaction

General procedure for gram-scale synthesis: $Pd(OAc)_2$ (224.5 mg, 1 mmol), $PCyPh_2$ (670 mg, 2.5 mmol), (2-bromobenzylidene)hydrazine (2388 mg, 12 mmol) and K_2CO_3 (4140 mg, 30 mmol) were loaded to a Schlenk flask (500 mL) equipped with a Teflon-coated stir bar and screw cap. The tube was carefully evacuated and backfilled with nitrogen for three cycles. Norbornene (1410 mg, 15 mmol) was then added under a stream of nitrogen purging from the Schlenk line. 2-Iodotoluene (1300 μL, 10 mmol) and DMF (0.1 M, 100 mL) were added by syringe. The mixture was stirred for 2 minutes at room temperature and placed into a preheated oil bath (130 °C) and stirred for 18 h. After completion of reaction, the reaction tube was allowed to reach room temperature. Ethyl acetate (~3 mL), and water (~2 mL) were added. The filtrate was concentrated under reduced pressure. The crude products were purified by flash column chromatography on silica gel (230-400 mesh) to afford the desired product.

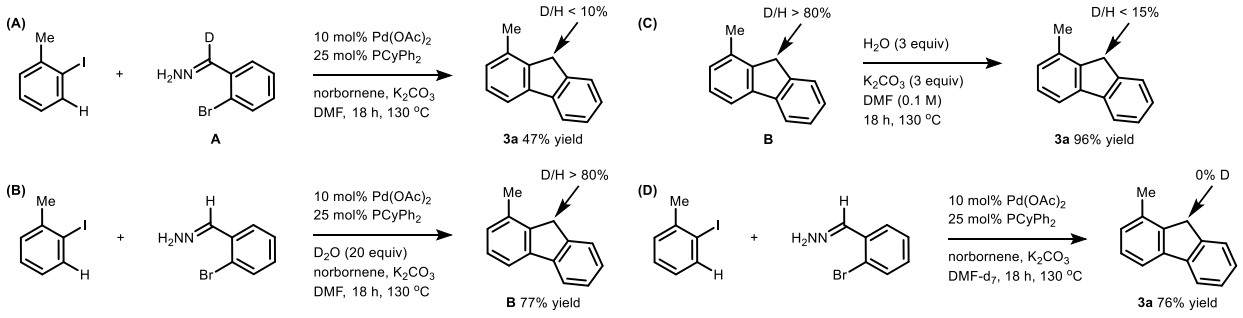
5. Procedure for three-component reaction

General procedure for three-component synthesis of fluorenes: $Pd(OAc)_2$ (4.5 mg, 0.02 mmol), $PCyPh_2$ (13.4 mg, 0.05 mmol), 2-bromobenzaldehyde (31 μL, 0.26 mmol) and K_2CO_3 (82.8 mg, 0.6 mmol) were loaded to a Schlenk tube equipped with a Teflon-coated stir bar and screw cap. The tube was carefully evacuated and backfilled with nitrogen for three cycles. Norbornene (28 mg, 0.3 mmol) was then added under a stream of nitrogen purging from the Schlenk line. 2-Iodotoluene (26 μL, 0.2 mmol), hydrazine hydrate (13 mg, 0.26 mmol) and DMF (0.1 M, 2.0 mL) were added by syringe. The tube was stirred for 5 minutes at room temperature and placed into a preheated oil bath (130 °C) and stirred for 18 h. After completion of reaction, the reaction tube was allowed to reach room temperature. Ethyl acetate (~3 mL), and water (~2 mL) were added. The filtrate was concentrated under reduced pressure. The crude products were purified by flash column chromatography on silica gel (230-400 mesh) to afford the desired product.

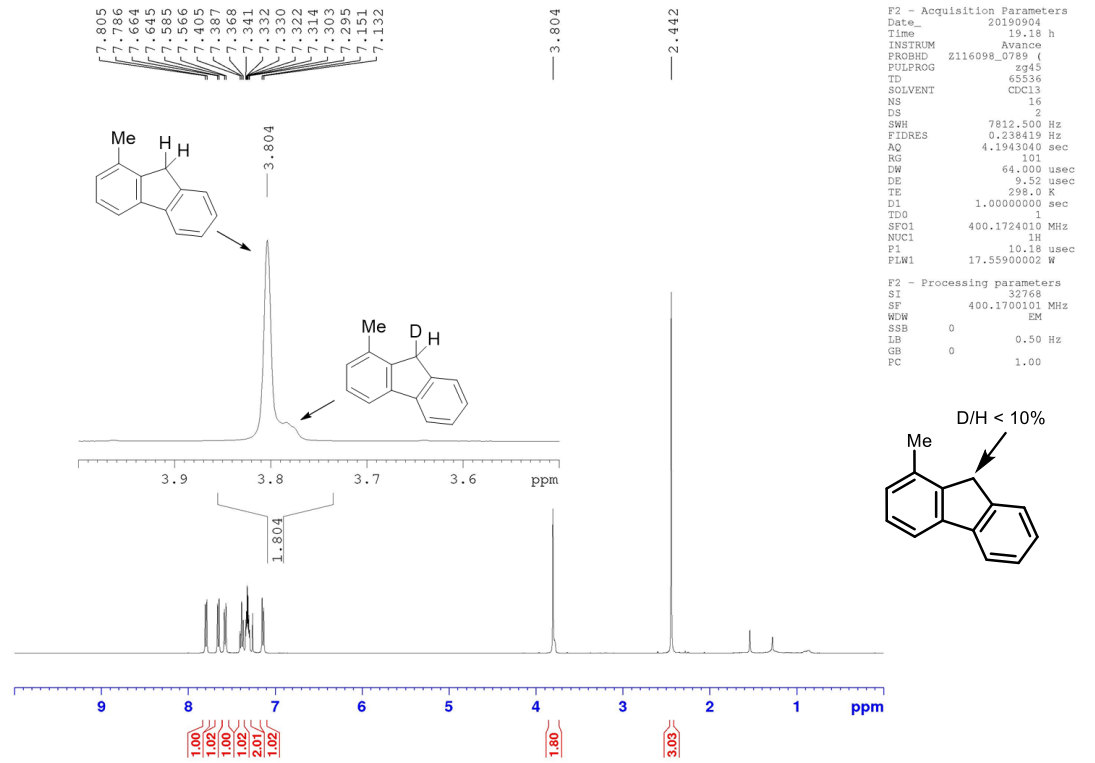
6. Procedure and NMR data for deuterium labelling study

Procedure for deuterium labelling study (Scheme S1A): Deuterium-labelled 2-bromobenzaldehyde was prepared according to reported literature procedure.[2] $Pd(OAc)_2$ (4.5 mg, 0.02 mmol), $PCyPh_2$ (13.4 mg, 0.05 mmol), deuterium labelled 2-bromobenzaldehyde hydrazone (48 mg, 0.24 mmol) and K_2CO_3 (82.8 mg, 0.6 mmol) were loaded to a Schlenk tube equipped with a Teflon-coated stir bar and screw cap. The tube was carefully evacuated and backfilled with nitrogen for three cycles. Norbornene (28 mg, 0.3 mmol) was then added under a stream of nitrogen purging from the Schlenk line. 2-Iodotoluene (26 μL, 0.2 mmol) and DMF (0.1 M, 2.0 mL) were added by syringe. The tube was stirred for 5 minutes at room temperature and placed into a preheated oil bath (130 °C) and stirred for 18 h. After completion of reaction, the reaction tube was allowed to reach room temperature. Ethyl acetate (~3 mL), and water (~2 mL) were added. The filtrate was concentrated under reduced pressure. The crude products were purified by flash column chromatography on silica gel (230-400 mesh) to afford a white powder (17.0 mg, 47% yield).

When deuterium labelled hydrazone **A** was tested under the standard reaction conditions, less than 10% deuterium was incorporated at the methylene position (Scheme S1A). The decreased yield of 47% might indicate a more difficult C-D activation of the hydrazone carbon at palladacycle **C**. However, the C9-proton of fluorene is weakly acidic and could undergo H/D exchange under basic conditions. We have prepared deuterium-labelled fluorene **B** by adding 20 equiv of D_2O into the standard reaction mixture (Scheme S1B). Treating **B** with 3 equiv of water and K_2CO_3 gave **3a** with less than 15% deuterium incorporation (Scheme S1C). When the reaction was carried out in DMF-d_7, no H/D exchange was observed at C9-H (Scheme S1D).



Scheme S1 Deuterium labelling study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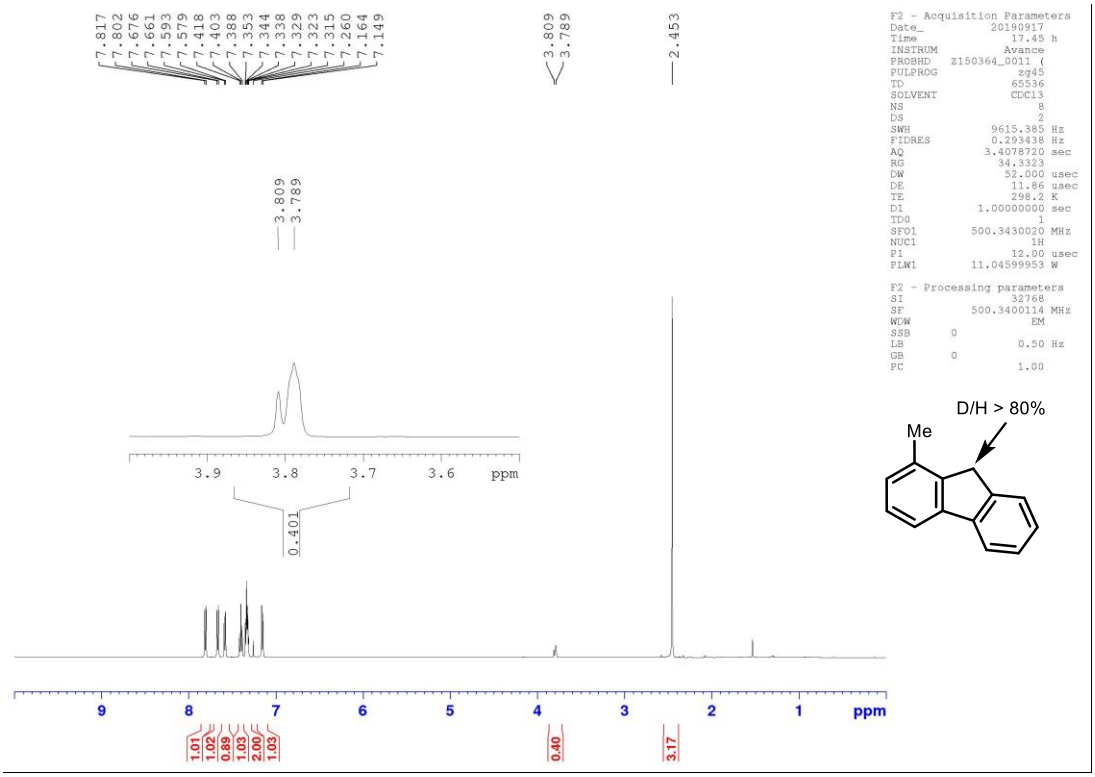
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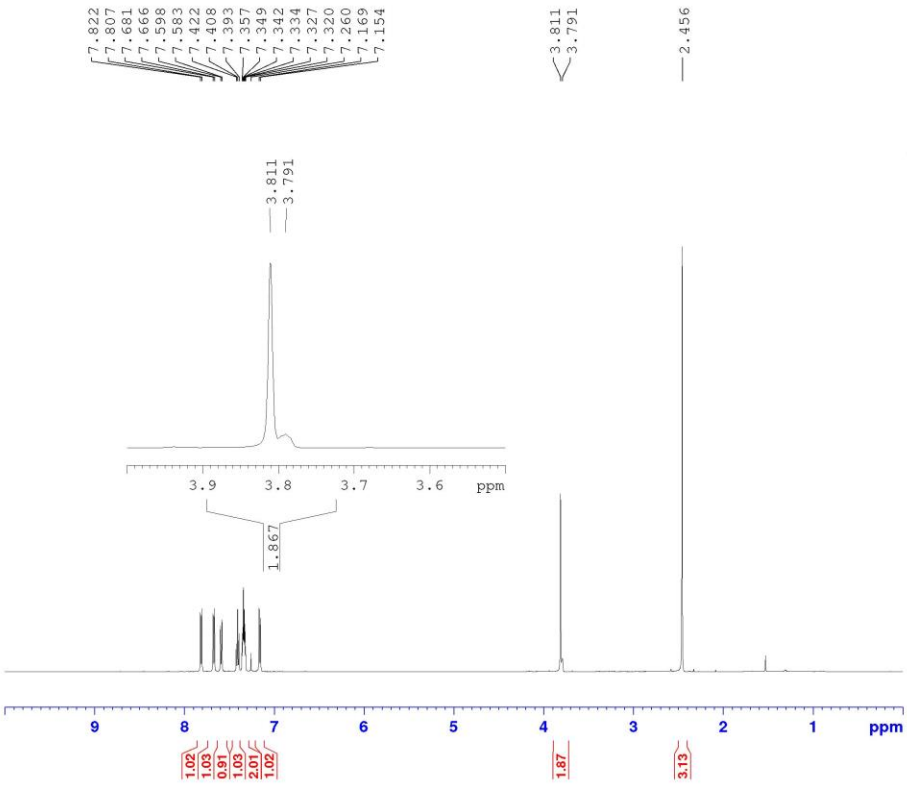


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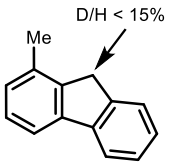
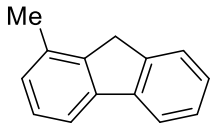


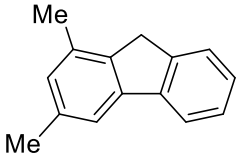
7. Characterization data for coupling products

1-Methyl-9*H*-fluorene (Table 2, entry 3a)[3]



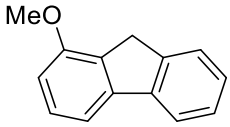
Yield: 81% (29.2 mg; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.45 (s, 3H), 3.81 (s, 2H), 7.16 (d, *J* = 7.4 Hz, 1H), 7.30 – 7.38 (m, 2H), 7.40 (t, *J* = 7.4 Hz, 1H), 7.59 (d, *J* = 7.4 Hz, 1H), 7.67 (d, *J* = 7.6 Hz, 1H), 7.81 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 19.0, 35.9, 117.4, 120.0, 125.0, 126.6, 126.7, 127.1, 127.8, 134.3, 141.4, 142.1, 142.1, 143.1.

1,3-Dimethyl-9*H*-fluorene (Table 2, entry 3b)[3]



Yield: 80% (31.0 mg); White solid; Melting point 86.6 – 88.6 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.42 (s, 3H), 2.47 (s, 3H), 3.77 (s, 2H), 6.99 (s, 1H), 7.32 (t, *J* = 7.4 Hz, 1H), 7.40 (t, *J* = 7.4 Hz, 1H), 7.49 (s, 1H), 7.58 (d, *J* = 7.4 Hz, 1H), 7.79 (d, *J* = 7.4 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 18.8, 21.4, 35.4, 117.9, 119.8, 125.0, 126.4, 126.6, 128.7, 133.8, 136.6, 139.1, 141.5, 142.0, 143.5; HRMS: calcd. for $C_{15}H_{14}$ $[M+H]^+$: 195.1168 found 195.1169.

1-Methoxy-9*H*-fluorene (Table 2, entry 3c)



Yield: 76% (29.8 mg); Light yellow solid; Melting point 47.8 – 49.4 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.88 (s, 2H), 3.97 (s, 3H), 6.87 (d, *J* = 8.0 Hz, 1H), 7.34 (t, *J* = 7.4, 1H), 7.37 – 7.43 (m, 2H), 7.46 (d, *J* = 7.5 Hz, 1H), 7.60 (d, *J* = 7.4 Hz, 1H), 7.81 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 34.3, 55.3, 108.6, 112.7, 120.1, 125.1, 126.6, 126.7, 128.4, 130.5, 141.8, 143.4, 143.4, 156.3; HRMS: calcd. for $C_{14}H_{13}O$ $[M+H]^+$: 197.0961 found 197.0961.

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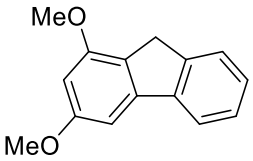
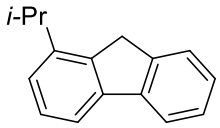


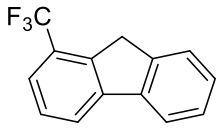
Yield: 70% (31.6 mg); Light yellow solid; Melting point 64.5 – 66.0 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.77 (s, 2H), 3.90 (s, 3H), 3.91 (s, 3H), 6.46 (d, *J* = 1.8 Hz, 1H), 6.96 (d, *J* = 1.8 Hz, 1H), 7.32 (t, *J* = 7.4 Hz, 1H), 7.38 (t, *J* = 7.4 Hz, 1H), 7.56 (d, *J* = 7.4 Hz, 1H), 7.74 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 33.7, 55.4, 55.7, 96.3, 97.5, 119.9, 123.2, 125.1, 126.5, 126.7, 141.7, 143.6, 144.1, 156.8, 161.0; HRMS: calcd. for $C_{15}H_{15}O_2$ $[M+H]^+$: 227.1067 found 227.1067.

1-Isopropyl-9*H*-fluorene (Table 2, entry 3e)



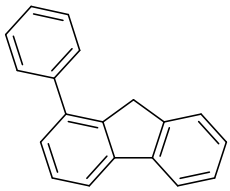
Yield: 79% (32.9 mg); White solid; Melting point 47.8 – 49.4 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.39 (s, 3H), 1.40 (s, 3H), 3.25 (hept, *J* = 6.9 Hz, 1H), 3.94 (s, 2H), 7.29 (d, *J* = 7.4 Hz, 1H), 7.35 (t, *J* = 7.4 Hz, 1H), 7.39 – 7.48 (m, 2H), 7.61 (d, *J* = 7.4 Hz, 1H), 7.70 (d, *J* = 7.4 Hz, 1H), 7.84 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 23.1, 31.1, 35.5, 117.5, 119.9, 123.2, 124.9, 126.6, 126.7, 127.5, 140.7, 141.5, 142.2, 143.0, 145.1; HRMS: calcd. for $C_{16}H_{17}$ $[M+H]^+$: 209.1325 found 209.1324.

1-Trifluoromethyl-9*H*-fluorene (Table 2, entry 3f)



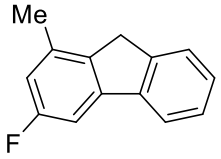
Yield: 48% (22.5 mg); Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.09 (s, 2H), 7.32 – 7.45 (m, 2H), 7.49 (t, *J* = 7.6 Hz, 1H), 7.53 – 7.63 (m, 2H), 7.82 (d, *J* = 7.4 Hz, 1H), 7.95 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 36.1, 120.1, 123.1, 123.3, 125.0, 125.6, 127.0, 127.2, 127.7, 140.1, 140.6, 142.9, 143.4; HRMS: calcd. for $C_{14}H_8F_3$ $[M-H]^-$: 233.0584 found 233.0584.

1-Phenyl-9*H*-fluorene (Table 2, entry 3g)[4]



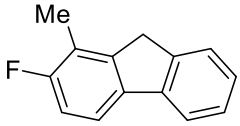
Yield: 58% (28.1 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.99 (s, 2H), 7.29 – 7.38 (m, 2H), 7.38 – 7.46 (m, 2H), 7.46 – 7.56 (m, 4H), 7.59 (d, *J* = 7.2 Hz, 2H), 7.83 (d, *J* = 7.5 Hz, 1H), 7.85 (d, *J* = 7.7 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 36.8, 118.9, 120.0, 124.8, 126.7, 126.8, 127.2, 127.3, 127.4, 128.5, 128.5, 139.1, 140.9, 141.1, 141.5, 142.1, 143.2.

3-Fluoro-1-methyl-9*H*-fluorene (Table 2, entry 3h)



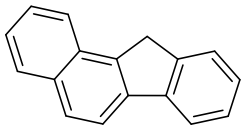
Yield: 76% (30.1 mg); White solid; Melting point 68.7 – 71.0 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.40 (s, 3H), 3.72 (s, 2H), 6.85 (dd, *J* = 10.2, 2.4 Hz, 1H), 7.29 (dd, *J* = 8.7, 2.4 Hz, 1H), 7.34 (td, *J* = 7.4, 1.2 Hz, 1H), 7.39 (t, *J* = 7.5 Hz, 1H), 7.56 (d, *J* = 7.4 Hz, 1H), 7.73 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 18.9 (d, *J* = 1.7 Hz), 35.2, 104.2 (d, *J* = 22.9 Hz), 114.4 (d, *J* = 22.7 Hz), 120.2, 125.0, 126.8, 127.1, 135.7 (d, *J* = 8.6 Hz), 137.2 (d, *J* = 2.2 Hz), 141.3 (d, *J* = 3.3 Hz), 142.9 (d, *J* = 9.4 Hz), 143.8, 162.7 (d, *J* = 242.5 Hz); HRMS: calcd. for $C_{14}H_{12}F$ $[M+H]^+$: 199.0918 found 199.0918.

2-Fluoro-1-methyl-9*H*-fluorene (Table 2, entry 3i)



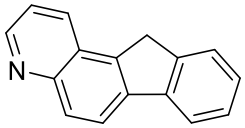
Yield: 70% (26.9 mg); White solid; Melting point 93.0 – 95.1 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.35 (d, *J* = 1.7 Hz, 3H), 3.78 (s, 2H), 7.07 (t, *J* = 9.1 Hz, 1H), 7.29 (t, *J* = 7.4 Hz, 1H), 7.38 (t, *J* = 7.4 Hz, 1H), 7.51 – 7.59 (m, 2H), 7.72 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 11.4 (d, *J* = 4.0 Hz), 35.9 (d, *J* = 3.1 Hz), 113.8 (d, *J* = 24.2 Hz), 117.9 (d, *J* = 9.1 Hz), 119.5, 121.4 (d, *J* = 18.7 Hz), 124.9, 126.1, 126.8, 137.0 (d, *J* = 2.6 Hz), 141.4, 142.9 (d, *J* = 2.0 Hz), 144.5 (d, *J* = 5.8 Hz), 160.6 (d, *J* = 243.3 Hz); HRMS: calcd. for $C_{14}H_{12}F$ $[M+H]^+$: 199.0918 found 199.0918.

11*H*-Benzo[*a*]fluorene (Table 2, entry 3j)[5]



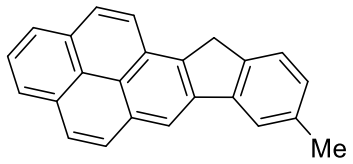
Yield: 59% (25.5 mg); ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.18 (s, 2H), 7.35 (t, *J* = 7.4 Hz, 1H), 7.43 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.5 Hz, 1H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.64 (d, *J* = 7.4 Hz, 1H), 7.85 (d, *J* = 7.4 Hz, 1H), 7.89 (d, *J* = 8.4 Hz, 1H), 7.91 – 7.98 (m, 2H), 8.02 (d, *J* = 8.2 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 35.6, 118.7, 119.6, 124.0, 124.9, 125.3, 126.3, 126.4, 126.8, 127.8, 128.9, 130.7, 132.8, 138.9, 139.8, 142.6, 143.3.

