

Supporting information

TEMPO-mediated late stage photochemical hydroxylation of biaryl sulfonium salts

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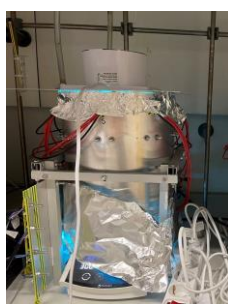
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General Information

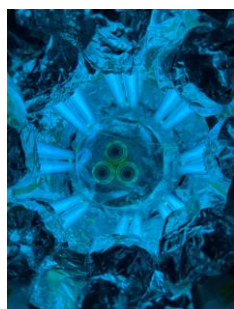
NMR spectra were obtained on an Agilent VNMRS 400 or a Bruker Av 600 using CDCl₃ or DMSO-d₆ as solvents. Chemical shifts are given in ppm and coupling constants (*J*) in Hz. The following abbreviations were used for ¹H NMR spectra to indicate the signal multiplicity: s (singlet), d (doublet), t (triplet), q (quartet) and m (multiplet) as well as combinations of them. Flash chromatography was performed on silica gel (60 M, 0.04-0.063 mm) by standard technique. All the chemicals used for synthesis were purchased from Sigma Aldrich, abcr, Alfa Aesar, TCI, Fisher, BLDpharma or ChemPUR. High resolution mass spectra (HRMS) were recorded on ThermoFisher Scientific LTQ Orbitrap XL spectrometer. IR spectra were measured on a PerkinElmer 100 FT-IR spectrometer with an UATR Diamond KRS-5 unit. The absorption spectra were measured from 500 nm to 240 nm with a medium scan speed on an Agilent Cary 60 UV-Vis Spectrophotometer (1 cm, quartz cells).

Crystallographic data were collected on a Bruker Kappa APEX II CCD-diffractometer with monochromatic Mo-K α radiation ($\lambda = 0.71073 \text{ \AA}$) and a CCD detector.

All reactions with UV-light were carried out using a 23 cm diameter steel cylinder photo-reactor equipped with 8 Heissner GmbH UV Lamps (Model: ZF 418, 18W, Type PL-L, 230 V, 4 pins 2G11), thus in total 144W at $\lambda = 254 \text{ nm}$. The cylindrical quartz vials (50 mL) were thus situated at approximately 11 cm from the light source. In order to avoid overheating of the reaction mixtures, the quartz vials were cooled with a ventilator located on top of the photo-reactor. No filters were utilized:



144 Watt UV reactor



Inside of UV reactor
with 8 x 18 W UV lamps



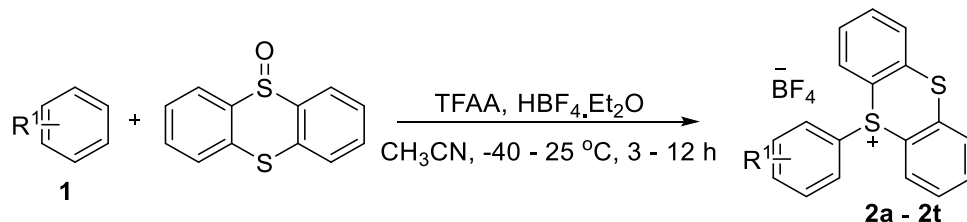
18 W UV lamp



50 mL quartz reaction vial

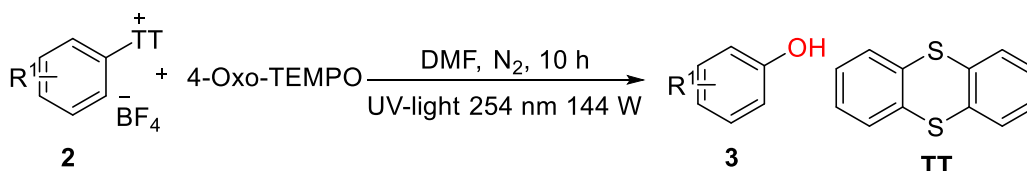
1. Experimental Section

1.1 General procedure A for the synthesis of aryl thianthrenium salts **2**^{1,2}



Under ambient atmosphere, a 50 mL round-bottom flask equipped with a magnetic stir bar was charged with simple arenes (2 mmol, 1.0 equiv.), thianthrene S-oxide (464 mg, 2 mmol, 1 equiv.) and CH₃CN (5 mL). After cooling to -40 °C, Trifluoroacetic anhydride (0.56 mL, 840mg, 4 mmol, 2.0 equiv.) was added in one portion, followed by the addition of HBF₄·OEt₂ (0.6 mL, 712 mg, 4.4 mmol, 2.2 equiv.) in one portion. The mixture was stirred at -40 °C for 1 h, then at ambient temperature for 3-12 h. The reaction mixture was concentrated under reduced pressure, and subsequently diluted with DCM (14 mL). The solution was poured onto a saturated aqueous NaHCO₃ solution (14 mL), and the layers were separated. The organic phase was washed with aqueous NaBF₄ solution (2 × 14 mL, 10%), and with water (2 × 14 mL). The organic phase was dried over Na₂SO₄, and the solvent was removed under reduced pressure. The residue was purified by chromatography on silica gel eluting with DCM / MeOH (20:1 (v/v)) to afford **2a – 2t**.

1.2 General procedure B for the hydroxylation of aryl sulfonium salts

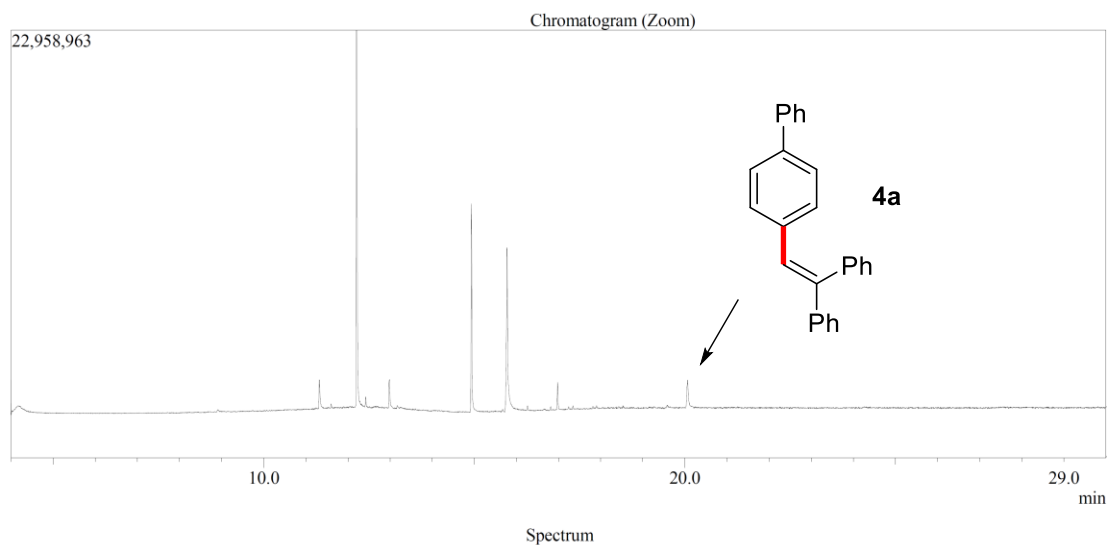


Under N₂ atmosphere, a 50 mL flat-bottom quartz vial equipped with a magnetic stir bar was charged with aryl thianthrenium salts **2** (0.4 mmol, 1.0 equiv.), 4-Oxo-TEMPO (3.2 mmol, 8 equiv.) and DMF (3 mL). The tube was sealed, and the mixture was stirred at room temperature under UV-light (254 nm, 144 W) for 10 h before quenching with aqueous saturated NaHCO₃ and dilution with EtOAc. The organic layer was washed with brine, dried using Na₂SO₄, filtered, and concentrated *in vacuo*, to give the crude product **3a – 3s**, which was purified by column chromatography on silica gel.