11*H*-Indeno[2,1-*f*]quinoline (Table 2, entry 3k)



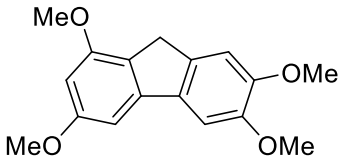
Yield: 48% (20.8 mg); Light brown solid; Melting point 125.4 – 128.3 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.13 (s, 2H), 7.34 (t, *J* = 7.4 Hz, 1H), 7.40 – 7.43 (m, 2H), 7.61 (d, *J* = 7.5 Hz, 1H), 7.83 (d, *J* = 7.5 Hz, 1H), 8.09 – 8.13 (m, 2H), 8.28 (dd, *J* = 8.5, 1.2 Hz, 1H), 8.89 (dd, *J* = 4.3, 1.5 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 35.2, 119.9, 121.3, 122.0, 124.9, 125.7, 126.7, 127.0, 129.2, 132.2, 139.5, 139.6, 141.8, 143.3, 147.9, 149.6; HRMS: calcd. for $C_{16}H_{12}N$ $[M+H]^+$: 218.0964 found 218.0964.

11*H*-Indeno[2,1-*a*]pyrene (Table 2, entry 3l)



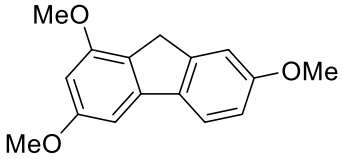
Yield: 62% (36.0 mg); Yellow solid; Melting point 209.8 – 213.3 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.54 (s, 3H), 4.35 (s, 2H), 7.22 (d, *J* = 7.9 Hz, 1H), 7.58 (d, *J* = 7.5 Hz, 1H), 7.87 (s, 1H), 7.97 (t, *J* = 7.6 Hz, 1H), 8.04 (d, *J* = 8.9 Hz, 1H), 8.09 – 8.20 (m, 5H), 8.50 (s, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.6, 35.3, 116.1, 120.9, 123.8, 124.0, 124.9, 125.1, 125.1, 125.2, 125.5, 126.8, 127.4, 127.9, 127.9, 128.1, 130.9, 131.0, 131.1, 136.6, 138.3, 139.7, 140.7, 142.4; HRMS: calcd. for $C_{24}H_{17}$ $[M+H]^+$: 305.1324 found 305.1323.

1,3,6,7-Tetramethoxy-9*H*-fluorene (Table 2, entry 3m)



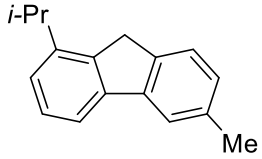
Yield: 58% (33.2 mg); Light yellow solid; Melting point 139.6 – 141.3 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.67 (s, 2H), 3.89 (s, 3H), 3.90 (s, 3H), 3.94 (s, 3H), 3.99 (s, 3H), 6.39 (d, *J* = 2.0 Hz, 1H), 6.83 (d, *J* = 2.0 Hz, 1H), 7.08 (s, 1H), 7.22 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 33.6, 55.3, 55.7, 56.1, 56.2, 95.7, 96.4, 103.0, 108.3, 122.9, 134.2, 136.6, 144.0, 148.5, 148.8, 156.5, 161.0; HRMS: calcd. for $C_{17}H_{18}O_4Na$ $[M+Na]^+$: 309.1097 found 309.1095.

1,3,7-Trimethoxy-9*H*-fluorene (Table 2, entry 3n)



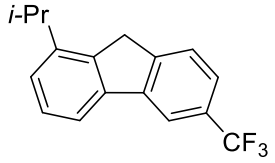
Yield: 65% (33.3 mg); Light yellow solid; Melting point 140.1 – 143.0 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.73 (s, 2H), 3.87 (s, 3H), 3.90 (s, 3H), 3.90 (s, 3H), 6.39 (d, *J* = 2.0 Hz, 1H), 6.86 (d, *J* = 2.0 Hz, 1H), 6.92 (dd, *J* = 8.4, 2.4 Hz, 1H), 7.10 (d, *J* = 2.5 Hz, 1H), 7.62 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 33.8, 55.3, 55.5, 55.6, 95.8, 96.4, 110.6, 112.7, 120.4, 122.3, 134.7, 143.5, 145.9, 156.6, 159.3, 161.0; HRMS: calcd. for $C_{16}H_{17}O_3$ $[M+H]^+$: 257.1172 found 257.1174.

1-Isopropyl-6-methyl-9*H*-fluorene (Table 2, entry 3o)



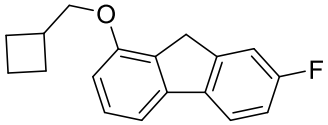
Yield: 90% (40.0 mg); White solid; Melting point 88.2 – 90.5 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.31 (s, 3H), 1.32 (s, 3H), 2.44 (s, 3H), 3.16 (hept, *J* = 6.9 Hz, 1H), 3.81 (s, 2H), 7.09 (d, *J* = 7.6, 1H), 7.20 (d, *J* = 7.2 Hz, 1H), 7.34 (t, *J* = 7.6 Hz, 1H), 7.40 (d, *J* = 7.6 Hz, 1H), 7.53 – 7.64 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.5, 23.0, 31.0, 35.0, 117.3, 120.4, 122.9, 124.5, 127.3, 127.5, 136.2, 140.0, 141.1, 141.5, 142.2, 145.0; HRMS: calcd. for $C_{17}H_{19}$ $[M+H]^+$: 223.1481 found 223.1481.

1-Isopropyl-6-(trifluoromethyl)-9*H*-fluorene (Table 2, entry 3p)



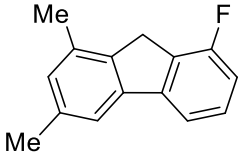
Yield: 60% (33.1 mg); Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.34 (s, 3H), 1.35 (s, 3H), 3.20 (hept, *J* = 6.9 Hz, 1H), 3.94 (s, 2H), 7.31 (d, *J* = 7.6 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 1H), 7.63 (d, *J* = 7.9 Hz, 1H), 7.69 (d, *J* = 7.6, 1H), 7.80 (s, 1H), 7.85 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 22.9, 31.1, 35.5, 118.2, 119.9, 121.8, 124.0, 124.4, 125.7, 127.8, 128.5, 140.1, 141.3, 143.2, 145.3, 145.5; HRMS: calcd. for $C_{17}H_{15}F_3$ $[M+H]^+$: 276.1120 found 276.1122.

1-(Cyclobutylmethoxy)-7-fluoro-9*H*-fluorene (Table 2, entry 3q)



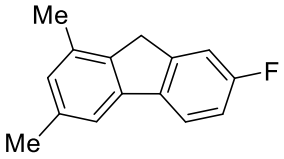
Yield: 67% (30.1 mg); Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ1.90 – 2.00 (m, 4H), 2.14 – 2.19 (m, 2H), 2.78 – 2.87 (m, 1H), 3.83 (s, 2H), 4.05 (d, *J* = 6.3 Hz, 2H), 6.80 (dd, *J* = 6.4, 2.5 Hz, 1H), 7.06 (td, *J* = 8.8, 2.1 Hz, 1H), 7.23 – 7.25 (m, 1H), 7.31 – 7.35 (m, 2H), 7.68 (dd, *J* = 8.6, 5.1 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 18.7, 24.8, 34.4 (d, *J* = 2.3 Hz), 34.8, 71.9, 109.5, 112.2, 112.3 (d, *J* = 22.7 Hz), 113.7 (d, *J* = 22.9 Hz), 120.8 (d, *J* = 8.8 Hz), 128.5, 130.7, 137.9, 142.5, 145.6 (d, *J* = 8.6 Hz), 155.9, 162.5 (d, *J* = 245.0 Hz); HRMS: calcd. for $C_{18}H_{18}FO$ $[M+H]^+$: 269.1336 found 269.1340.

8-Fluoro-1,3-dimethyl-9*H*-fluorene (Table 2, entry 3r)



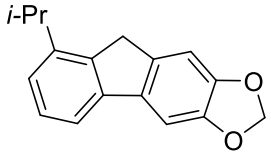
Yield: 83% (35.2 mg); White solid; Melting point 76.4 – 77.8 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.40 (s, 3H), 2.45 (s, 3H), 3.76 (s, 2H), 6.93 – 7.05 (m, 2H), 7.35 (td, *J* = 7.8, 5.0 Hz, 1H), 7.45 (s, 1H), 7.53 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 18.7, 21.4, 31.9, 113.0 (d, *J* = 20.7 Hz), 115.7 (d, *J* = 3.2 Hz), 118.4, 128.7 (d, *J* = 6.9 Hz), 128.8, 129.4, 134.1, 136.9, 138.8, 140.7 (d, *J* = 2.5 Hz), 145.4 (d, *J* = 6.5 Hz), 159.7 (d, *J* = 246.3 Hz); HRMS: calcd. for $C_{15}H_{14}F$ $[M+H]^+$: 213.1074 found 213.1074.

7-Fluoro-1,3-dimethyl-9*H*-fluorene (Table 2, entry 3s)



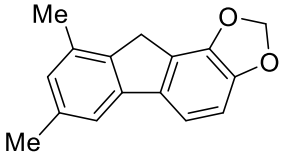
Yield: 80% (33.9 mg); Light yellow solid; Melting point 105.5 – 107.2 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.38 (s, 3H), 2.44 (s, 3H), 3.71 (s, 2H), 6.95 (s, 1H), 7.07 (td, *J* = 8.9, 2.4 Hz, 1H), 7.24 (dd, *J* = 9.0, 2.3 Hz, 1H), 7.40 (s, 1H), 7.67 (dd, *J* = 8.3, 5.1 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 18.7, 21.4, 35.5 (d, *J* = 2.5 Hz), 112.2 (d, *J* = 22.7 Hz), 113.7 (d, *J* = 22.9 Hz), 117.7, 120.6 (d, *J* = 8.9 Hz), 128.4, 133.8, 136.9, 138.0 (d, *J* = 2.3 Hz), 138.9 (d, *J* = 2.1 Hz), 140.6, 145.5 (d, *J* = 8.4 Hz), 162.2 (d, *J* = 243.9 Hz); HRMS: calcd. for $C_{15}H_{14}F$ $[M+H]^+$: 213.1074 found 213.1073.

8-Isopropyl-9*H*-fluoreno[2,3-*d*][1,3]dioxole (Table 2, entry 3t)



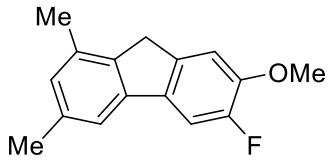
Yield: 80% (40.3 mg); Light yellow solid; Melting point 113.2 – 115.2 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.33 (s, 3H), 1.34 (s, 3H), 3.16 (hept, *J* = 6.9 Hz, 1H), 3.77 (s, 2H), 6.01 (s, 2H), 7.02 (s, 1H), 7.17 (d, *J* = 7.6 Hz, 1H), 7.22 (s, 1H), 7.35 (t, *J* = 7.6 Hz, 1H), 7.49 (d, *J* = 7.4 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 23.0, 30.9, 35.3, 100.5, 101.1, 105.6, 116.4, 122.0, 127.4, 135.9, 136.7, 140.7, 141.5, 144.7, 147.0, 147.1; HRMS: calcd. for $C_{17}H_{17}O_2$ $[M+H]^+$: 253.1223 found 253.1223.

7,9-Dimethyl-10*H*-fluoreno[1,2-*d*][1,3]dioxole (Table 2, entry 3u)



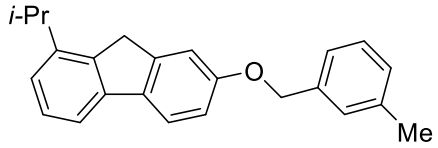
Yield: 80% (38.1 mg); Light yellow solid; Melting point 166.4 – 169.4 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.37 (s, 3H), 2.42 (s, 3H), 3.69 (s, 2H), 6.02 (s, 2H), 6.87 (d, *J* = 7.9 Hz, 1H), 6.92 (s, 1H), 7.25 (d, *J* = 7.9 Hz, 1H), 7.36 (s, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 18.7, 21.4, 31.7, 101.0, 107.2, 112.7, 117.6, 123.2, 128.2, 133.9, 136.8, 138.1, 138.2, 141.1, 143.5, 146.6; HRMS: calcd. for $C_{16}H_{15}O_2$ $[M+H]^+$: 239.1067 found 239.1068.

6-Fluoro-7-methoxy-1,3-dimethyl-9*H*-fluorene (Table 2, entry 3v)



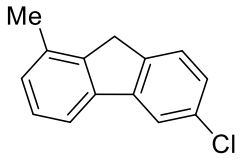
Yield: 63% (38.1 mg); White solid; Melting point 136.1 – 138.3 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.36 (s, 3H), 2.42, (s, 3H), 3.66 (s, 2H), 3.94 (s, 3H), 6.92, (s, 1H), 7.14 (d, *J* = 7.8 Hz, 1H), 7.30 (s, 1H), 7.41 (d, *J* = 11.1 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 18.7, 21.4, 35.4, 55.5, 107.5 (d, *J* = 19.8 Hz), 110.1 (d, *J* = 1.7 Hz), 117.4, 128.1, 133.7, 134.9 (d, *J* = 7.6 Hz), 136.9, 139.1, 139.1 (d, *J* = 3.3 Hz), 140.9 (d, *J* = 2.6 Hz), 146.7 (d, *J* = 11.8 Hz), 152.3 (d, *J* = 243.2 Hz); HRMS: calcd. for $C_{16}H_{16}FO$ $[M+Na]^+$: 243.1180 found 243.1183.

1-Isopropyl-7-((3-methylbenzyl)oxy)-9*H*-fluorene (Table 2, entry 3w)



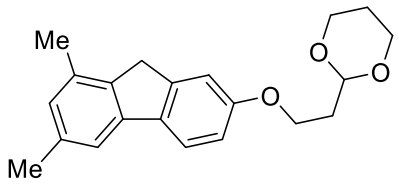
Yield: 66% (43.3 mg); Light yellow solid; Melting point 68.0 – 69.5 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.35 (s, 3H), 1.37 (s, 3H), 2.42 (s, 3H), 3.19 (hept, *J* = 6.9 Hz, 1H), 3.87 (s, 2H), 5.11 (s, 2H), 7.03 (dd, *J* = 8.4, 2.4 Hz, 1H), 7.14 – 7.24 (m, 3H), 7.30 – 7.37(m, 3H), 7.37 (s, 1H), 7.57 (dd, *J* = 7.5, 1.0 Hz, 1H), 7.69 (d, *J* = 8.3 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.4, 23.0, 31.0, 35.6, 70.3, 111.5, 113.7, 116.7, 120.5, 122.1, 124.6, 127.4, 128.2, 128.5, 128.7, 135.4, 137.0, 138.2, 140.1, 141.4, 144.7, 144.8, 158.4; HRMS: calcd. for $C_{24}H_{25}O$ $[M+H]^+$: 329.1900 found 329.1905.

6-Chloro-1-methyl-9*H*-fluorene (Table 2, entry 3x)



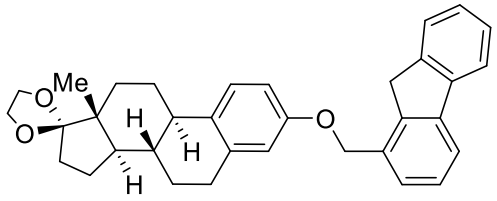
Yield: 48% (20.6 mg); White solid; Melting point 85.5 – 89.0 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.42 (s, 3H), 3.75 (s, 2H), 7.16 (d, *J* = 7.5 Hz, 1H), 7.24 – 7.28 (m, 1H), 7.32 (t, *J* = 7.5 Hz, 1H), 7.46 (d, *J* = 8.0 Hz, 1H), 7.60 (d, *J* = 7.6 Hz, 1H), 7.73 (d, *J* = 1.9 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 18.9, 35.5, 117.6, 120.2, 126.0, 126.5, 127.3, 128.4, 132.7, 134.4, 140.2, 141.2, 142.5, 143.8; HRMS: calcd. for $C_{14}H_{10}Cl$ $[M-H]^-$: 213.0477 found 213.0478.

2-(2-((6,8-Dimethyl-9*H*-fluoren-2-yl)oxy)ethyl)-1,3-dioxane (Table 2, entry 3y)



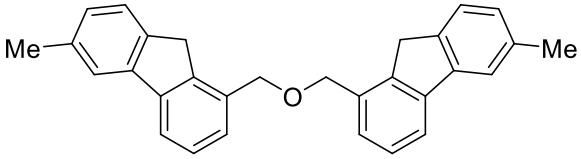
Yield: 74% (48.0 mg); Yellow solid; Melting point 149.5 – 152.2 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.30 – 1.50 (m, 1H), 2.07 – 2.20 (m, 3H), 2.37 (s, 3H), 2.43 (s, 3H), 3.70 (s, 2H), 3.82 (td, *J* = 12.3, 2.4 Hz, 2H), 4.10 – 4.24 (m, 4H), 4.84 (t, *J* = 5.2 Hz, 1H), 6.90 (s, 1H), 6.93 (dd, *J* = 8.3, 2.4 Hz, 1H), 7.12 (d, *J* = 2.3 Hz, 1H), 7.36 (s, 1H), 7.63 (d, *J* = 8.2 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 18.8, 21.4, 25.8, 35.2, 35.5, 63.5, 66.9, 99.5, 111.2, 113.3, 117.2, 120.4, 127.6, 133.6, 135.0, 136.6, 138.5, 141.4, 145.2, 158.3; HRMS: calcd. for $C_{21}H_{24}O_3Na$ $[M+Na]^+$: 347.1618 found 347.1616.

(8*R*,9*S*,13*S*,14*S*)-3-((9*H*-fluoren-1-yl)methoxy)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydrospiro[cyclopenta[*a*]phenanthrene-17,2'-[1,3]dioxolane] (Table 2, entry 3z)



Yield: 76% (45.3 mg); Light orange solid; Melting point 96.7 – 102.6 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.89 (s, 3H), 1.27 – 1.49 (m, 5H), 1.61 – 1.67 (m, 1H), 1.75 – 1.87 (m, 3H), 1.88 – 1.92 (m, 1H), 2.00 – 2.06 (m, 1H), 2.23 – 2.27 (m, 1H), 2.31 – 2.34 (m, 1H), 2.85 – 2.88 (m, 2H), 3.88 – 3.98 (m, 6H), 5.18, (s, 2H), 6.76 (d, *J* = 2.6 Hz, 1H), 6.82 (dd, *J* = 8.0 Hz, 1H), 7.23 (d, *J* = 8.6 Hz, 1H), 7.31 (td, *J* = 7.4, 0.9 Hz, 1H), 7.36 – 7.43 (m, 3H), 7.55 (d, *J* = 7.4 Hz, 1H), 7.78 (dd, *J* = 7.1, 1.2 Hz, 1H), 7.80, (d, *J* = 7.5Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 14.4, 22.4, 22.7, 26.2, 27.0, 29.9, 30.7, 31.6, 34.3, 35.6, 39.1, 43.7, 46.2, 49.4, 64.6, 65.3, 68.6, 112.2, 114.7, 119.5, 119.7, 120.0, 125.0, 126.3, 126.4, 126.8, 126.9, 127.3, 133.1, 133.4, 138.2, 141.5, 141.8, 142.2, 143.1, 156.7; HRMS: calcd. for $C_{34}H_{37}O_3$ $[M+H]^+$: 493.2737 found 493.2740.

8,8'-(Oxybis(methylene))bis(3-methyl-9*H*-fluorene) (Scheme 1, compound 4)



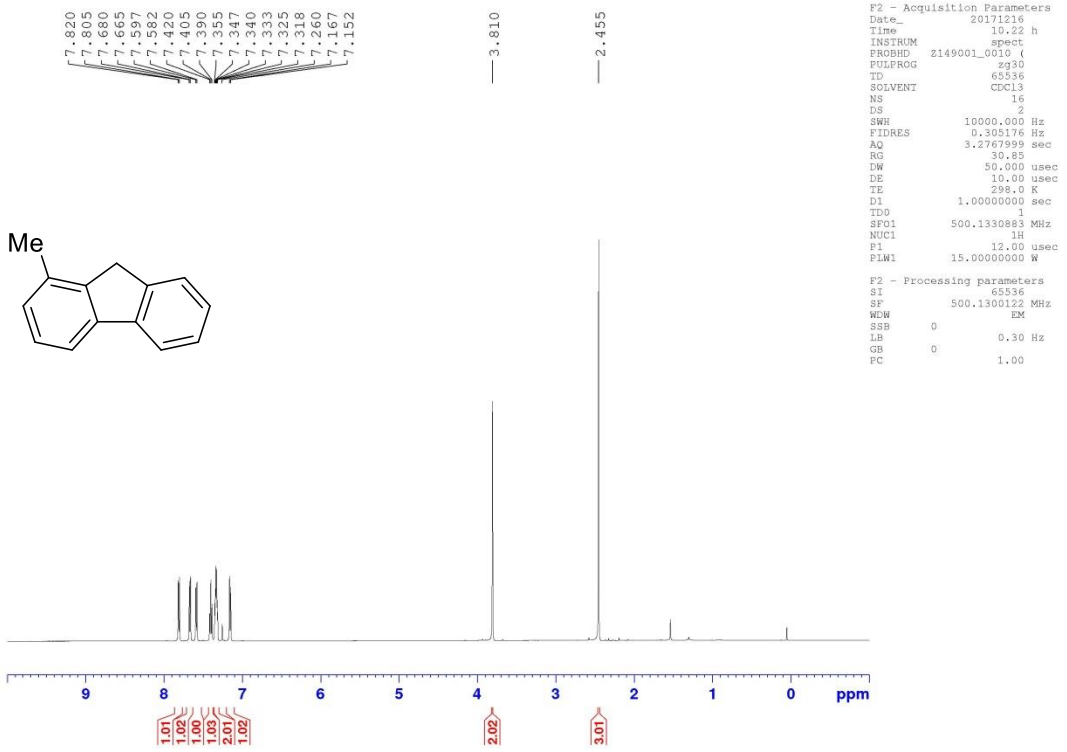
Yield: 51% (41.1 mg); Light orange solid; Melting point 174.0 – 176.6 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.47 (s, 6H), 3.84 (s, 4H), 4.75 (s, 4H), 7.13 (d, *J* = 7.6 Hz, 1H), 7.4 (d, *J* = 7.3 Hz, 1H), 7.38 – 7.43 (m, 4H), 7.62 (s, 2H), 7.74 (d, *J* = 7.3 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.5, 35.1, 70.4, 119.3, 120.5, 124.7, 126.3, 127.1, 127.7, 134.3, 136.3, 140.2, 141.7, 142.0, 142.2; HRMS: calcd. for $C_{30}H_{26}ONa$ $[M+Na]^+$: 425.1876 found 425.1876.

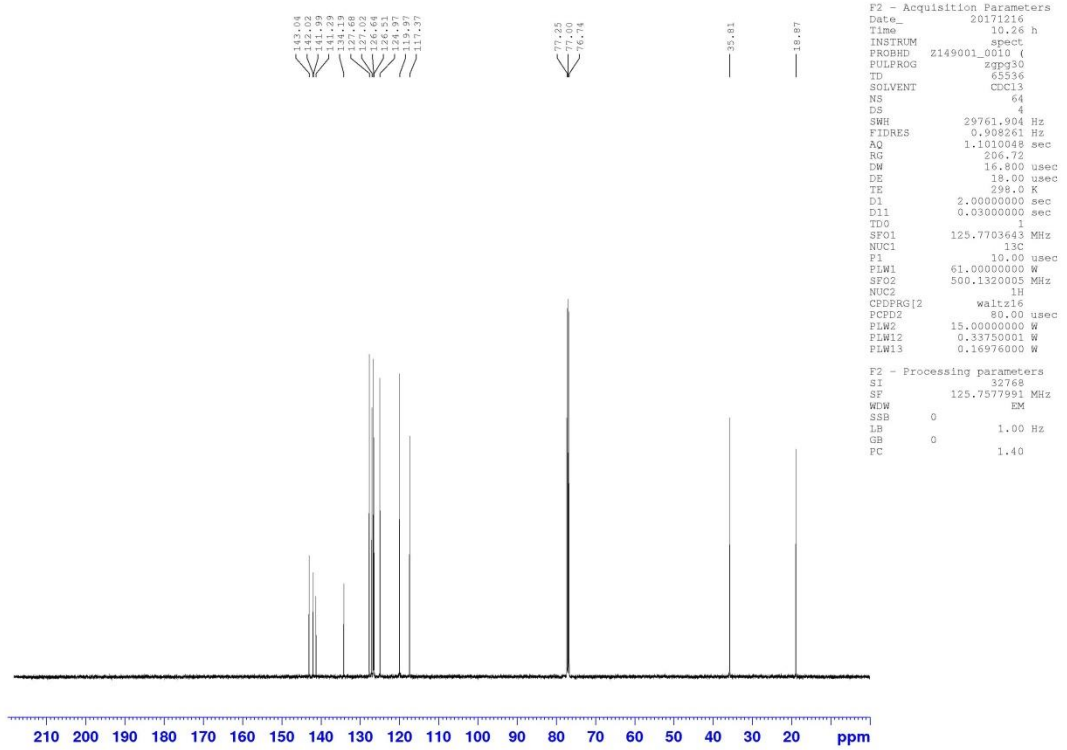
8. References

1. K. G. Kulkarni, B. Miokovic, M. Sauder, and G. K. Murphy, *Org. Biomol. Chem.,* 2016, **14,** 9907-9911.
2. L.-Z. Dai and M. Shi, *Chem. Eur. J..*, 2010, **16,** 2496-2502.
3. S. J. Hwang, H. J. Kim, and S. Chang, *Org. Lett.,* 2009, **11,** 4588-4591.
4. H. Rapoport and G, Smolinsky, *J. Am. Chem. Soc.,* 1960, **82,** 934-941.
5. R. G. Harvey, J. Pataki, C. Cortez, P. Di Raddo, and C. X. Yang, *J. Org. Chem.,* 1991, **56,** 1210-1217.

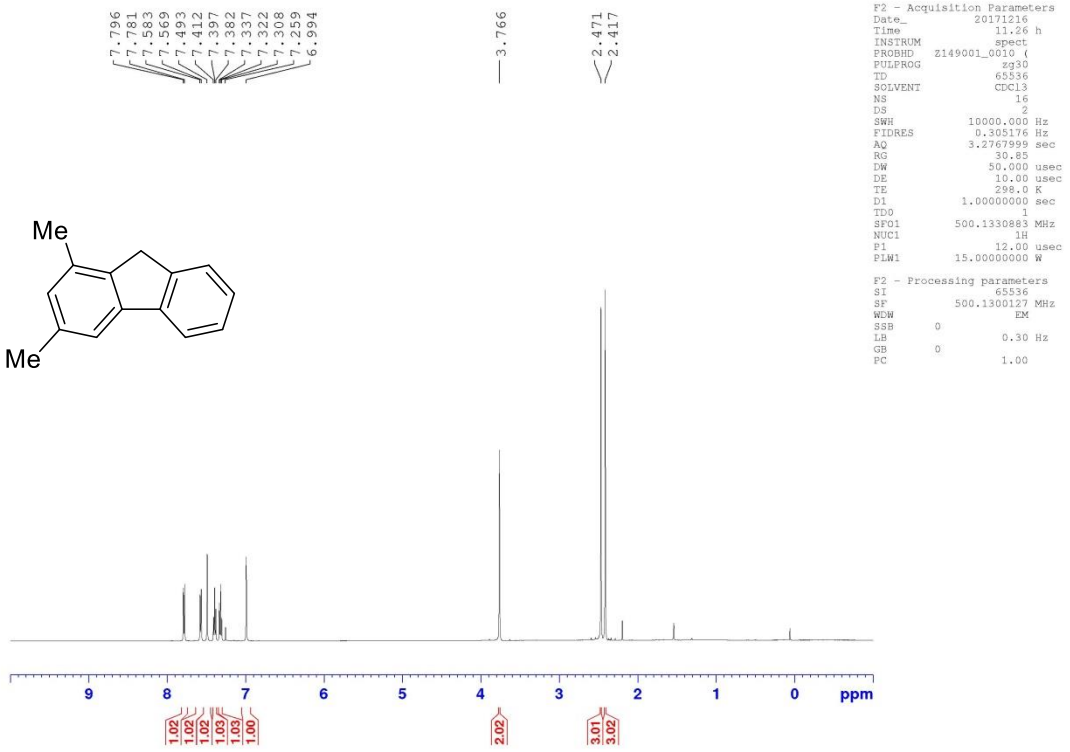
9. ^{1}H and ^{13}C NMR spectra

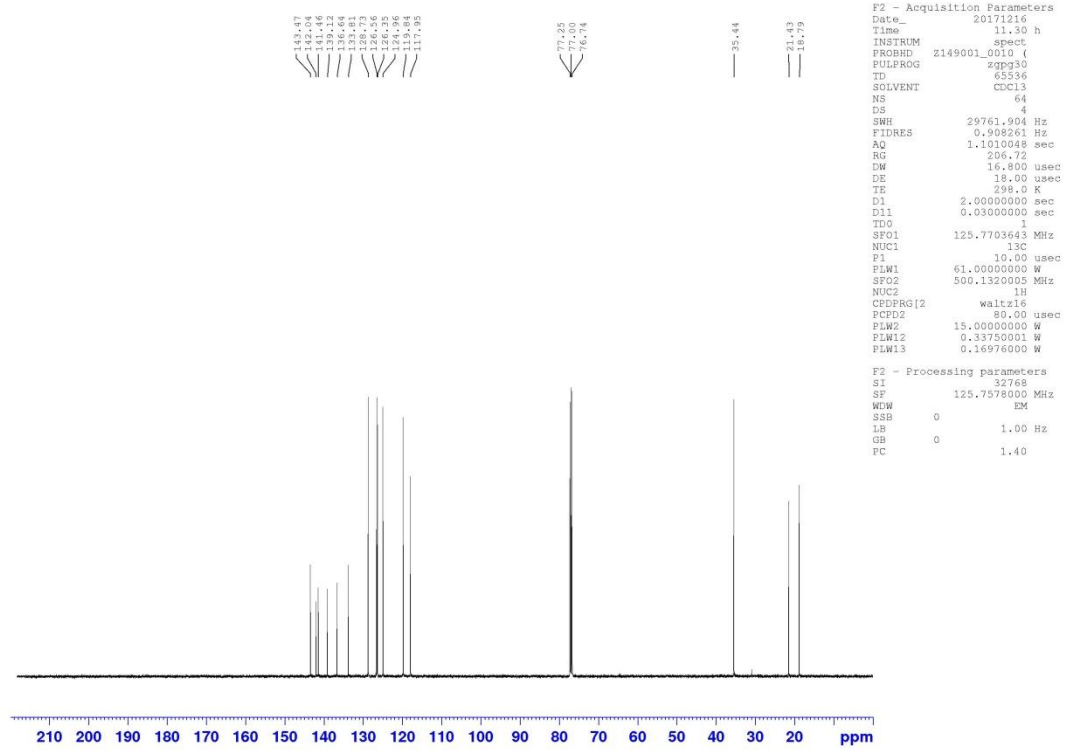
^{1}H NMR and ^{13}C NMR spectra for 1-Methyl-9*H*-fluorene (Table 2, entry 3a)



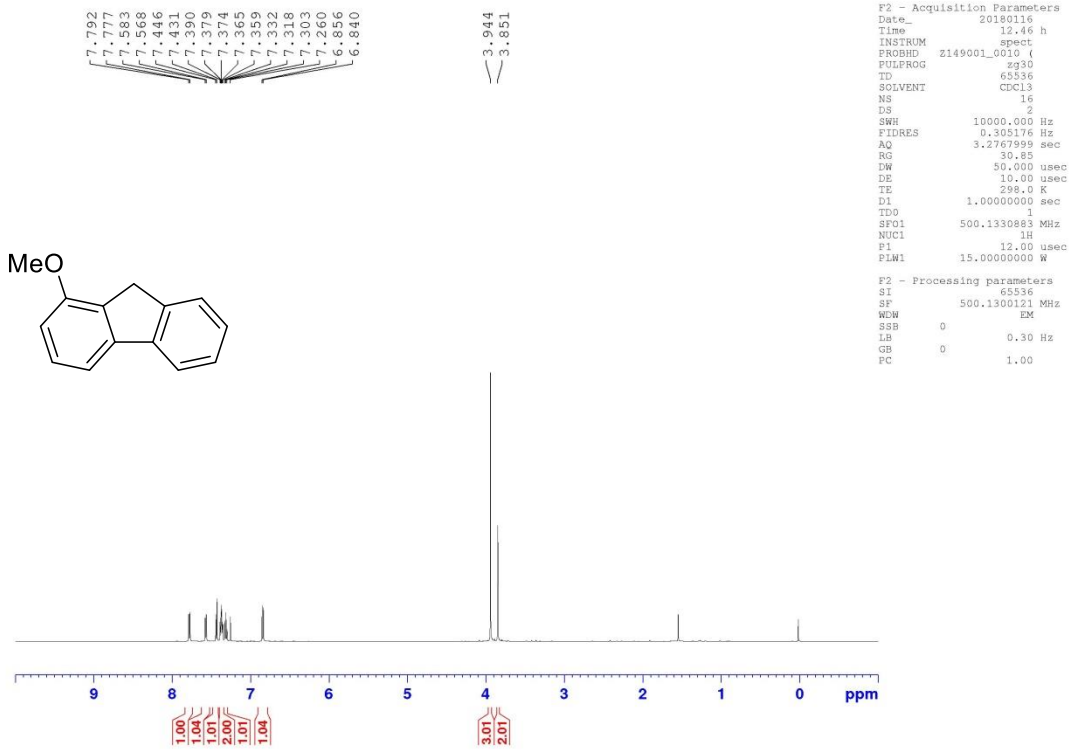


^{1}H NMR and ^{13}C NMR spectra for 1,3-Dimethyl-9*H*-fluorene (Table 2, entry 3b)





^{1}H NMR and ^{13}C NMR spectra for 1-Methoxy-9*H*-fluorene (Table 2, entry 3c)

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FIDRES              0.305176 Hz
AQ                3.2767999 sec
RG                    30.85
DW                   50.000 usec
DE                   10.00 usec
TE                   298.0 K
D1              1.00000000 sec
TD0                      1
SFO1           500.1330883 MHz
NUC1                    1H
P1                   12.00 usec
PLW1           15.00000000 W

F2 - Processing parameters
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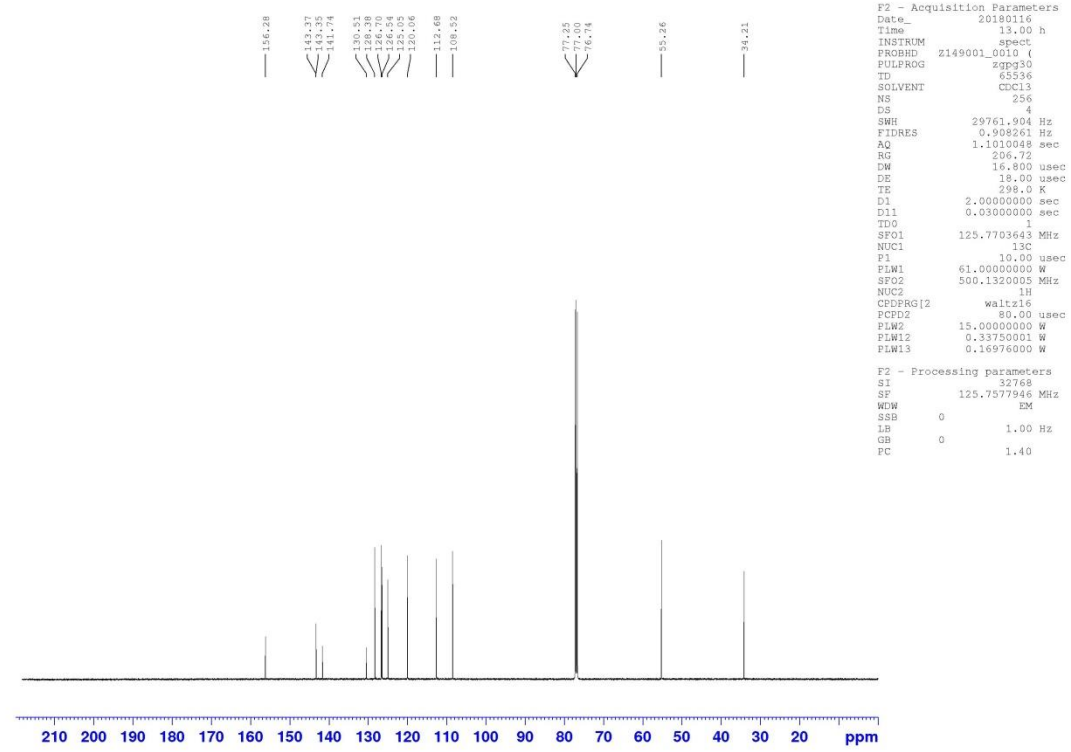


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TD                   65536
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NS                     256
DS                       4
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FIDRES              0.908261 Hz
AQ                1.1010048 sec
RG                   206.72
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DE                   18.00 usec
TE                   298.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1
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NUC1                    13C
P1                   10.00 usec
PLW1           61.00000000 W
SFO2           500.1320005 MHz
NUC2                    1H
CPDPRG[2            waltz16
PCPD2                80.00 usec
PLW2           15.00000000 W
PLW12           0.33750001 W
PLW13           0.16976000 W

F2 - Processing parameters
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SF             125.7577946 MHz
WDW                     EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.40
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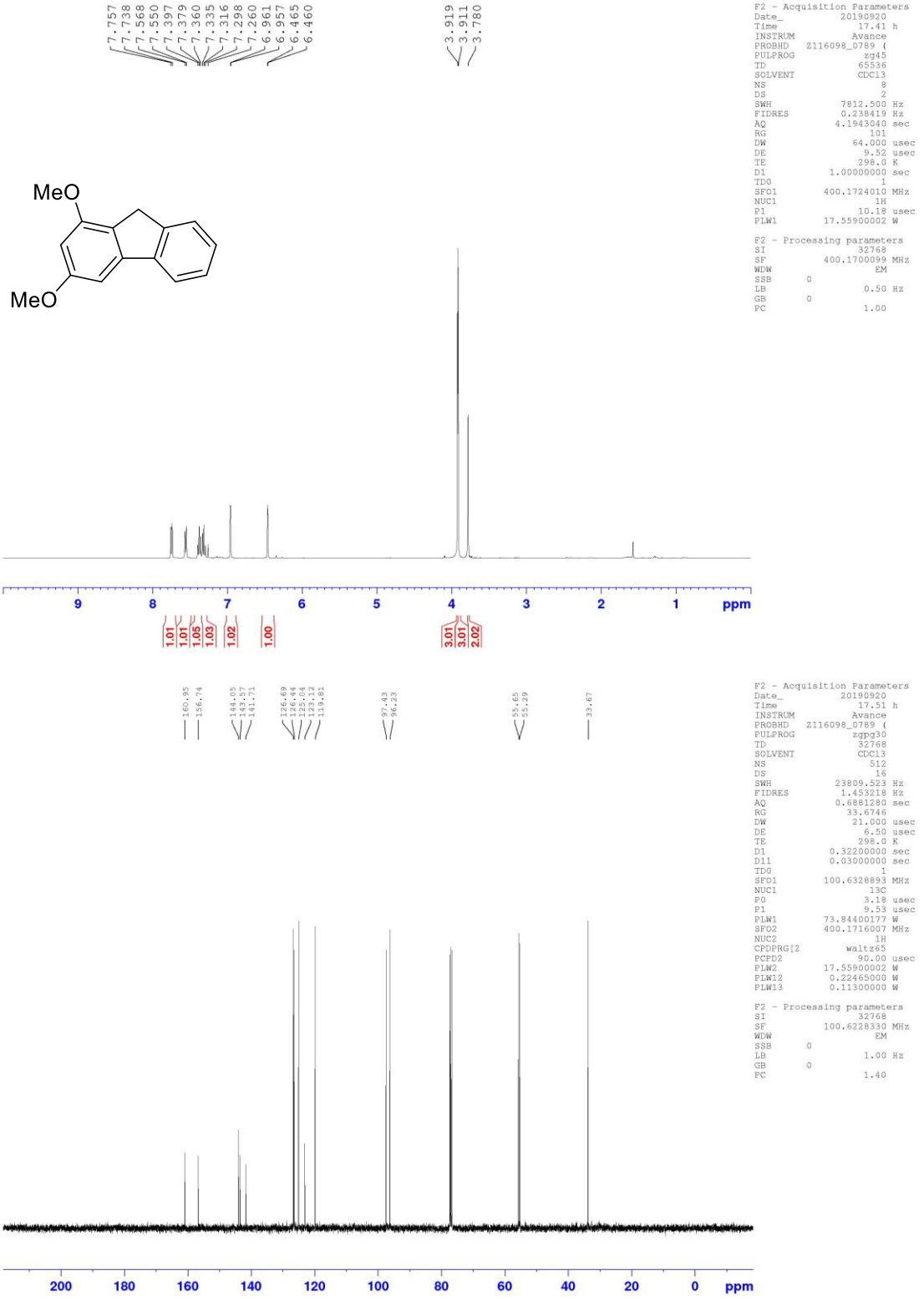
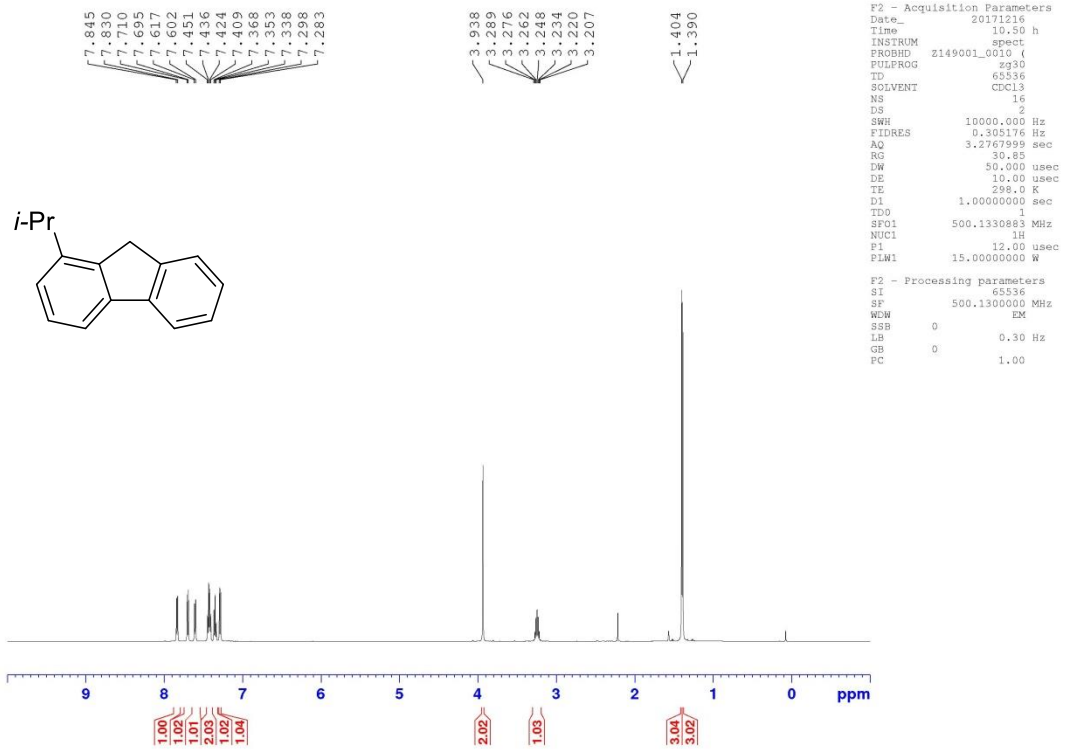
^{1}H NMR and ^{13}C NMR spectra for 1,3-Dimethoxy-9*H*-fluorene (Table 2, entry 3d)



^{1}H NMR and ^{13}C NMR spectra for 1-Isopropyl-9*H*-fluorene (Table 2, entry 3e)

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TD                  65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH              10000.000 Hz
FIDRES               0.305176 Hz
AQ                3.2767999 sec
RG                    30.85
DW                  50.000 usec
DE                  10.00 usec
TE                  298.0 K
D1               1.00000000 sec
TD0                      1
SFO1           500.1330883 MHz
NUC1                    1H
P1                  12.00 usec
PLW1            15.00000000 W