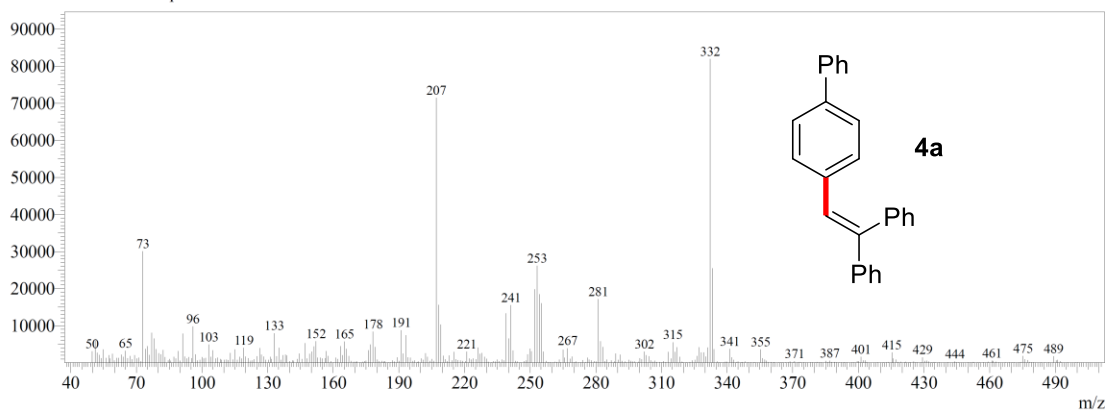
1.3 Mechanistic experiments

Mechanistic experiments were carried out along general procedure **B**, with the addition of an additive to the reaction mixture before the start of the reaction (1,4-dinitrobenzene, 1,1-diphenylethylene, or BHT), in 2 equivalents, as noted in the article. The reaction

work-up is otherwise identical. GCMS profile of the crude reaction mixture in the case of 1,1-diphenylethylene (product **4a**):



Line#:1 R.Time:20.065(Scan#:3214)
MassPeaks:451
RawMode:Averaged 19.980-20.140(3197-3229) BasePeak:332.25(81908)
BG Mode:None Group 1 - Event 1 Scan



2. UV-vis absorption spectroscopic measurements

The UV-Vis spectroscopy was used to measure the absorption of **2a** and **2a** with 4-Oxo-TEMPO (medium scan speed on an Agilent Cary 60 UV-Vis Spectrophotometer, 1 cm, quartz cells). As shown in **Figure S1**, **2a** exhibits the main absorption within 260-350 nm and almost no absorption can be seen shorter than 260 nm. In order to eliminate the influence of 4-Oxo-TEMPO to **2a**, 8 equiv. 4-Oxo-TEMPO was added to measure the absorption which is consistent with the amount during the synthesis, and the red line reveals no obvious difference as **2a**.

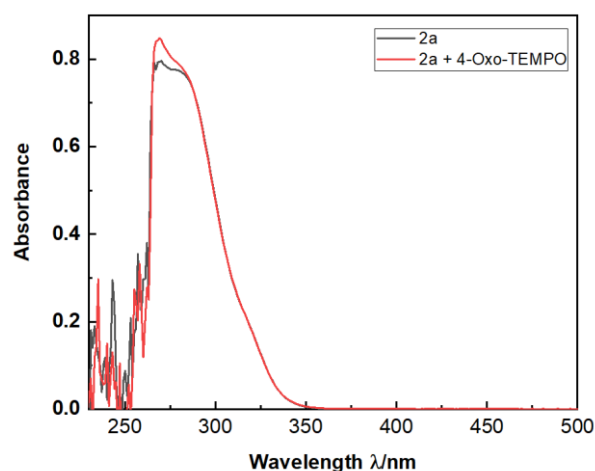


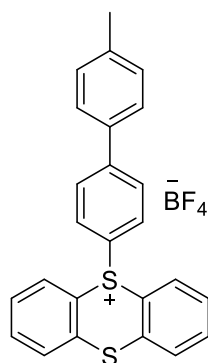
Figure S1 UV absorption spectroscopy

3. Characterization of Products

Thianthrenium salts **2a**, **2m**, and **2s** were prepared as previously described.³

3.1 Characterization of aryl thianthrenium salts

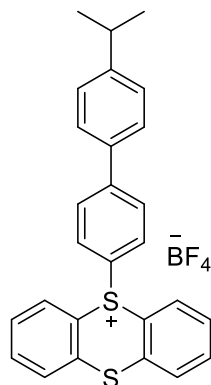
4-Methylbiphenyl derived thianthrenium salt **2b**



Following the general procedure A afforded the product as a yellow solid (749 mg, 80% yield, 2 mmol); Chromatography column, DCM /MeOH = 20:1.

¹H NMR (600 MHz, DMSO-*d*₆) δ 8.61 (dd, *J* = 7.9, 1.2 Hz, 2H), 8.06 (dd, *J* = 7.9, 1.1 Hz, 2H), 7.93 (td, *J* = 7.7, 1.4 Hz, 2H), 7.88 (td, *J* = 7.7, 1.3 Hz, 2H), 7.82 (d, *J* = 8.8 Hz, 2H), 7.54 (d, *J* = 8.2 Hz, 2H), 7.28 (d, *J* = 8.8 Hz, 2H), 7.25 (d, *J* = 8.1 Hz, 2H), 2.30 (s, 3H). ¹⁹F NMR (565 MHz, DMSO-*d*₆) δ -148.09 (s, 1F), -148.15 (s, 3F). ¹³C NMR (151 MHz, DMSO-*d*₆) δ 144.2 (s), 138.6 (s), 135.6 (s), 135.4 (s), 134.9 (s), 134.7 (s), 130.3 (s), 129.8 (s), 129.8 (s), 128.7 (s), 128.3 (s), 126.9 (s), 122.9 (s), 119.3 (s), 20.7 (s). HRMS-ESI (*m/z*): Calculated for C₂₅H₁₉S₂: [M – BF₄]⁺ 383.0922; Found, 383.0920. IR: 3630, 3083, 2324, 1567, 1482, 1448, 1392, 1287, 1197, 1049, 807, 760, 703 cm⁻¹.

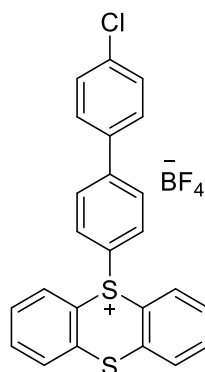
4-Isopropylbiphenyl derived thianthrenium salt **2c**



Following the general procedure A afforded the product as a yellow solid (712 mg, 71% yield, 2 mmol); Chromatography column, DCM /MeOH = 20:1.

^1H NMR (600 MHz, DMSO- d_6) δ 8.62 (dd, $J = 7.9, 1.3$ Hz, 2H), 8.08 (dd, $J = 7.9, 1.2$ Hz, 2H), 7.94 (td, $J = 7.7, 1.4$ Hz, 2H), 7.88 (td, $J = 7.7, 1.3$ Hz, 2H), 7.83 (d, $J = 8.9$ Hz, 2H), 7.57 (d, $J = 8.4$ Hz, 2H), 7.33 (d, $J = 8.3$ Hz, 2H), 7.29 (d, $J = 8.8$ Hz, 2H), 2.90 (hept, $J = 6.9$ Hz, 1H), 1.19 (d, $J = 6.9$ Hz, 6H). ^{19}F NMR (565 MHz, DMSO- d_6) δ -148.17 (s, 1F), -148.22 (s, 3F). ^{13}C NMR (151 MHz, DMSO- d_6) δ 149.3 (s), 144.3 (s), 135.6 (s), 135.4 (s), 135.2 (s), 134.8 (s), 130.3 (s), 129.7 (s), 128.7 (s), 128.3 (s), 127.1 (s), 127.0 (s), 123.0 (s), 119.2 (s), 33.1 (s), 23.7 (s). HRMS-ESI (m/z): Calculated for $\text{C}_{27}\text{H}_{23}\text{S}_2$: $[\text{M} - \text{BF}_4]^+$ 411.1234; Found, 411.1229. IR: 3098, 2956, 2868, 2324, 1991, 1646, 1567, 1485, 1451, 1287, 1092, 1052, 964, 820, 764, 710, 660 cm^{-1} .

4-Chlorobiphenyl derived thianthrenium salt **2d**

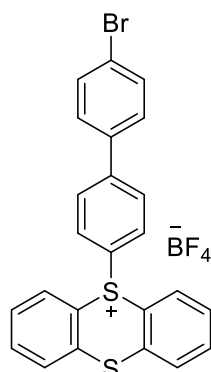


Following the general procedure A afforded the product as a yellow solid (825 mg, 84% yield, 2 mmol); Chromatography column, DCM /MeOH = 20:1.

^1H NMR (400 MHz, DMSO- d_6) δ 8.62 (d, $J = 7.8$ Hz, 2H), 8.07 (d, $J = 7.7$ Hz, 2H), 7.97 – 7.91 (m, 2H), 7.88 (td, $J = 7.7, 1.3$ Hz, 2H), 7.84 (d, $J = 8.7$ Hz, 2H), 7.67 (d, $J = 8.6$ Hz, 2H), 7.50 (d, $J = 8.6$ Hz, 2H), 7.30 (d, $J = 8.7$ Hz, 2H). ^{19}F NMR (376 MHz, DMSO- d_6) δ -148.14 (s, 1F), -148.20 (s, 3F). ^{13}C NMR (101 MHz, DMSO- d_6) δ 142.9 (s), 136.5 (s), 135.7 (s), 135.5 (s), 134.9 (s), 133.9 (s), 130.3 (s), 129.8 (s), 129.2 (s), 128.9 (s), 128.8 (s), 128.5 (s), 123.9 (s), 119.2 (s). HRMS-ESI (m/z): Calculated for $\text{C}_{24}\text{H}_{16}\text{ClS}_2$: $[\text{M} - \text{BF}_4]^+$ 403.0377; Found, 403.0372. IR: 3082, 2925, 2323, 2157, 1814,

1591, 1566, 1472, 1453, 1384, 1266, 1169, 1027, 813, 751, 701, 659 cm^{-1} .