F2 - Processing parameters
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SSB          0
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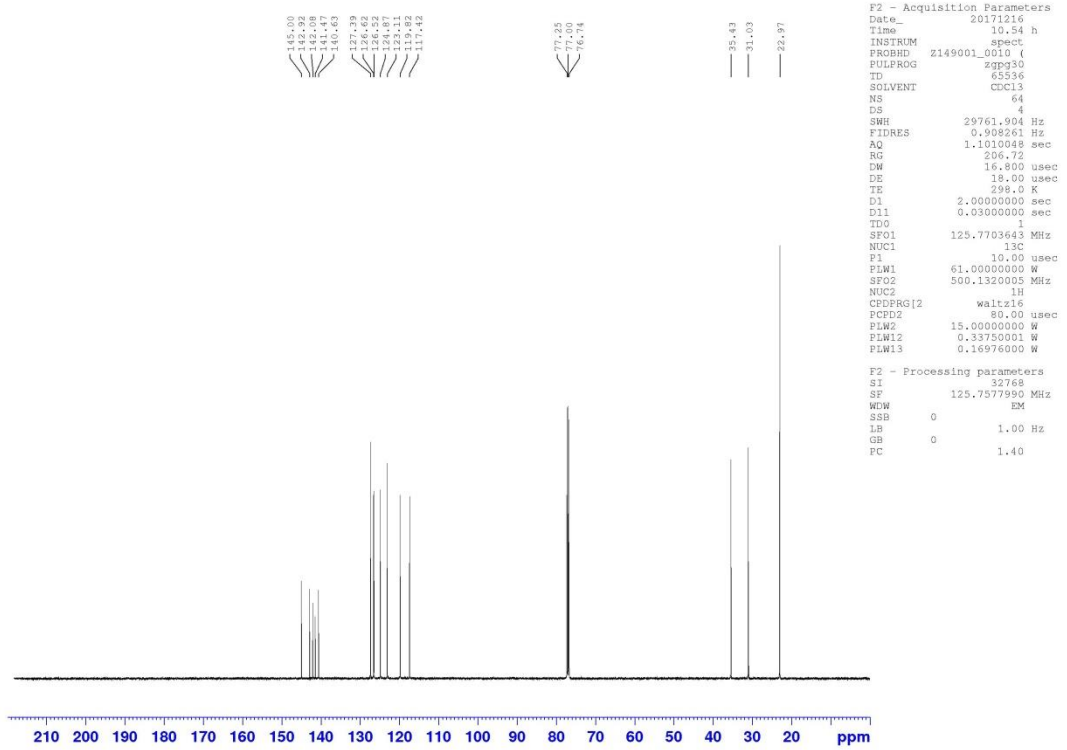


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TD                  65536
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NS                      64
DS                       4
SWH              29761.904 Hz
FIDRES               0.908261 Hz
AQ                1.1010048 sec
RG                   206.72
DW                  16.800 usec
DE                  18.00 usec
TE                  298.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      1
SFO1           125.7703643 MHz
NUC1                    13C
P1                  10.00 usec
PLW1            61.00000000 W
SFO2           500.1320005 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2               80.00 usec
PLW2            15.00000000 W
PLW12            0.33750001 W
PLW13            0.16976000 W

F2 - Processing parameters
SI                   32768
SF             125.7577990 MHz
WDW                     EM
SSB          0
LB                   1.00 Hz
GB           0
PC                   1.40
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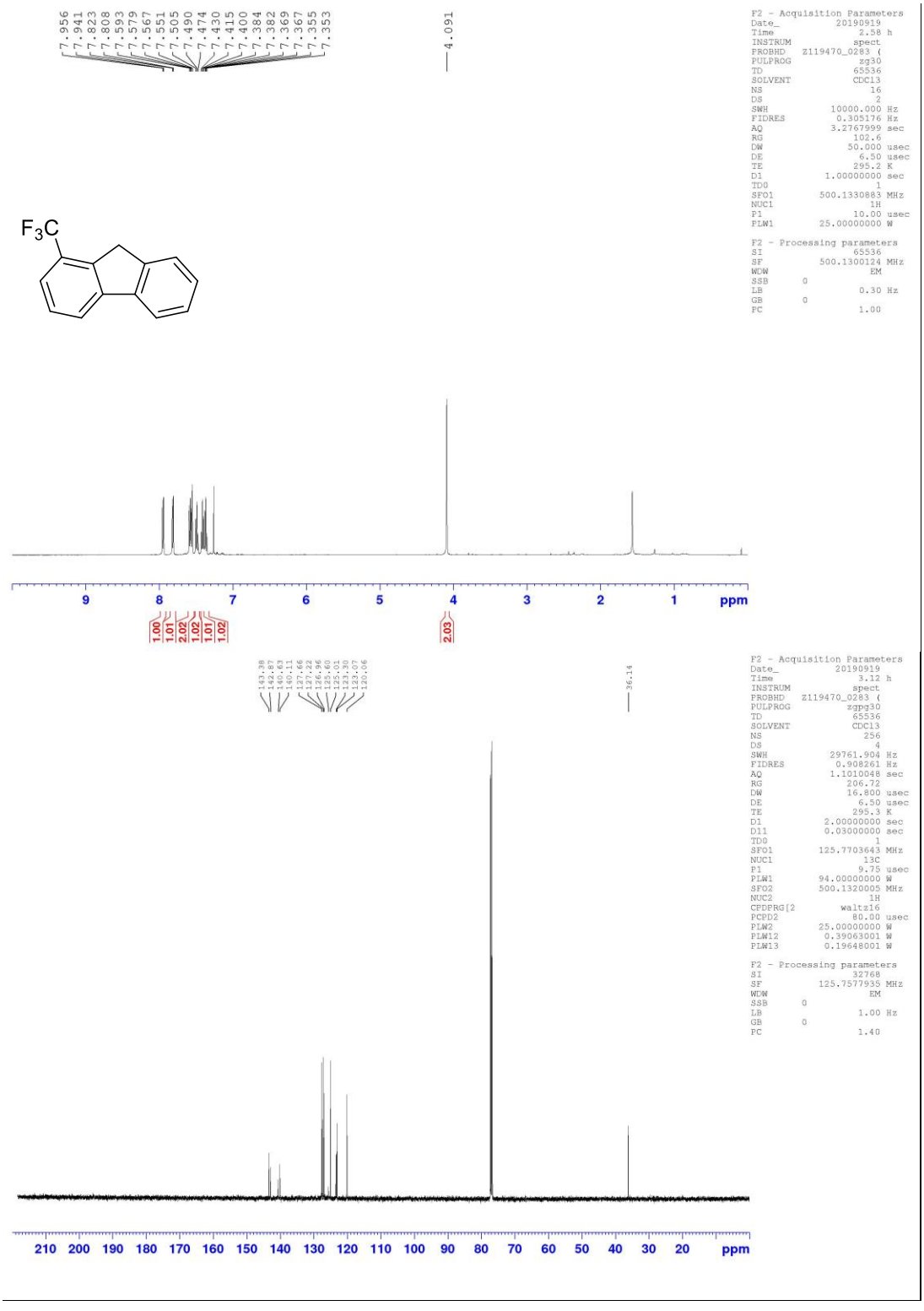
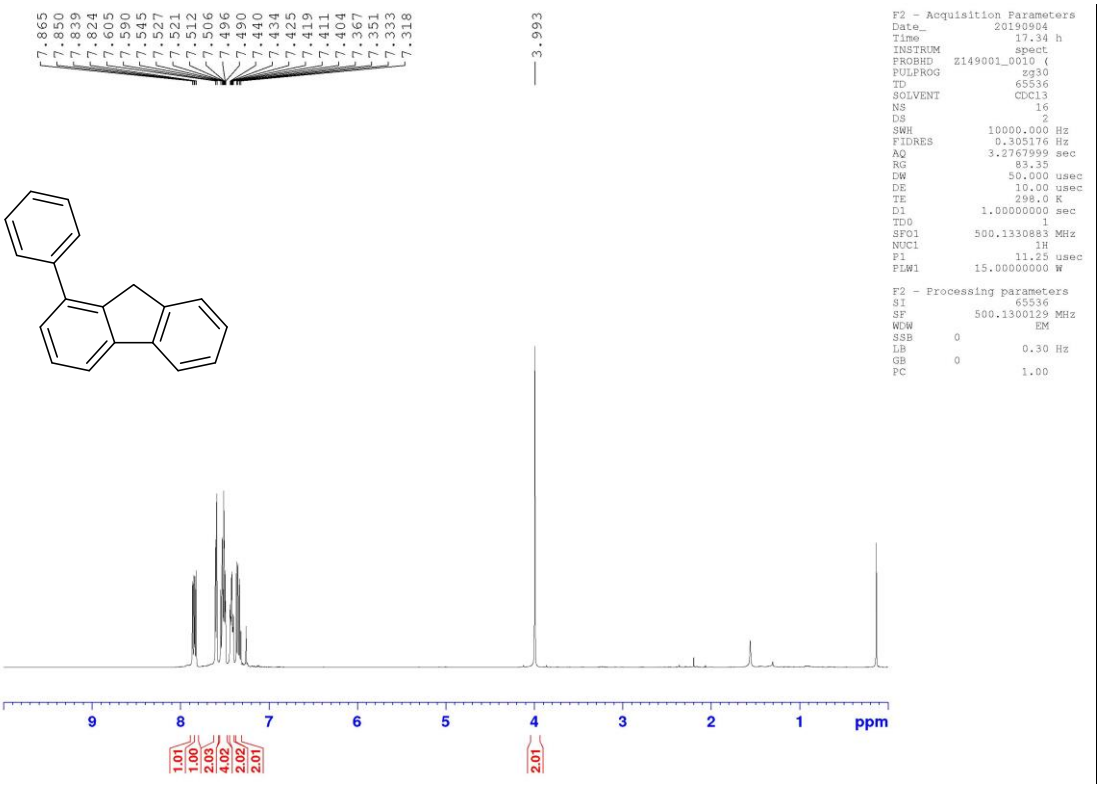
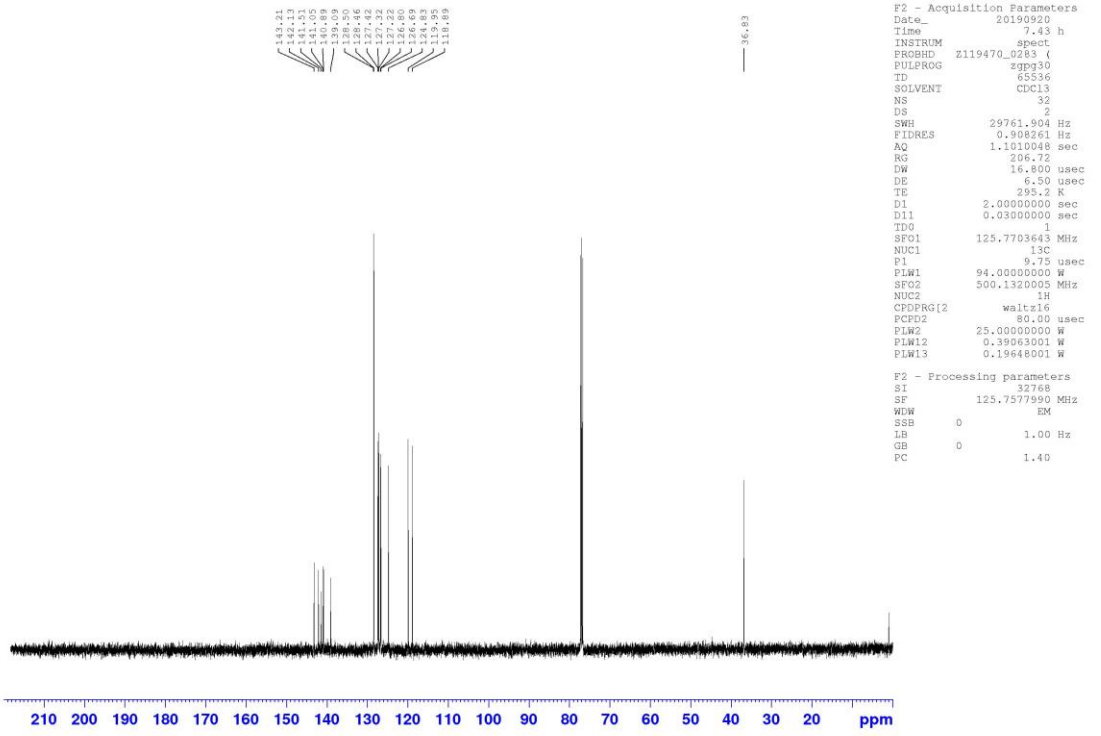
^{1}H NMR and ^{13}C NMR spectra for 1-Trifluoromethyl-9*H*-fluorene (Table 2, entry 3f)

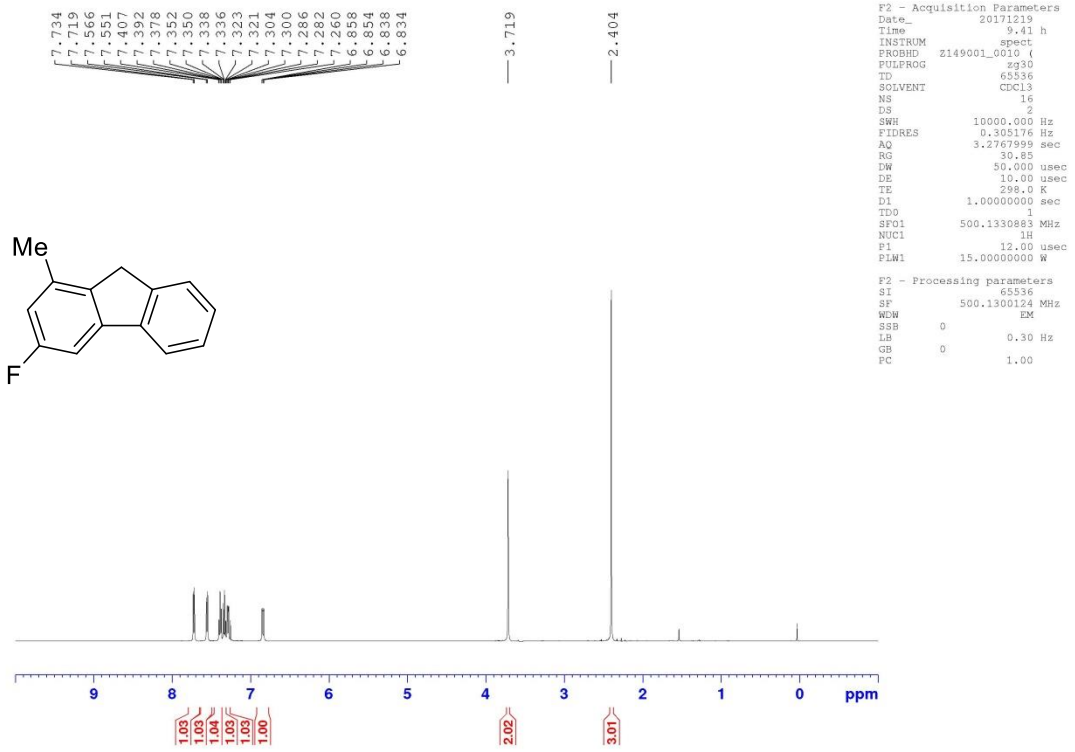


^{1}H NMR and ^{13}C NMR spectra for 1-Phenyl-9*H*-fluorene (Table 2, entry 3g)





^{1}H NMR and ^{13}C NMR spectra for 3-Fluoro-1-methyl-9*H*-fluorene (Table 2, entry 3h)

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AQ                3.2767999 sec
RG                    30.85
DW                   50.000 usec
DE                   10.00 usec
TE                   298.0 K
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TD0                      1
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NUC1                    1H
P1                   12.00 usec
PLW1           15.00000000 W

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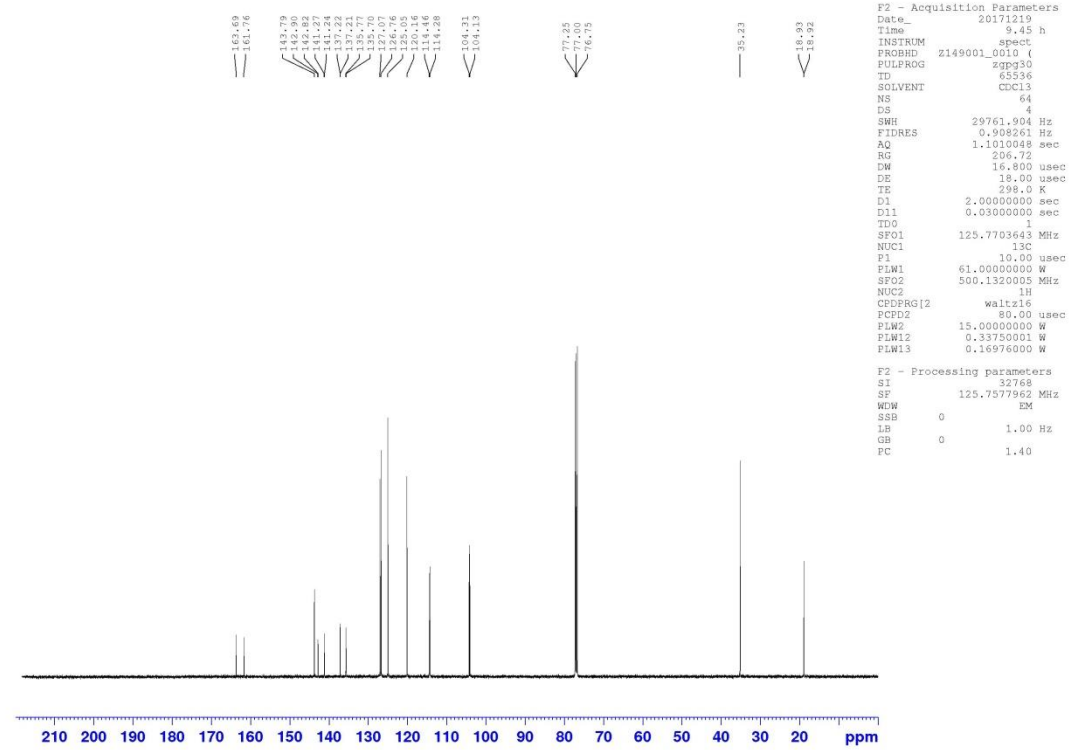


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FIDRES              0.908261 Hz
AQ                1.1010048 sec
RG                   206.72
DW                   16.800 usec
DE                   18.00 usec
TE                   298.0 K
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D11             0.03000000 sec
TD0                      1
SFO1           125.7703643 MHz
NUC1                    13C
P1                   10.00 usec
PLW1           61.00000000 W
SFO2           500.1320005 MHz
NUC2                    1H
CPDPRG[2            waltz16
PCPD2                80.00 usec
PLW2           15.00000000 W
PLW12           0.33750001 W
PLW13           0.16976000 W