4-Bromobiphenyl derived thianthrenium salt **2e**

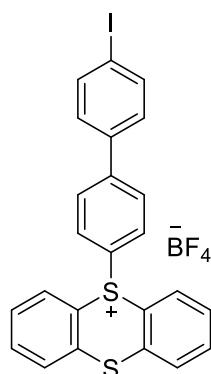


Following the general procedure A afforded the product as a white solid (895 mg, 84% yield, 2 mmol); Chromatography column, DCM /MeOH = 20:1.

^1H NMR (600 MHz, DMSO- d_6) δ 8.63 (dd, $J = 7.9, 1.2$ Hz, 2H), 8.07 (dd, $J = 7.9, 1.1$ Hz, 2H), 7.94 (td, $J = 7.7, 1.4$ Hz, 2H), 7.89 (td, $J = 7.7, 1.3$ Hz, 2H), 7.84 (d, $J = 8.8$ Hz, 2H), 7.64 (d, $J = 8.7$ Hz, 2H), 7.60 (d, $J = 8.7$ Hz, 2H), 7.30 (d, $J = 8.8$ Hz, 2H).

^{19}F NMR (565 MHz, DMSO- d_6) δ -148.10 (s, 1F), -148.15 (s, 3F). ^{13}C NMR (151 MHz, DMSO- d_6) δ 142.9 (s), 136.8 (s), 135.6 (s), 135.4 (s), 134.9 (s), 132.0 (s), 130.3 (s), 129.7 (s), 129.1 (s), 128.8 (s), 128.4 (s), 123.9 (s), 122.5 (s), 119.2 (s). HRMS-ESI (m/z): Calculated for $\text{C}_{24}\text{H}_{16}\text{BrS}_2$: $[\text{M} - \text{BF}_4]^+$ 446.9871; Found, 446.9870. IR: 3632, 3555, 3085, 2683, 2322, 1997, 1630, 1588, 1567, 1474, 1449, 1384, 1288, 1266, 1049, 810, 757, 702, 658 cm^{-1} .

4-Iodobiphenyl derived thianthrenium salt **2f**

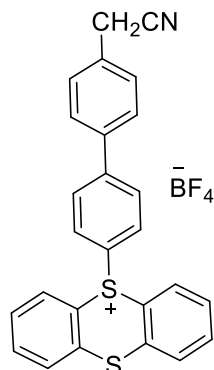


Following the general procedure A afforded the product as a white solid (864 mg, 74% yield, 2 mmol); Chromatography column, DCM /MeOH = 20:1.

^1H NMR (600 MHz, DMSO- d_6) δ 8.62 (dd, $J = 7.9, 1.2$ Hz, 2H), 8.08 (dd, $J = 7.9, 1.1$ Hz, 2H), 7.94 (td, $J = 7.7, 1.3$ Hz, 2H), 7.88 (td, $J = 7.8, 1.2$ Hz, 2H), 7.83 (dd, $J = 8.5, 6.0$ Hz, 4H), 7.45 (d, $J = 8.5$ Hz, 2H), 7.29 (d, $J = 8.8$ Hz, 2H). ^{19}F NMR (565 MHz, DMSO- d_6) δ -148.17 (s, 1F), -148.23 (s, 3F). ^{13}C NMR (151 MHz, DMSO- d_6) δ 143.1 (s), 137.9 (s), 137.1 (s), 135.7 (s), 135.4 (s), 134.8 (s), 130.3 (s), 129.6 (s), 129.1 (s), 128.8 (s), 128.3 (s), 123.9 (s), 119.2 (s), 95.8 (s). HRMS-ESI (m/z): Calculated for

C₂₄H₁₆IS₂: [M – BF₄]⁺ 494.9733; Found, 494.9728. IR: 3828, 3077, 2924, 2325, 2084, 1996, 1818, 1569, 1450, 1380, 1287, 1260, 1199, 1044, 844, 806, 751, 701, 658 cm⁻¹.