F2 - Processing parameters
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WDW                     EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.40
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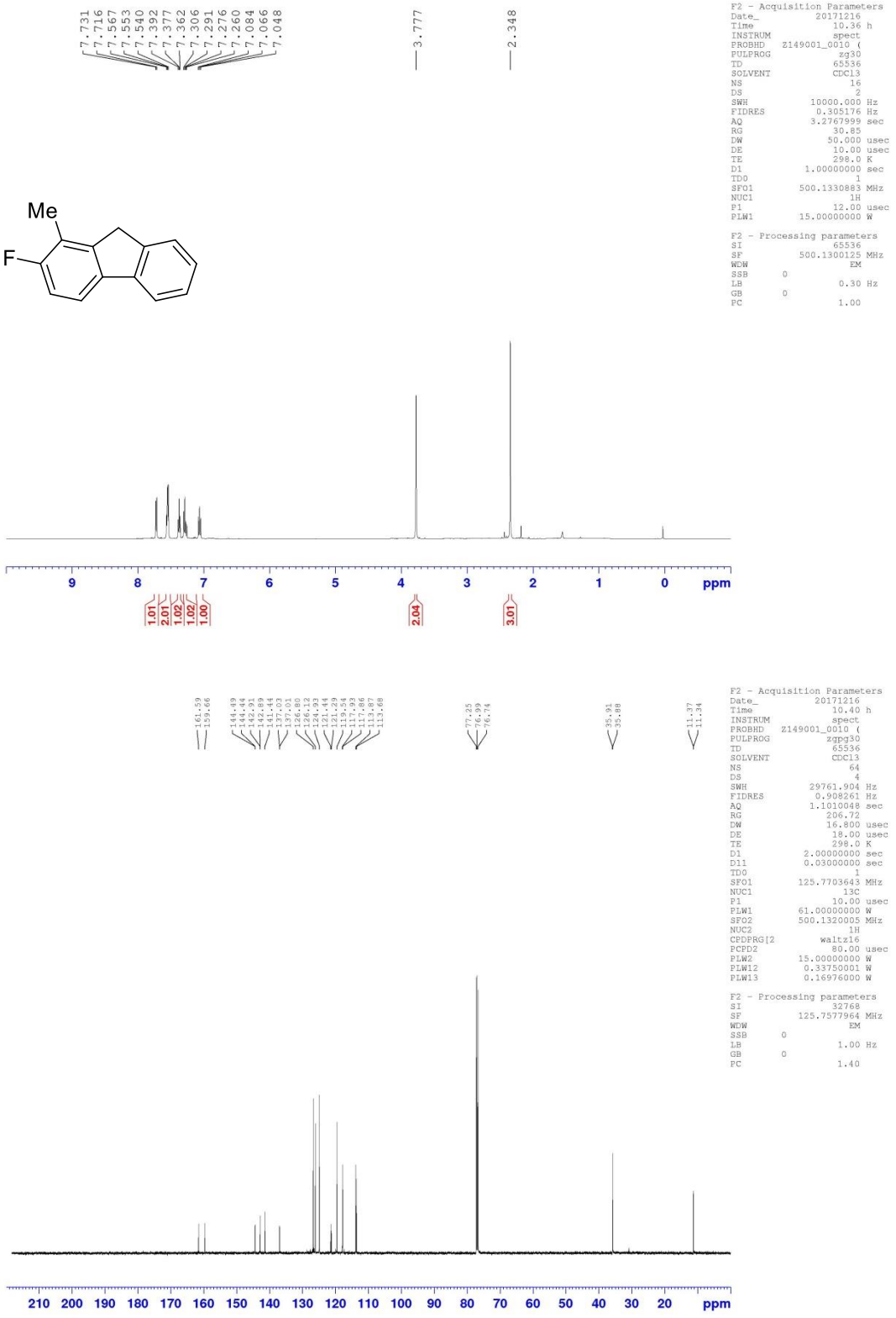
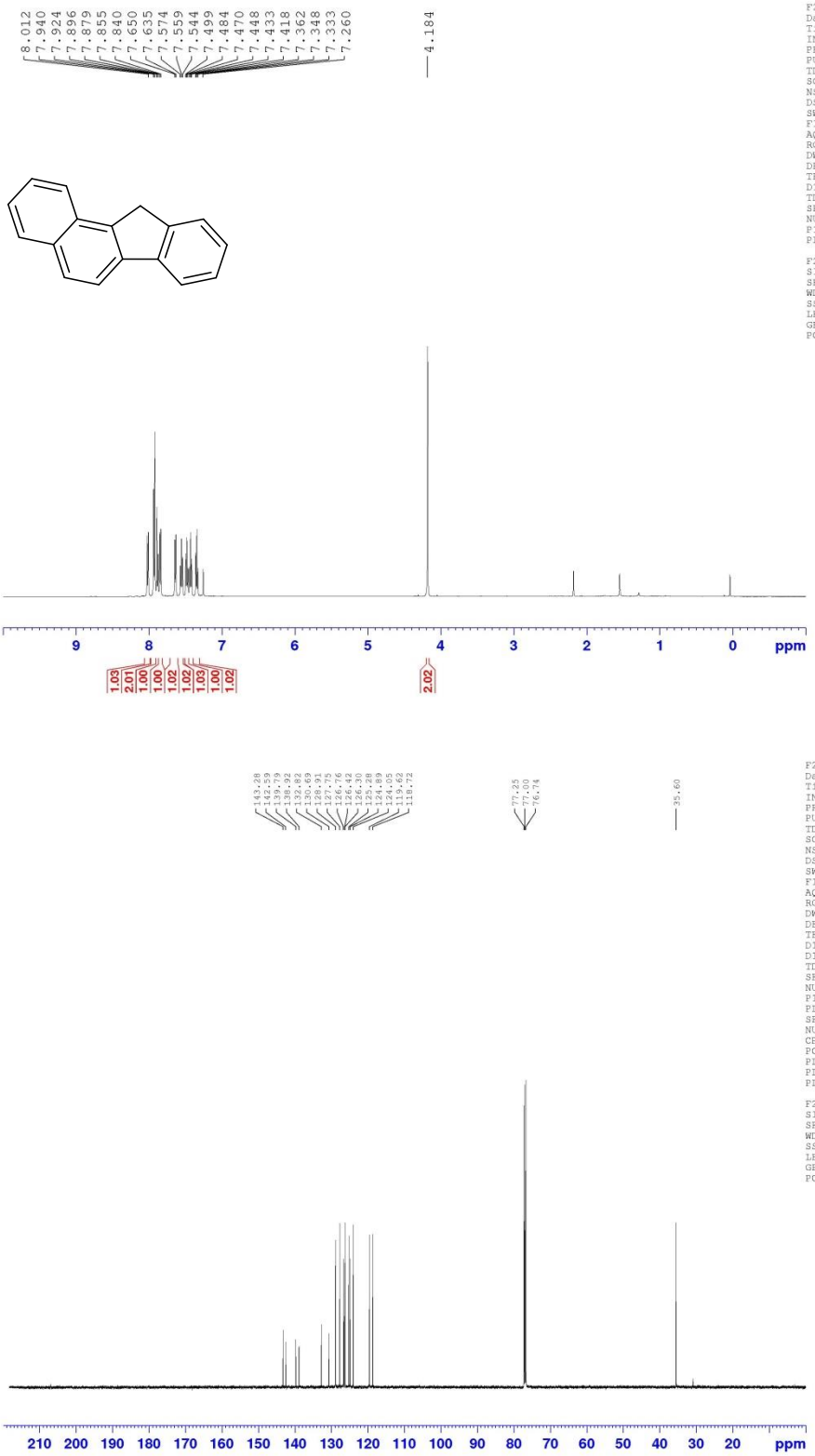
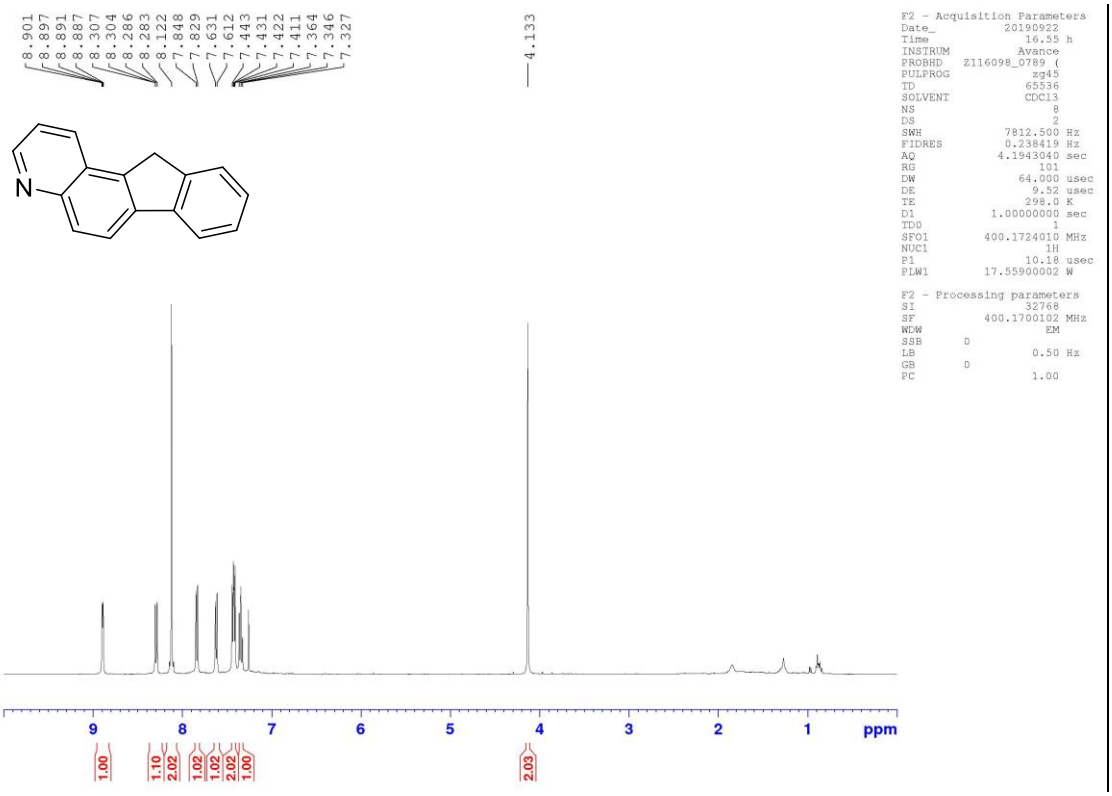
^{1}H NMR and ^{13}C NMR spectra for 2-Fluoro-1-methyl-9*H*-fluorene (Table 2, entry 3i)



^{1}H NMR and ^{13}C NMR spectra for 11*H*-Benzo[*a*]fluorene (Table 2, entry 3j)



^{1}H NMR and ^{13}C NMR spectra for 11*H*-Indeno[2,1-*f*]quinoline (Table 2, entry 3k)

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DS                        2
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AQ                4.1943040 sec
RG                      101
DW                   64.000 usec
DE                    9.52 usec
TE                   298.0 K
D1              1.00000000 sec
TD0                       1
SFO1            400.1724010 MHz
NUC1                     1H
P1                   10.18 usec
PLW1            17.55900002 W

F2 - Processing parameters
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WDW                      EM
SSB          0
LB                    0.50 Hz
GB           0
PC                    1.00
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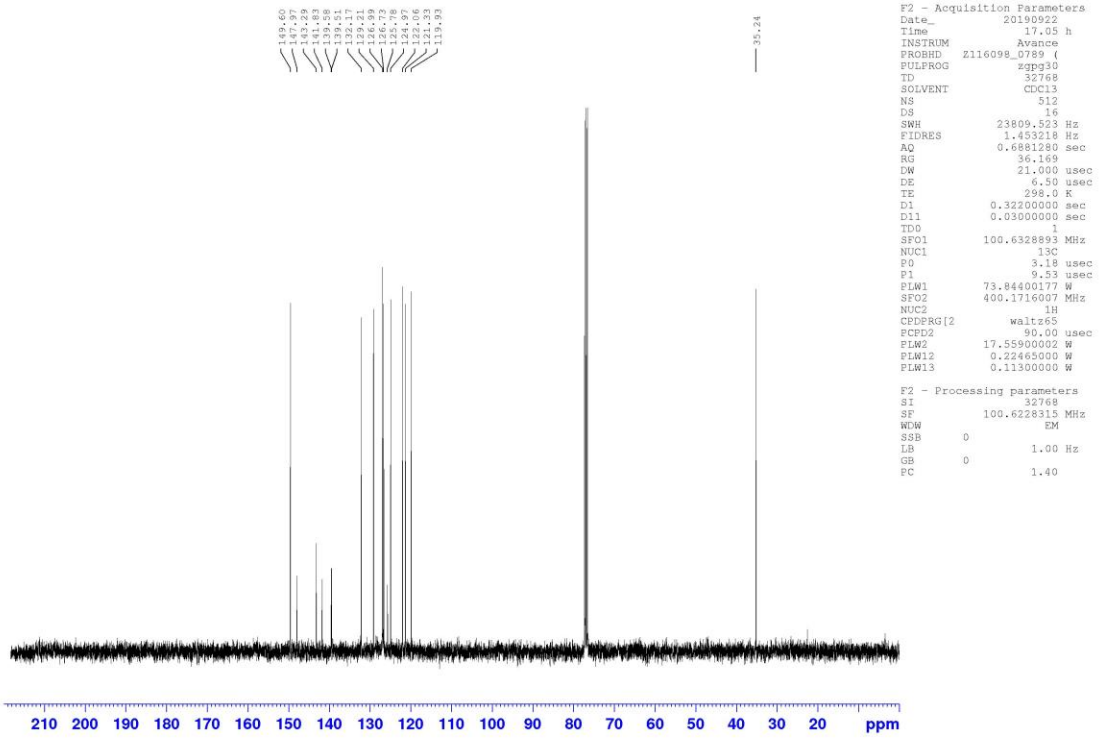


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PULPROG              zgpg30
TD                    32768
SOLVENT               CDCl3
NS                      512
DS                       16
SWH              23809.523 Hz
FIDRES               1.453218 Hz
AQ                0.6881280 sec
RG                    36.169
DW                   21.000 usec
DE                    6.50 usec
TE                   298.0 K
D1              0.32200000 sec
D11             0.03000000 sec
TD0                       1
SFO1            100.6328893 MHz
NUC1                     13C
P0                    3.18 usec
P1                    9.53 usec
PLW1            73.84400177 W
SFO2            400.1716007 MHz
NUC2                     1H
CPDPRG[2              waltz65
PCPD2                90.00 usec
PLW2            17.55900002 W
PLW12            0.22465000 W
PLW13            0.11300000 W

F2 - Processing parameters
SI                    32768
SF              100.6228315 MHz
WDW                      EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.40
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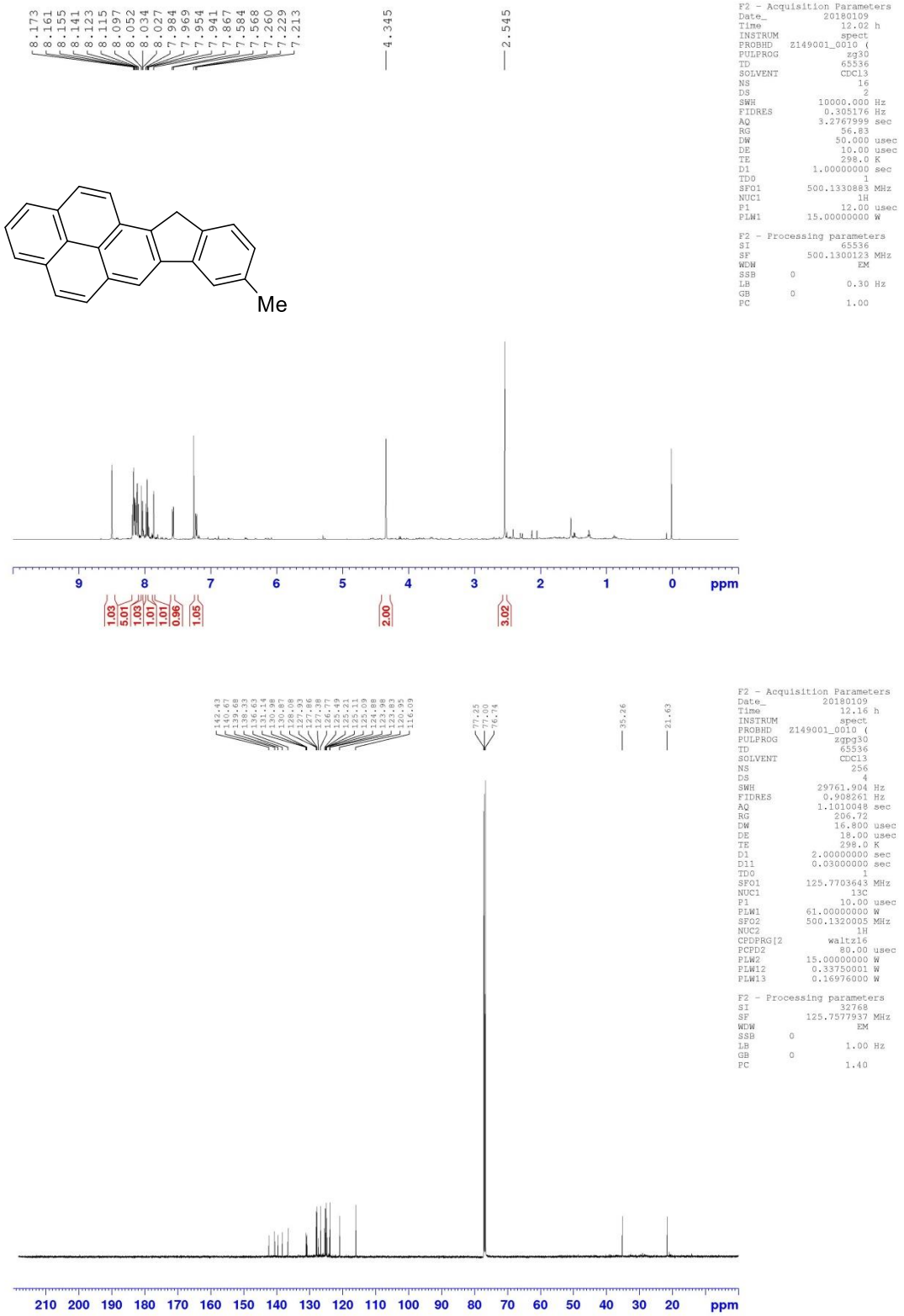
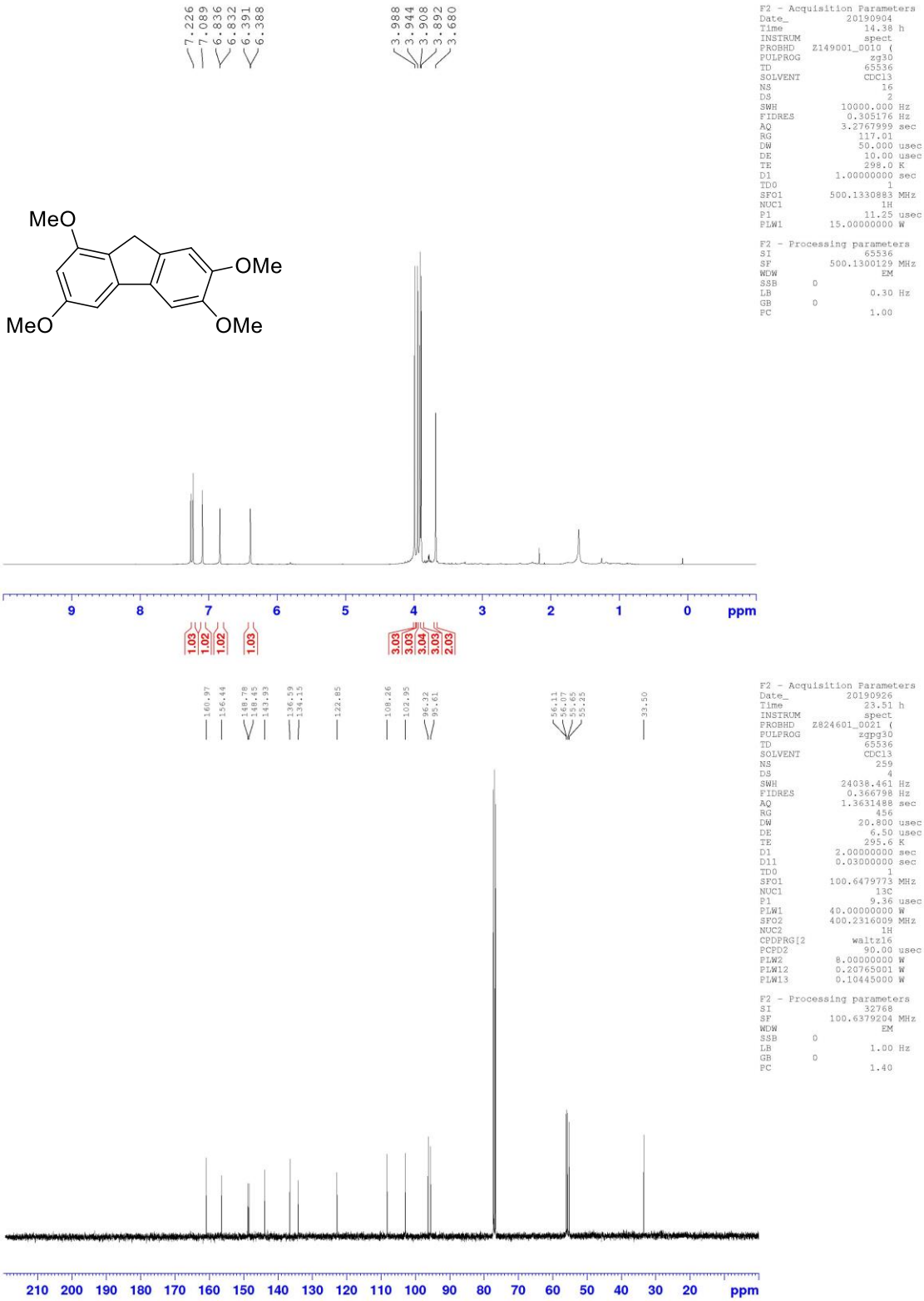
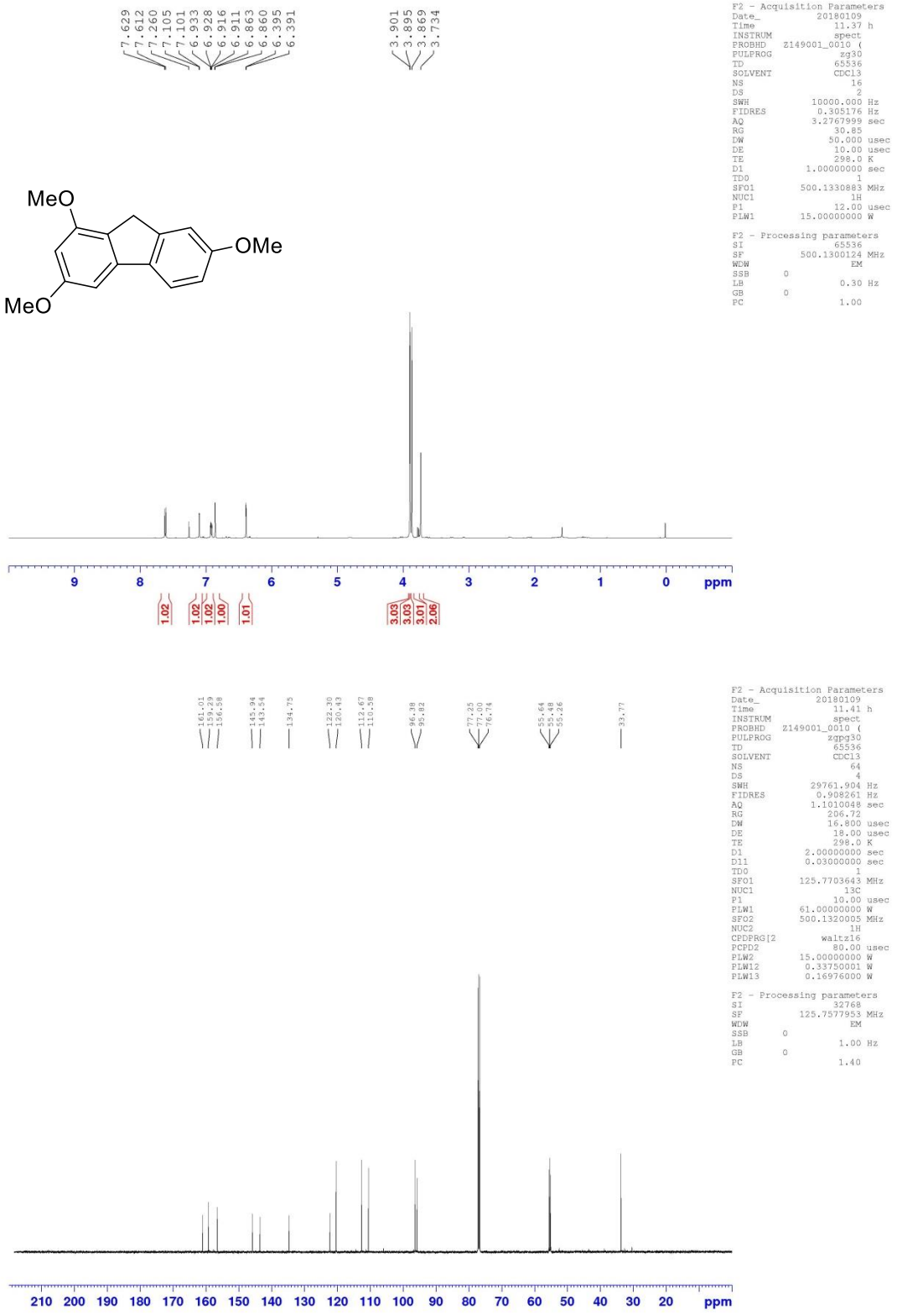
^{1}H NMR and ^{13}C NMR spectra for 11*H*-Indeno[2,1-*a*]pyrene (Table 2, entry 3l)



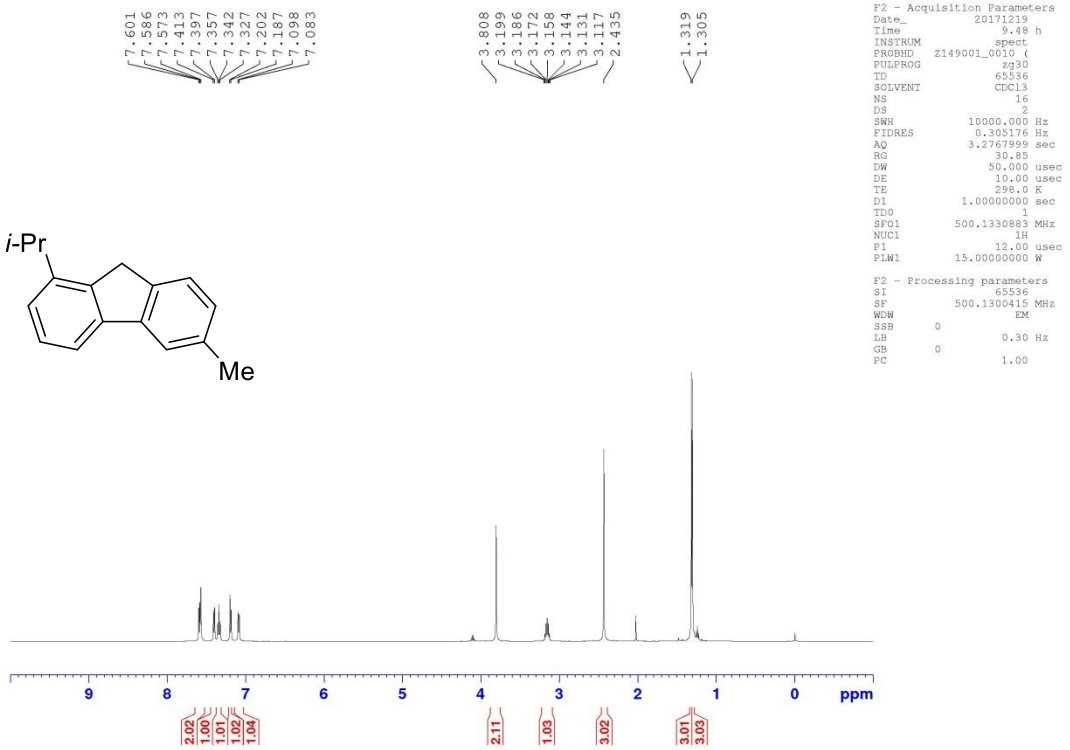
^{1}H NMR and ^{13}C NMR spectra for 1,3,6,7-Tetramethoxy-9*H*-fluorene (Table 2, entry 3m)



^{1}H NMR and ^{13}C NMR spectra for 1,3,7-Trimethoxy-9*H*-fluorene (Table 2, entry 3n)



^{1}H NMR and ^{13}C NMR spectra for 1-Isopropyl-6-methyl-9*H*-fluorene (Table 2, entry 3o)

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F2 - Acquisition Parameters
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PROBHD    Z149001_0010 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               10000.000 Hz
FIDRES                0.305176 Hz
AQ                3.2767999 sec
RG                    30.85
DW                    50.000 usec
DE                    10.00 usec
TE                    298.0 K
D1                1.00000000 sec
TD0                       1
SFO1           500.1330883 MHz
NUC1                     1H
P1                    12.00 usec
PLW1             15.00000000 W

F2 - Processing parameters
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SF             500.1300415 MHz
WDW                      EM
SSB                   0
LB                    0.30 Hz
GB                    0
PC                    1.00
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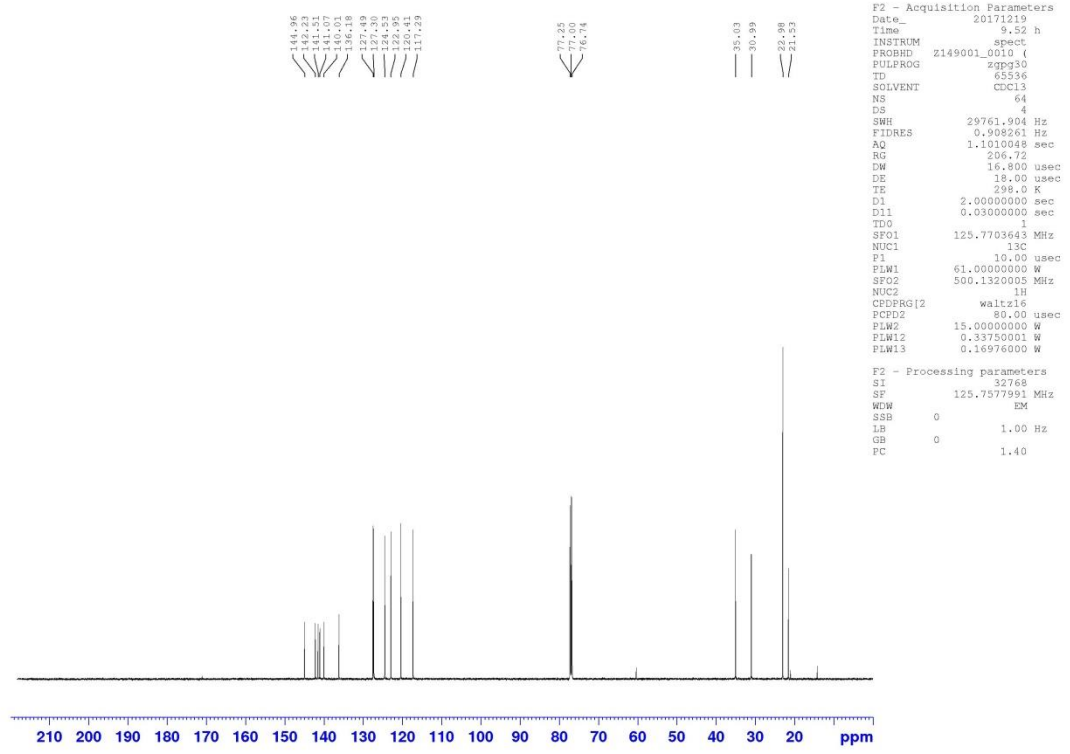


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F2 - Acquisition Parameters
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Time                  9.52 h
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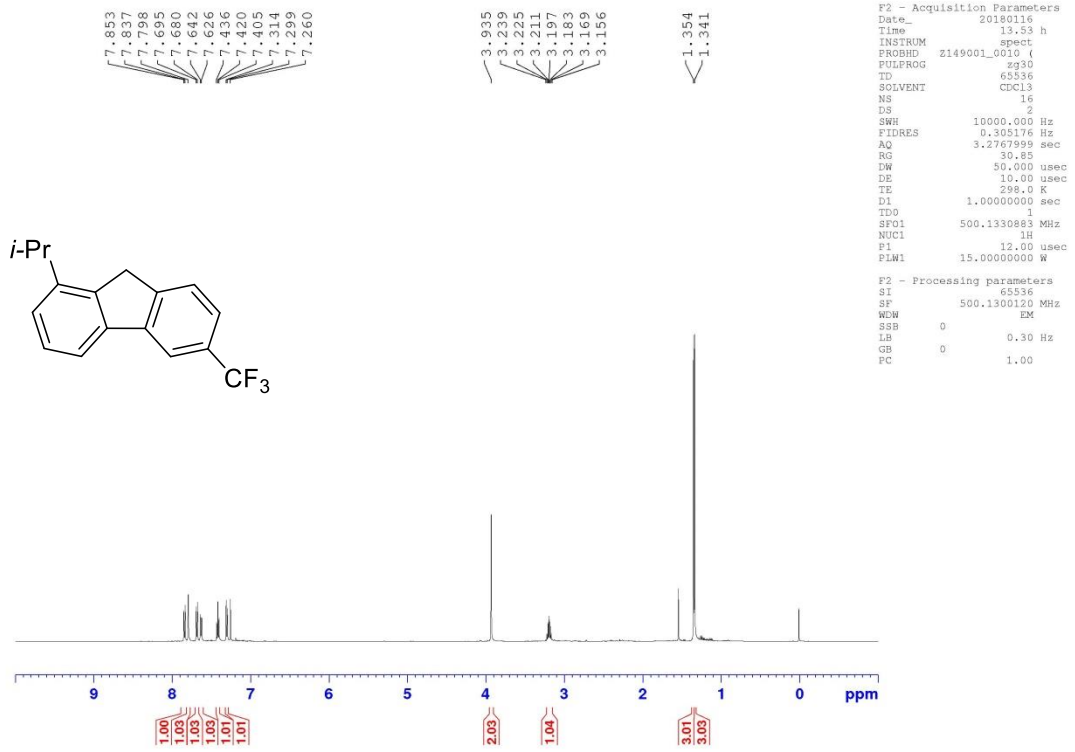
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       64
DS                        4
SWH               29761.904 Hz
FIDRES                0.908261 Hz
AQ                1.1010048 sec
RG                    206.72
DW                    16.800 usec
DE                    18.00 usec
TE                    298.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1
SFO1           125.7703643 MHz
NUC1                     13C
P1                    10.00 usec
PLW1             61.00000000 W
SFO2           500.1320005 MHz
NUC2                     1H
CPDPRG[2             waltz16
PCPD2                 80.00 usec
PLW2             15.00000000 W
PLW12             0.33750001 W
PLW13             0.16976000 W

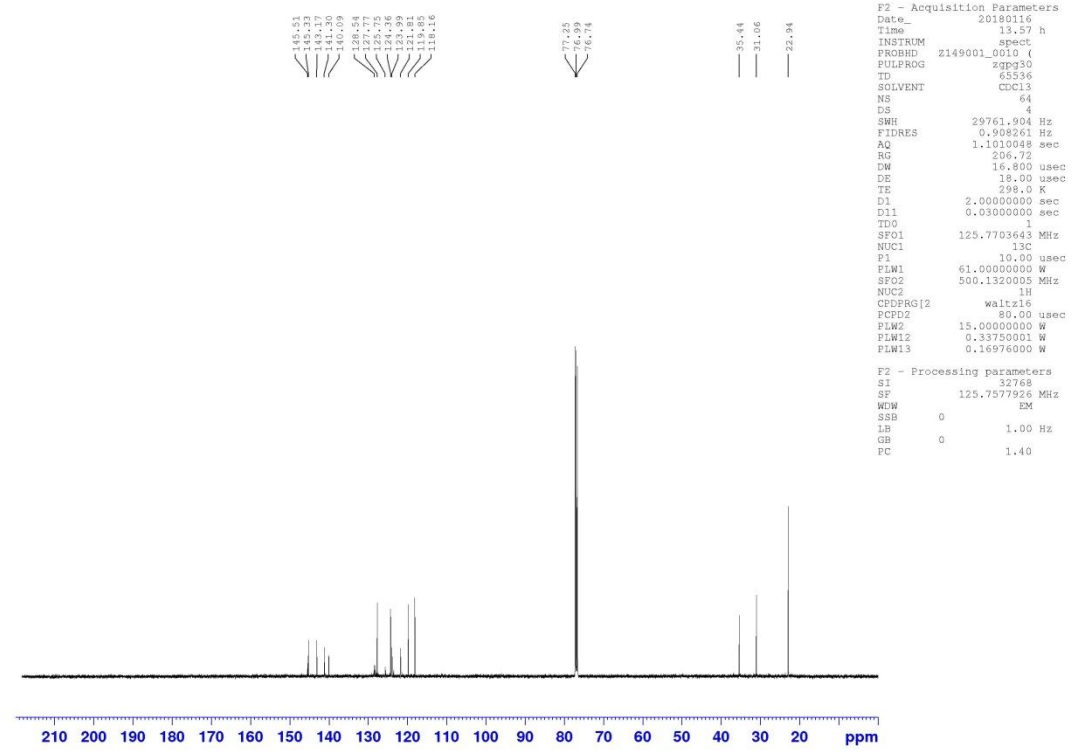
F2 - Processing parameters
SI                    32768
SF             125.7577991 MHz
WDW                      EM
SSB                   0
LB                    1.00 Hz
GB                    0
PC                    1.40
```

^{1}H NMR and ^{13}C NMR spectra for 1-Isopropyl-6-(trifluoromethyl)-9*H*-fluorene (Table 2, entry 3p)




```
F2 - Acquisition Parameters
Date_              20180116
Time                 13.53 h
INSTRUM              spect
PROBHD    Z149001_0010 (
PULPROG              zg30
TD                  65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH              10000.000 Hz
FIDRES           0.305176 Hz
AQ               3.2767999 sec
RG                   30.85
DW                 50.000 usec
DE                 10.00 usec
TE                 298.0 K
D1              1.00000000 sec
TD0                      1
SFO1          500.1330883 MHz
NUC1                    1H
P1                 12.00 usec
PLW1           15.00000000 W

F2 - Processing parameters
SI                   65536
SF            500.1300120 MHz
WDW                     EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

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F2 - Acquisition Parameters
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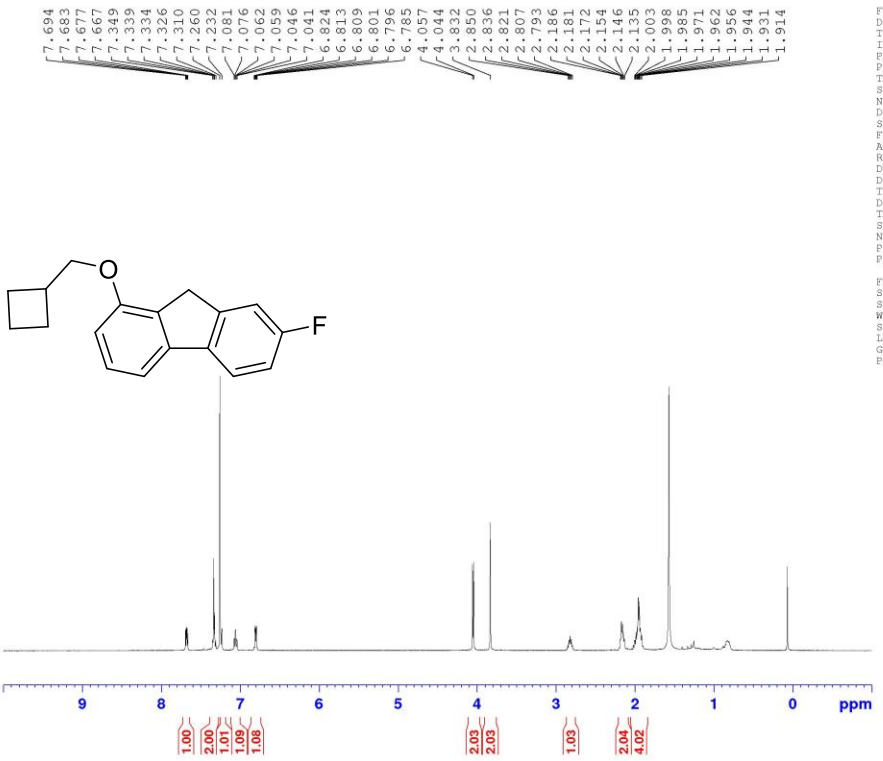
Time                 13.57 h
INSTRUM              spect
PROBHD    Z149001_0010 (
PULPROG            zgpg30
TD                  65536
SOLVENT              CDCl3
NS                      64
DS                       4
SWH              29761.904 Hz
FIDRES           0.908261 Hz
AQ               1.1010048 sec
RG                  206.72
DW                 16.800 usec
DE                 18.00 usec
TE                 298.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1
SFO1          125.7703643 MHz
NUC1                    13C
P1                 10.00 usec
PLW1           61.00000000 W
SFO2          500.1320005 MHz
NUC2                    1H
CPDPRG[2            waltz16
PCPD2              80.00 usec
PLW2           15.00000000 W
PLW12           0.33750001 W
PLW13           0.16976000 W

F2 - Processing parameters
SI                   32768
SF            125.7577926 MHz
WDW                     EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

^{1}H NMR and ^{13}C NMR spectra for 1-(Cyclobutylmethoxy)-7-fluoro-9*H*-fluorene (Table 2, entry 3q)

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Time                   7.33 h
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PROBHD    Z119470_0283 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               10000.000 Hz
FIDRES                0.305176 Hz
AQ                3.2767999 sec
RG                      142.5
DW                   50.000 usec
DE                    6.50 usec
TE                    295.1 K
D1                1.00000000 sec
TD0                       1
SFO1           500.1330883 MHz
NUC1                     1H
P1                   10.00 usec
PLW1          25.00000000 W

F2 - Processing parameters
SI                    65536
SF             500.1300129 MHz
WDW                      EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    1.00
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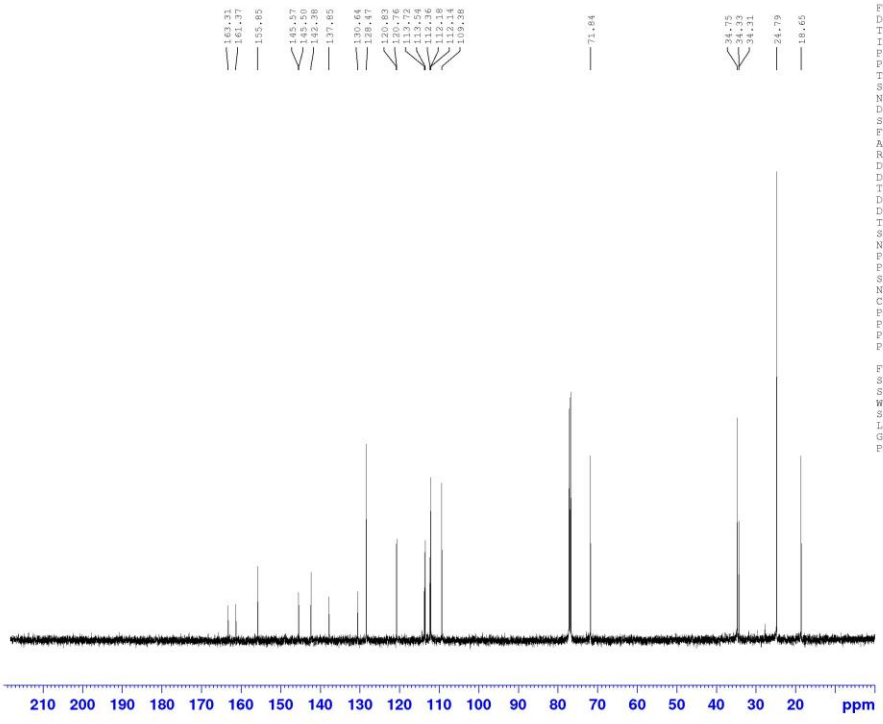


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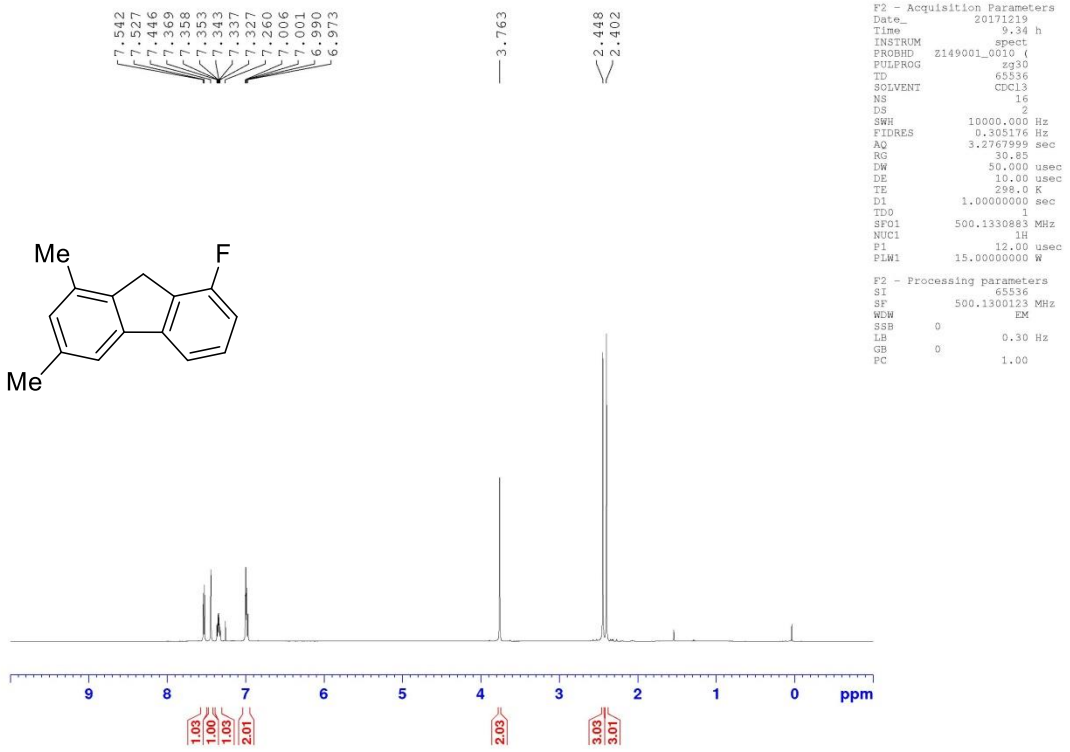
F2 - Acquisition Parameters
Date_              20190926
Time                   8.06 h
INSTRUM               spect
PROBHD    Z119470_0283 (
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       46
DS                        4
SWH               29761.904 Hz
FIDRES                0.908261 Hz
AQ                1.1010048 sec
RG                    206.72
DW                   16.800 usec
DE                    6.50 usec
TE                    295.1 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1
SFO1           125.7703643 MHz
NUC1                     13C
P1                    9.75 usec
PLW1          94.00000000 W
SFO2           500.1320005 MHz
NUC2                     1H
CPDPRG[2             waltz16
PCPD2                80.00 usec
PLW2          25.00000000 W
PLW12          0.39063001 W
PLW13          0.19648001 W

F2 - Processing parameters
SI                    32768
SF             125.7577991 MHz
WDW                      EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.40
```

^{1}H NMR and ^{13}C NMR spectra for 8-Fluoro-1,3-dimethyl-9*H*-fluorene (Table 2, entry 3r)

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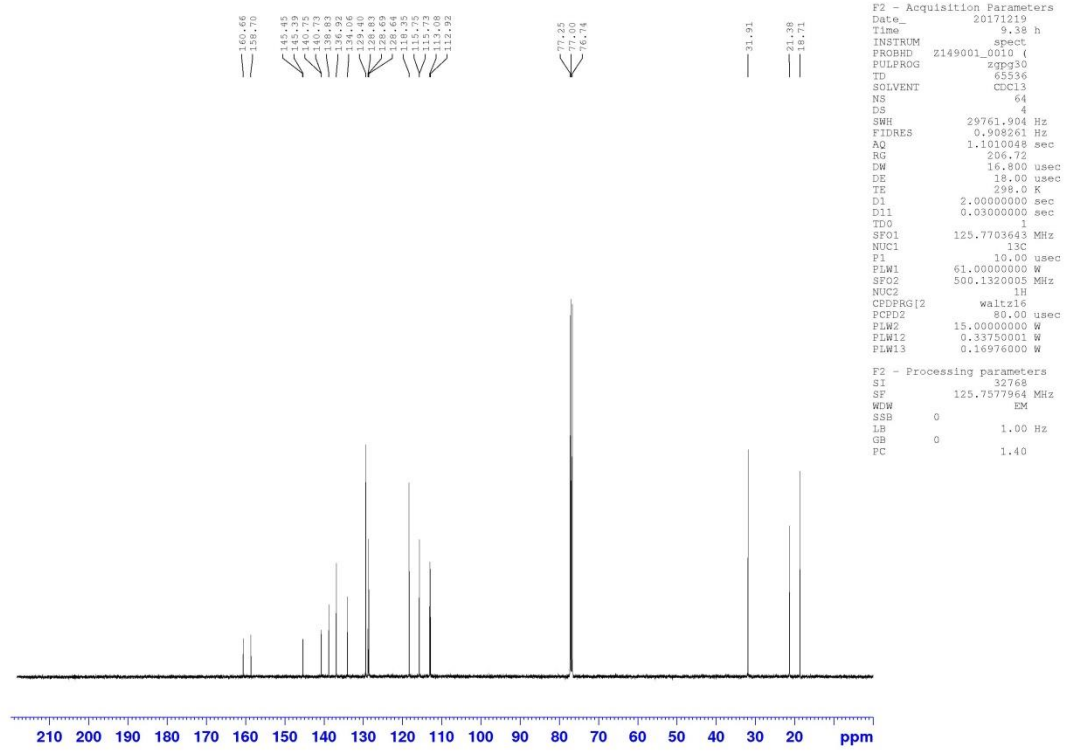
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               10000.000 Hz
FIDRES                0.305176 Hz
AQ                3.2767999 sec
RG                    30.85
DW                    50.000 usec
DE                    10.00 usec
TE                    298.0 K
D1                1.00000000 sec
TD0                       1
SFO1           500.1330883 MHz
NUC1                     1H
P1                    12.00 usec
PLW1           15.00000000 W

F2 - Processing parameters
SI                    65536
SF             500.1300123 MHz
WDW                      EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    1.00
```

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F2 - Acquisition Parameters
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Time                  9.38 h
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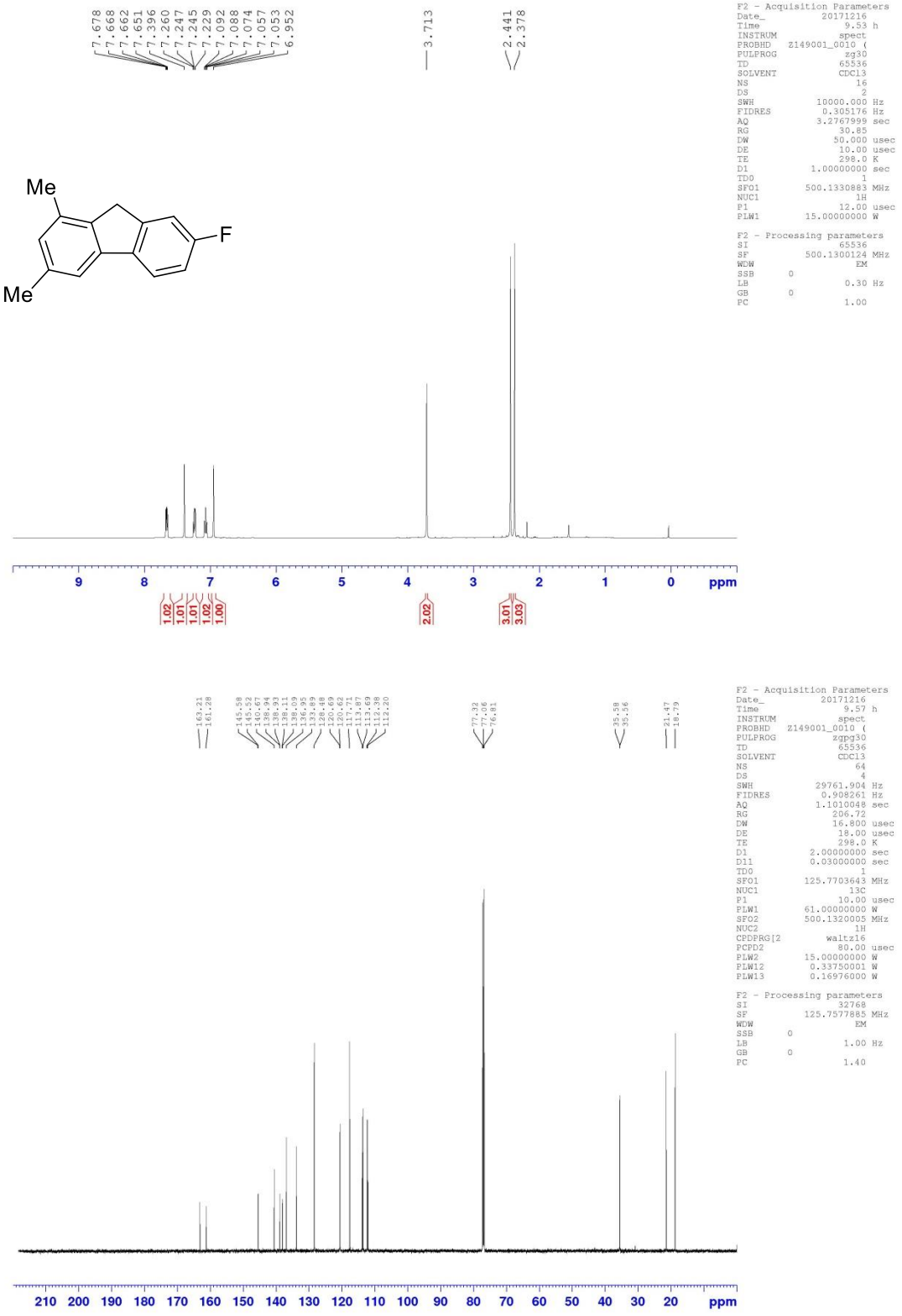
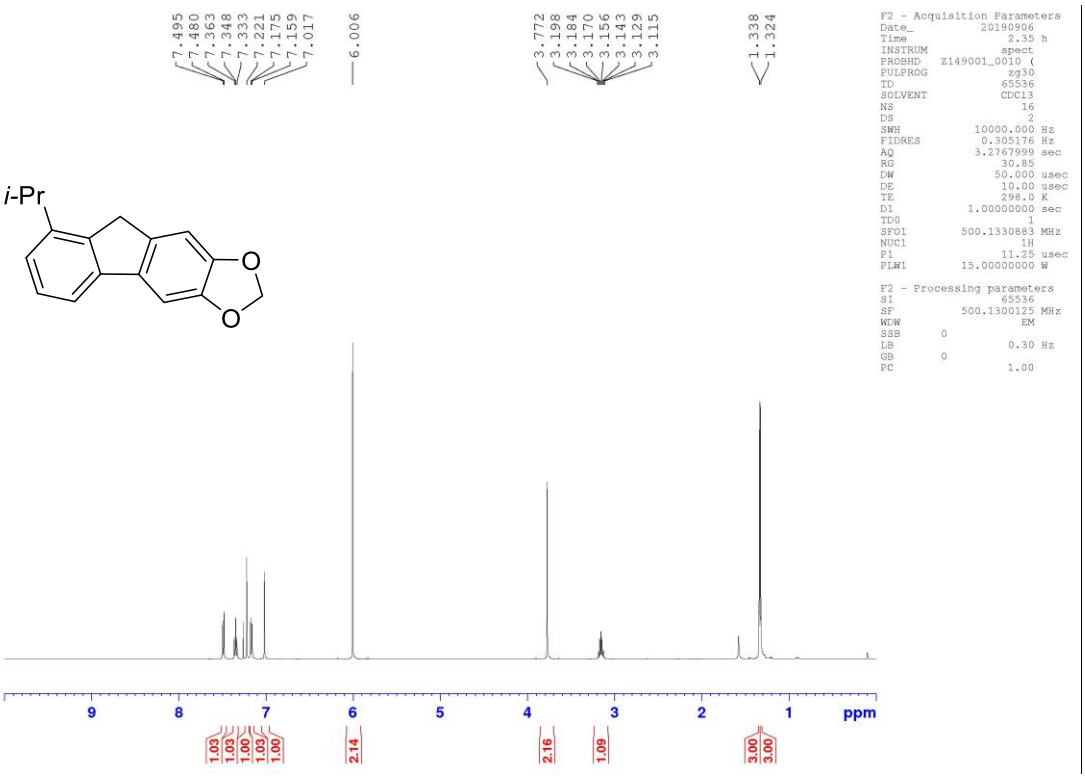
PROBHD    Z149001_0010 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       64
DS                        4
SWH               29761.904 Hz
FIDRES                0.908261 Hz
AQ                1.1010048 sec
RG                    206.72
DW                    16.800 usec
DE                    18.00 usec
TE                    298.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1
SFO1           125.7703643 MHz
NUC1                     13C
P1                    10.00 usec
PLW1           61.00000000 W
SFO2           500.1320005 MHz
NUC2                     1H
CPDPRG[2             waltz16
PCPD2                 80.00 usec
PLW2           15.00000000 W
PLW12           0.33750001 W
PLW13           0.16976000 W

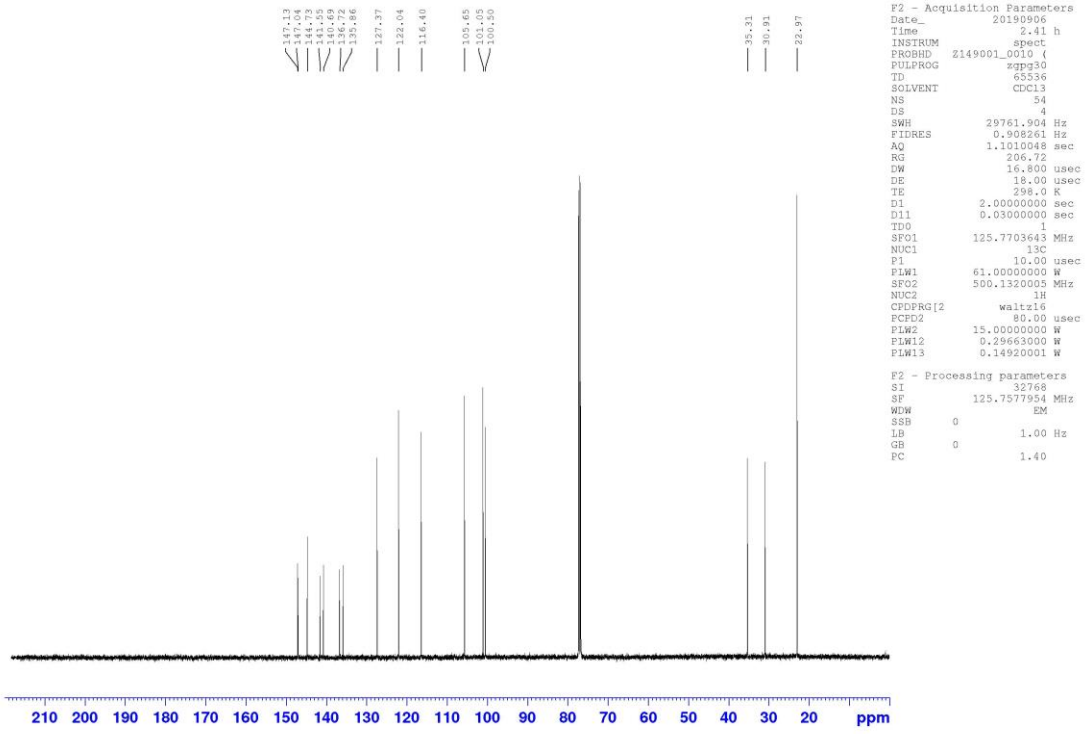
F2 - Processing parameters
SI                    32768
SF             125.7577964 MHz
WDW                      EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.40
```

^{1}H NMR and ^{13}C NMR spectra for 7-Fluoro-1,3-dimethyl-9*H*-fluorene (Table 2, entry 3s)

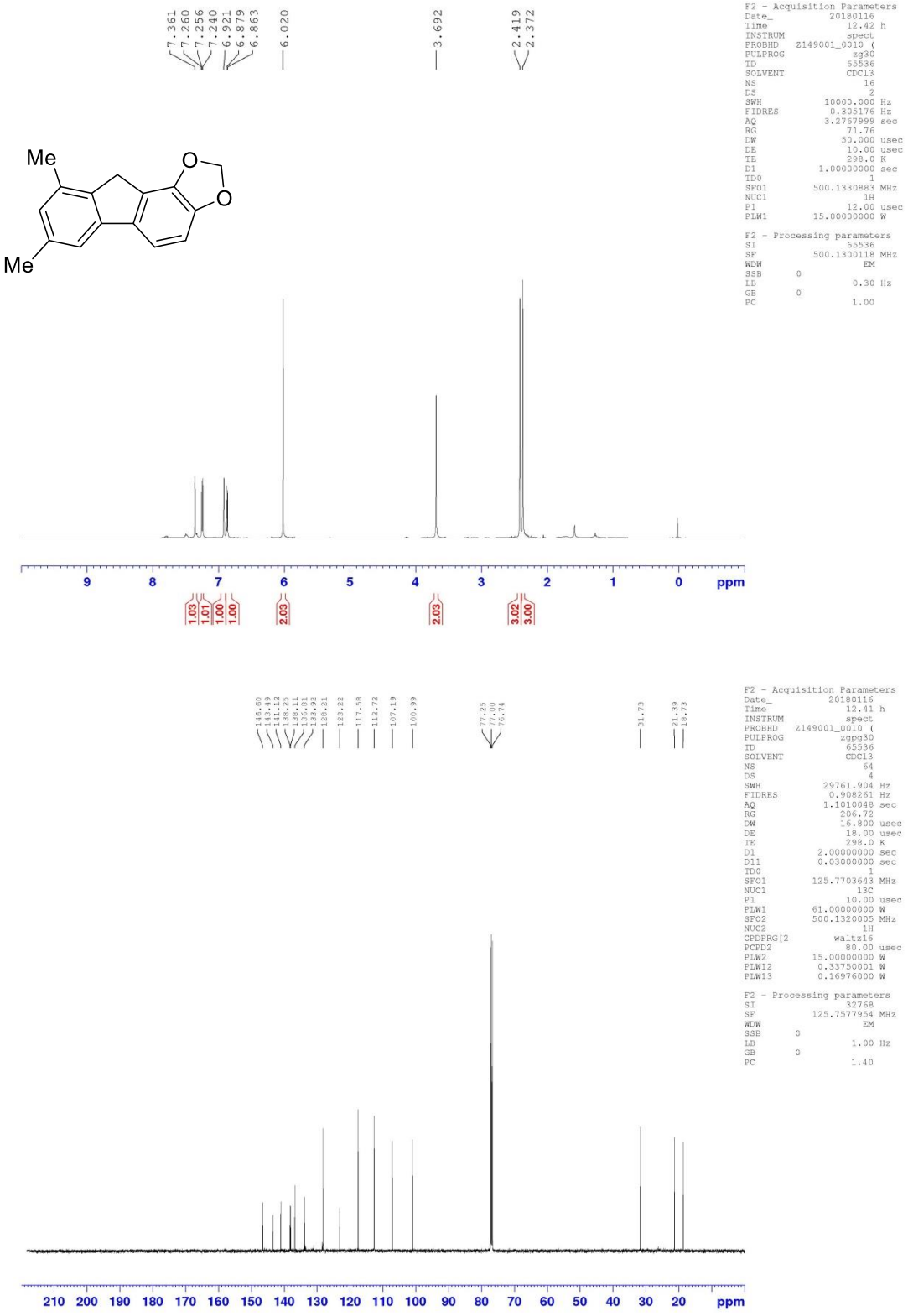


^{1}H NMR and ^{13}C NMR spectra for 8-Isopropyl-9*H*-fluoreno[2,3-*d*][1,3]dioxole (Table 2, entry 3t)

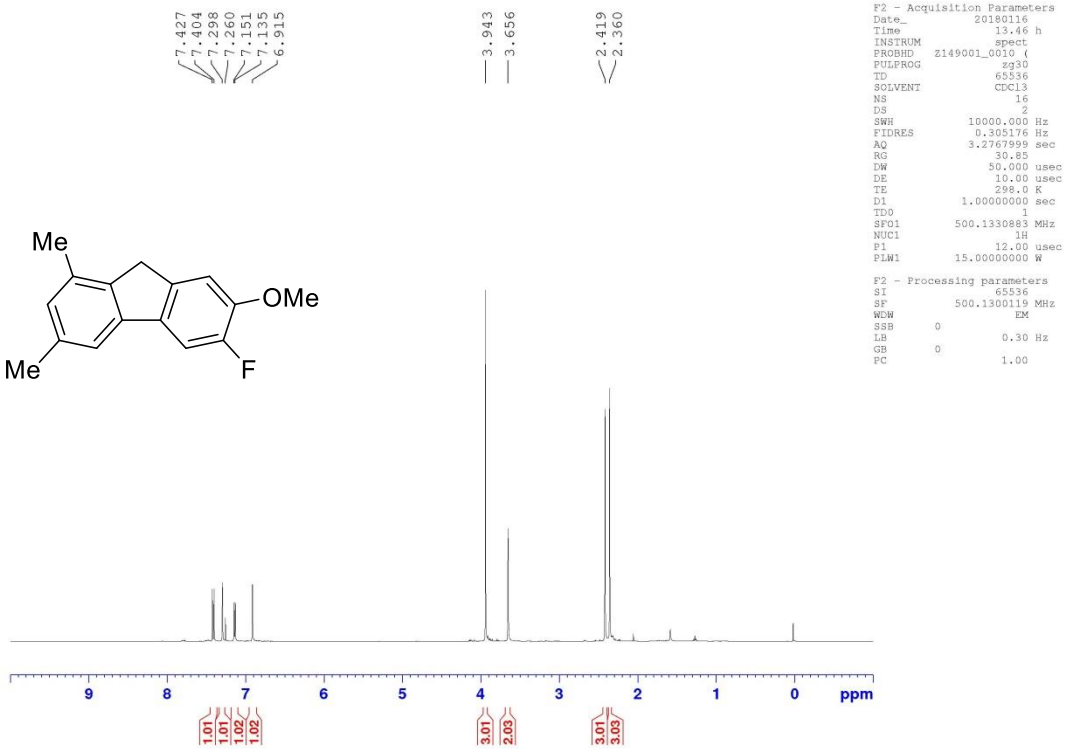


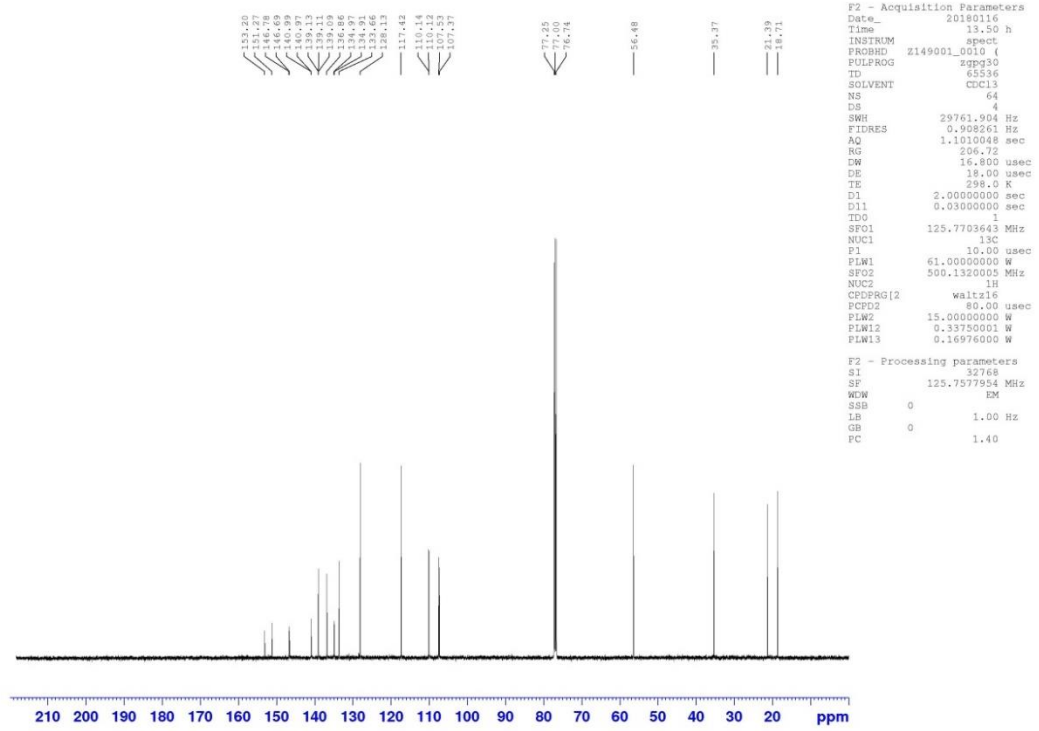


^{1}H NMR and ^{13}C NMR spectra for 7,9-Dimethyl-10*H*-fluoreno[1,2-*d*][1,3]dioxole (Table 2, entry 3u)

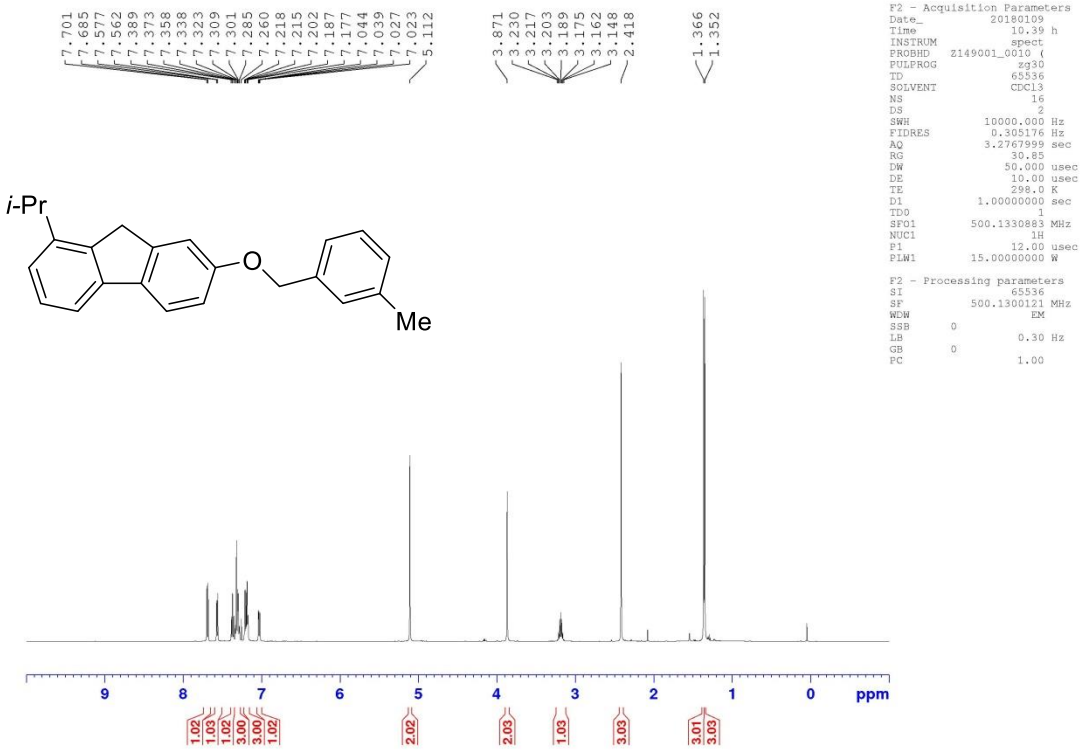


^{1}H NMR and ^{13}C NMR spectra for 6-Fluoro-7-methoxy-1,3-dimethyl-9*H*-fluorene (Table 2, entry 3v)





^{1}H NMR and ^{13}C NMR spectra for 1-Isopropyl-7-((3-methylbenzyl)oxy)-9*H*-fluorene (Table 2, entry 3w)

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PULPROG              zg30
TD                  65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH              10000.000 Hz
FIDRES               0.305176 Hz
AQ                3.2767999 sec
RG                    30.85
DW                   50.000 usec
DE                   10.00 usec
TE                   298.0 K
D1             1.00000000 sec
TD0                      1
SFO1          500.1330883 MHz
NUC1                    1H
P1                   12.00 usec
PLW1          15.00000000 W

F2 - Processing parameters
SI                   65536
SF            500.1300121 MHz
WDW                     EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    1.00
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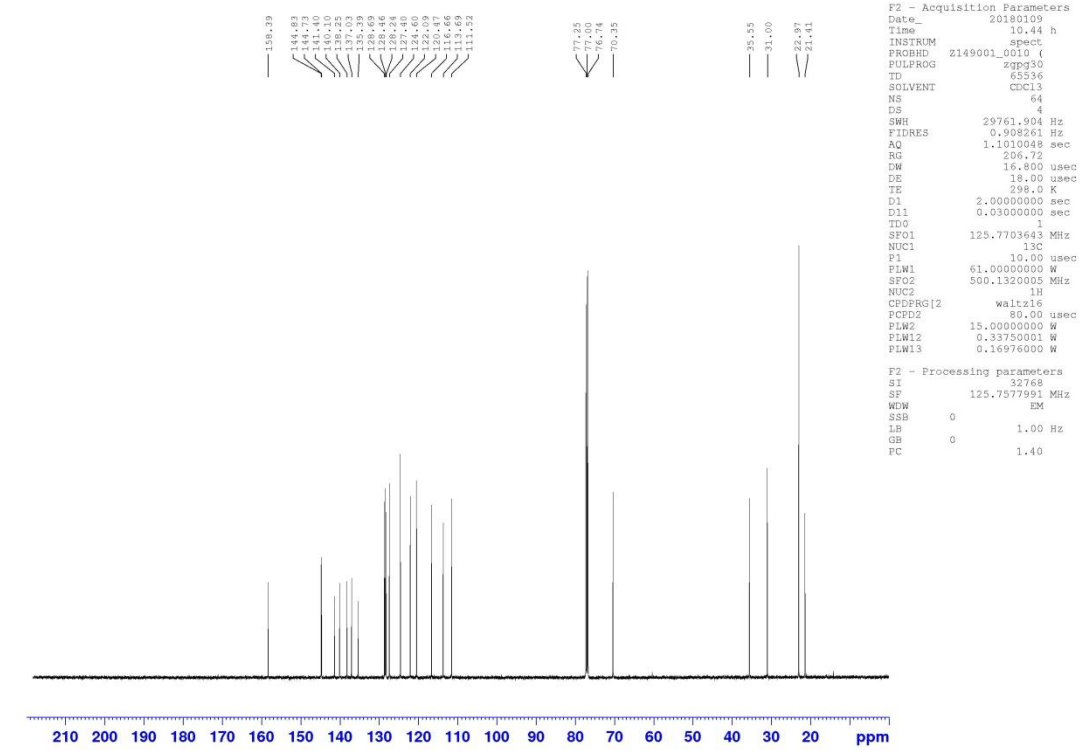


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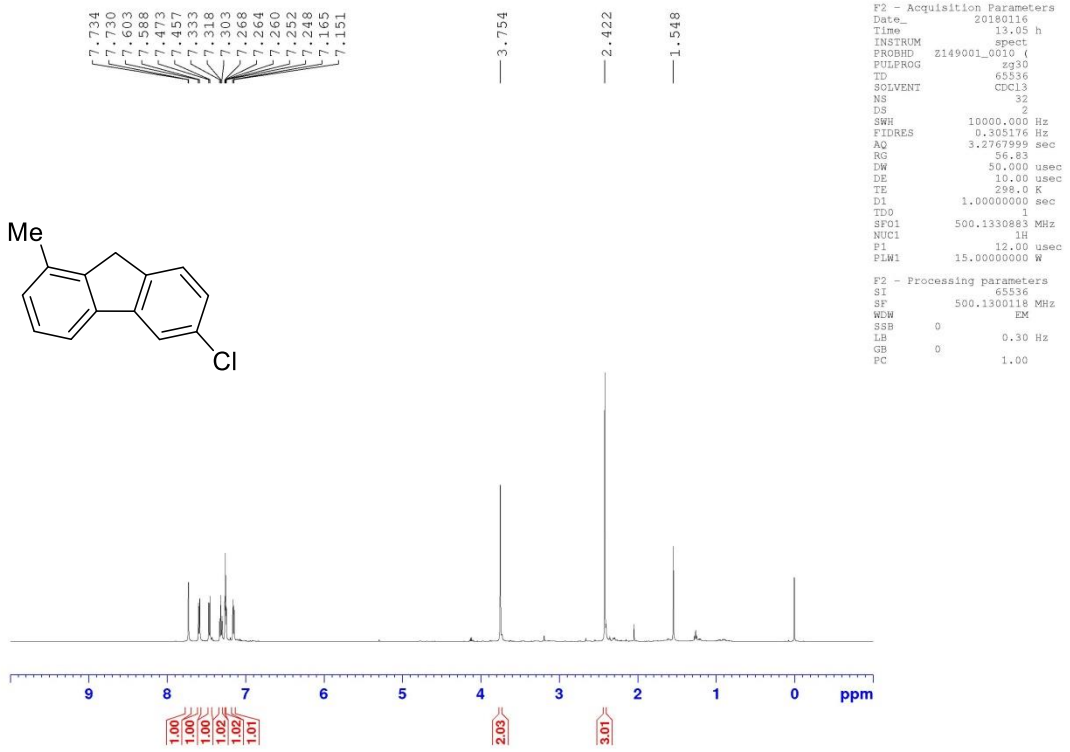
Time                 10.44 h
INSTRUM              spect
PROBHD    Z149001_0010 (
PULPROG            zgpg30
TD                  65536
SOLVENT              CDCl3
NS                      64
DS                       4
SWH              29761.904 Hz
FIDRES               0.908261 Hz
AQ                1.1010048 sec
RG                   206.72
DW                   16.800 usec
DE                   18.00 usec
TE                   298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1
SFO1          125.7703643 MHz
NUC1                    13C
P1                   10.00 usec
PLW1          61.00000000 W
SFO2          500.1320005 MHz
NUC2                    1H
CPDPRG[2          waltz16
PCPD2                80.00 usec
PLW2          15.00000000 W
PLW12          0.33750001 W
PLW13          0.16976000 W

F2 - Processing parameters
SI                   32768
SF            125.7577991 MHz
WDW                     EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.40
```

^{1}H NMR and ^{13}C NMR spectra for 6-Chloro-1-methyl-9*H*-fluorene (Table 2, entry 3x)

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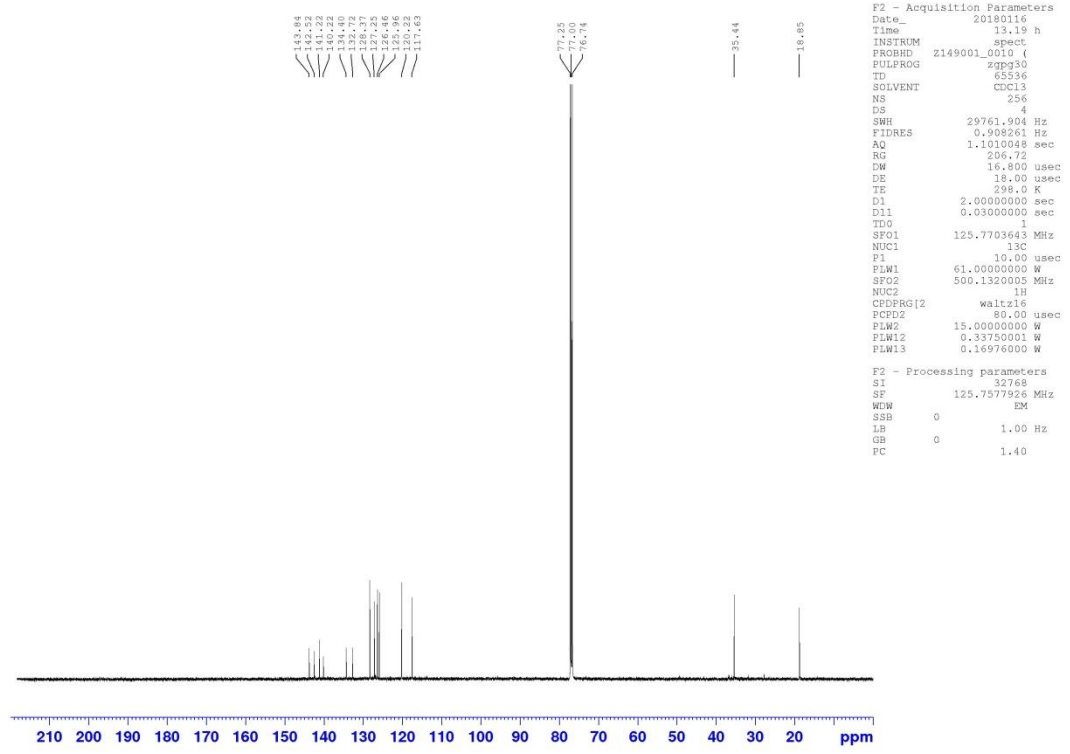
INSTRUM               spect
PROBHD    Z149001_0010 (
PULPROG                zg30
TD                    65536
SOLVENT              CDCl3
NS                       32
DS                        2
SWH              10000.000 Hz
FIDRES               0.305176 Hz
AQ                 3.2767999 sec
RG                      56.83
DW                   50.000 usec
DE                   10.00 usec
TE                   298.0 K
D1                1.00000000 sec
TD0                       1
SFO1           500.1330883 MHz
NUC1                     1H
P1                   12.00 usec
PLW1          15.00000000 W

F2 - Processing parameters
SI                    65536
SF             500.1300118 MHz
WDW                      EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    1.00
```

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F2 - Acquisition Parameters
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Time                 13.19 h
INSTRUM               spect
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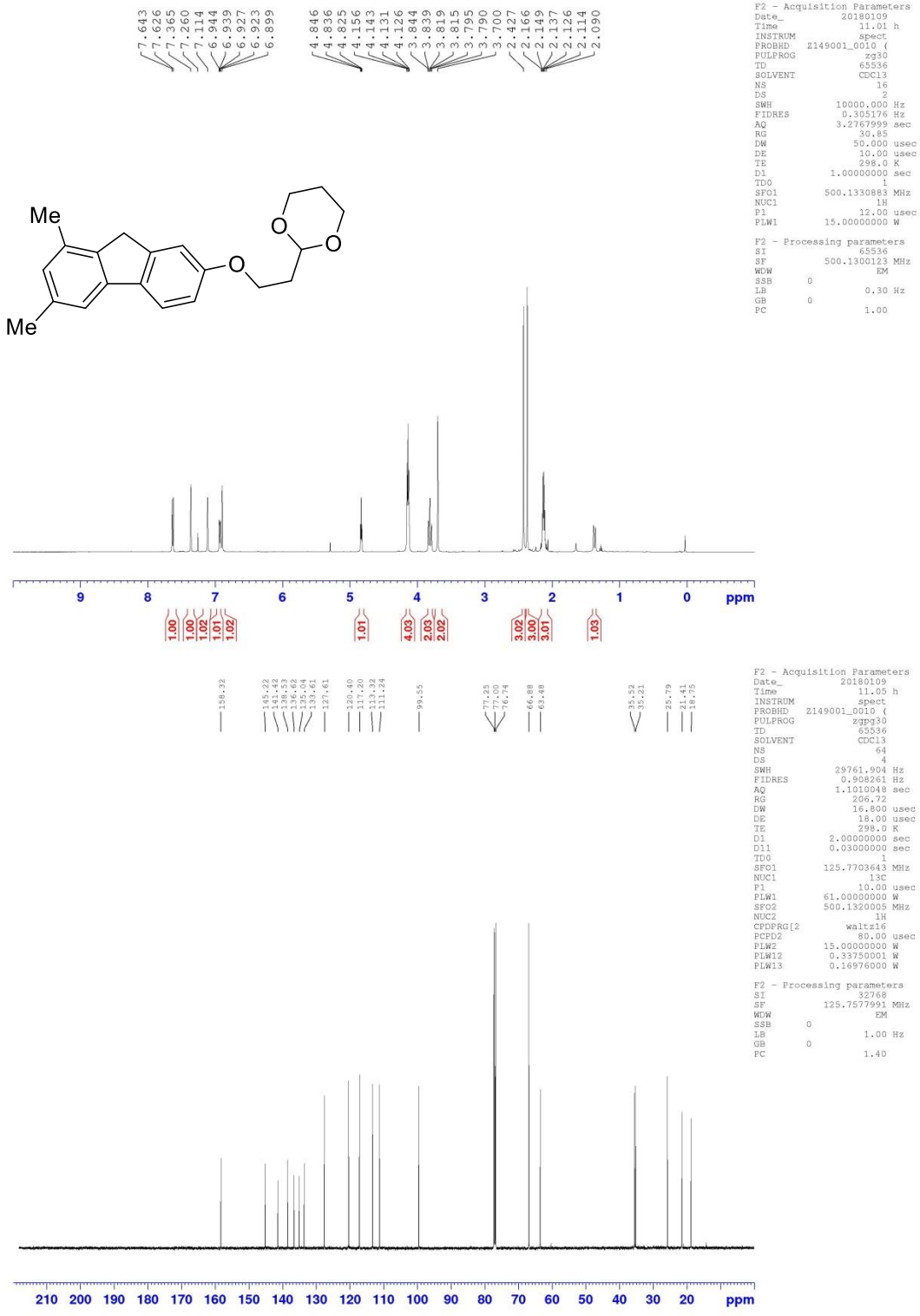
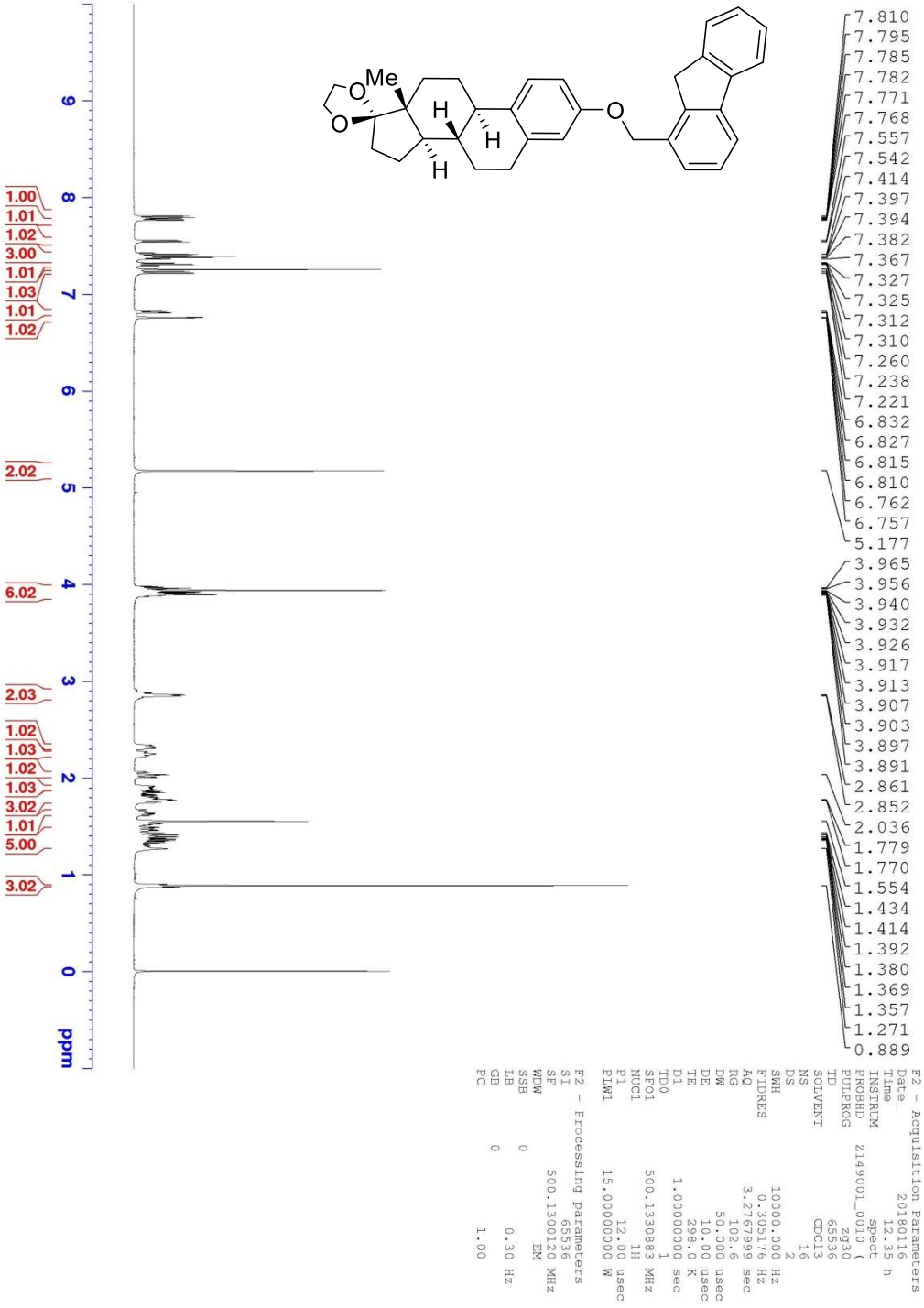
PULPROG             zgpg30
TD                    65536
SOLVENT              CDCl3
NS                      256
DS                        4
SWH              29761.904 Hz
FIDRES               0.908261 Hz
AQ                 1.1010048 sec
RG                     206.72
DW                   16.800 usec
DE                   18.00 usec
TE                   298.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1
SFO1           125.7703643 MHz
NUC1                     13C
P1                   10.00 usec
PLW1          61.00000000 W
SFO2           500.1320005 MHz
NUC2                     1H
CPDPRG[2             waltz16
PCPD2                80.00 usec
PLW2          15.00000000 W
PLW12          0.33750001 W
PLW13          0.16976000 W

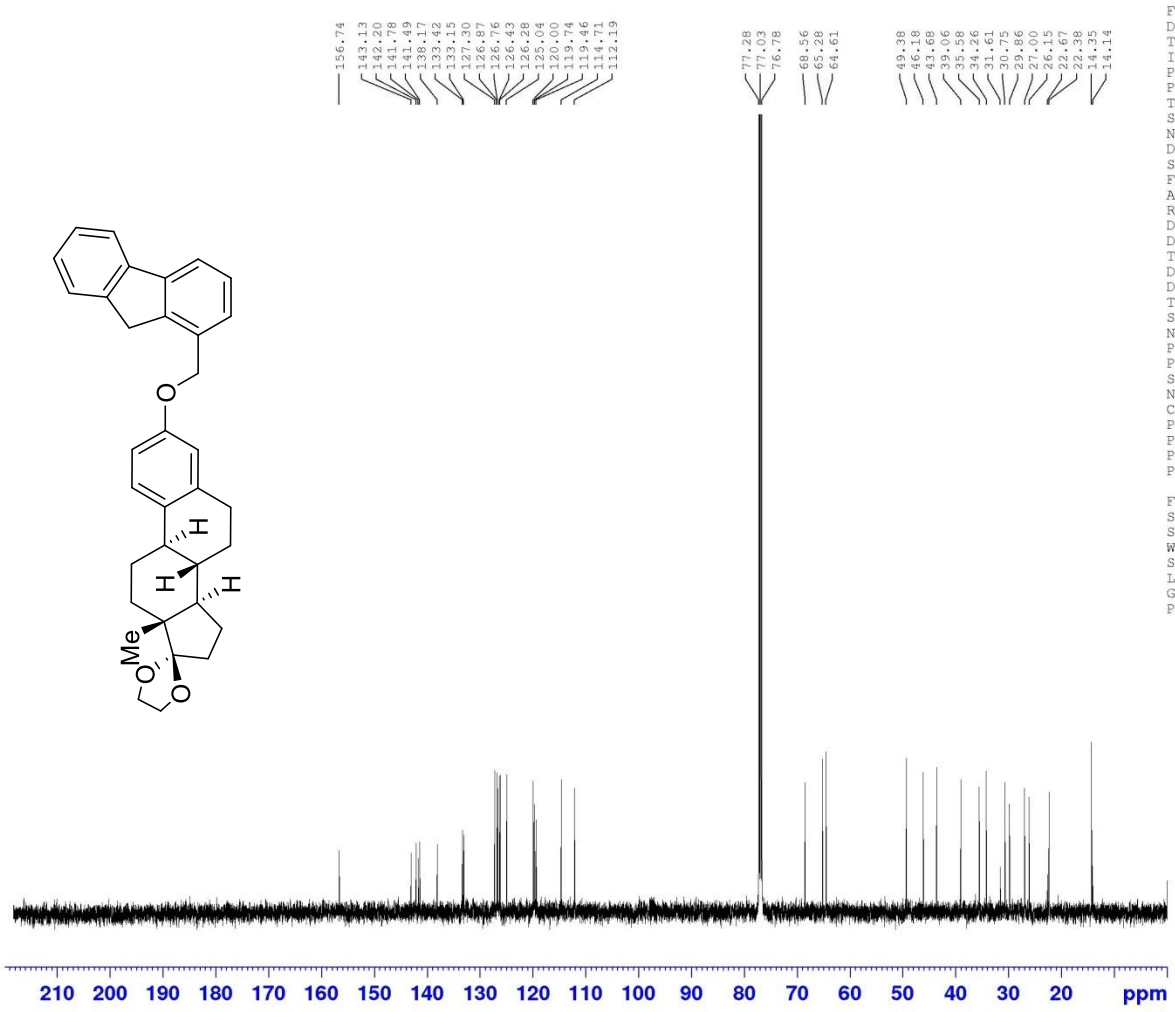
F2 - Processing parameters
SI                    32768
SF             125.7577926 MHz
WDW                      EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.40
```

^{1}H NMR and ^{13}C NMR spectra for 2-(2-((6,8-Dimethyl-9*H*-fluoren-2-yl)oxy)ethyl)-1,3-dioxane (Table 2, entry 3y)



^{1}H NMR and ^{13}C NMR spectra for (8*R*,9*S*,13*S*,14*S*)-3-((9*H*-fluoren-1-yl)methoxy)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydrospiro[cyclopenta[*a*]phenanthrene-17,2'-[1,3]dioxolane] (Table 2, entry 3z)



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F2 - Acquisition Parameters
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Time                  12.33 h
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PROBHD    Z149001_0010 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       64
DS                        4
SWH               29761.904 Hz
FIDRES             0.908261 Hz
AQ                1.1010048 sec
RG                   206.72
DW                   16.800 usec
DE                   18.00 usec
TE                   298.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1
SFO1           125.7703643 MHz
NUC1                    13C
P1                   10.00 usec
PLW1           61.00000000 W
SFO2           500.1320005 MHz
NUC2                    1H
CPDPRG[2            waltz16
PCPD2                80.00 usec
PLW2           15.00000000 W
PLW12           0.33750001 W
PLW13           0.16976000 W

F2 - Processing parameters
SI                    32768
SF             125.7577885 MHz
WDW                      EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.40
```

^{1}H NMR and ^{13}C NMR spectra for 8,8'-(Oxybis(methylene))bis(3-methyl-9*H*-fluorene) (Scheme 1, compound 4)