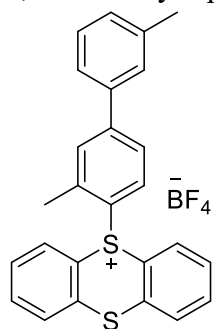
4-Biphenylacetonitrile derived thianthrenium salt **2g**



Following the general procedure A afforded the product as a pale yellow solid (868 mg, 88% yield, 2 mmol); Chromatography column, DCM /MeOH = 10:1.

¹H NMR (600 MHz, DMSO-d₆) δ 8.62 (dd, *J* = 7.9, 0.9 Hz, 2H), 8.04 (d, *J* = 7.8 Hz, 2H), 7.93 (td, *J* = 7.7, 1.1 Hz, 2H), 7.88 (td, *J* = 7.8, 1.0 Hz, 2H), 7.84 (d, *J* = 8.7 Hz, 2H), 7.67 (d, *J* = 8.3 Hz, 2H), 7.45 (d, *J* = 8.3 Hz, 2H), 7.31 (d, *J* = 8.7 Hz, 2H), 4.08 (s, 2H). ¹⁹F NMR (565 MHz, DMSO-d₆) δ -147.95 (s, 1F), -148.00 (s, 3F). ¹³C NMR (151 MHz, DMSO-d₆) δ 143.6 (s), 137.0 (s), 135.6 (s), 135.5 (s), 134.9 (s), 132.1 (s), 130.3 (s), 129.8 (s), 129.0 (s), 128.7 (s), 128.5 (s), 127.6 (s), 123.6 (s), 119.1 (s), 119.1 (s), 22.2 (s). HRMS-ESI (*m/z*): Calculated for C₂₆H₁₈NS₂: [M – BF₄]⁺ 408.0875; Found, 408.0870. IR: 3628, 3082, 2925, 2251, 1567, 1483, 1449, 1394, 1288, 1266, 1034, 803, 758, 704, 658 cm⁻¹.

3,3'-Dimethylbiphenyl derived thianthrenium salt **2h**

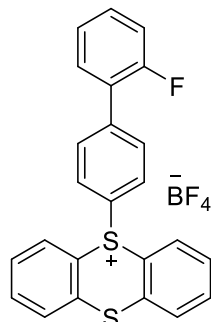


Following the general procedure A afforded the product as a yellow solid (681 mg, 70% yield, 2 mmol); Chromatography column, DCM /MeOH = 20:1.

¹H NMR (600 MHz, DMSO-d₆) δ 8.48 (d, *J* = 8.0 Hz, 2H), 8.12 (d, *J* = 7.9 Hz, 2H), 7.94 – 7.90 (m, 2H), 7.86 (d, *J* = 1.3 Hz, 1H), 7.85 – 7.82 (m, 2H), 7.63 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.48 (s, 1H), 7.44 (d, *J* = 7.9 Hz, 1H), 7.35 (t, *J* = 7.7 Hz, 1H), 7.23 (d, *J* = 7.6 Hz, 1H), 7.05 (d, *J* = 8.6 Hz, 1H), 2.73 (s, 3H), 2.34 (s, 3H). ¹⁹F NMR (565 MHz, DMSO-d₆) δ -148.16 (s, 1F), -148.21 (s, 3F). ¹³C NMR (151 MHz, DMSO-d₆) δ 145.3 (s), 140.2 (s), 138.4 (s), 137.5 (s), 136.2 (s), 134.9 (s), 134.6 (s), 131.7 (s), 130.7 (s), 129.8 (s), 129.8 (s), 129.6 (s), 129.0 (s), 127.7 (s), 125.7 (s), 124.2 (s), 120.9 (s), 118.4

(s), 21.0 (s), 20.2 (s). HRMS-ESI (m/z): Calculated for C₂₆H₂₁S₂: [M – BF₄]⁺ 397.1079; Found, 397.1075. IR: 3632, 3081, 2919, 2333, 1592, 1564, 1447, 1384, 1271, 1050, 881, 760, 698 cm⁻¹.

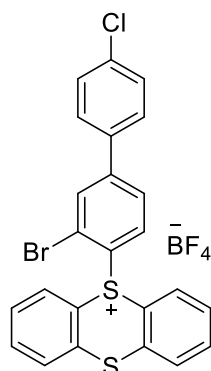
2-Fluorobiphenyl derived thianthrenium salt **2i**



Following the general procedure A afforded the product as a white solid (779 mg, 82% yield, 2 mmol); Chromatography column, DCM /MeOH = 20:1.

¹H NMR (600 MHz, DMSO-d₆) δ 8.64 (d, *J* = 7.8 Hz, 2H), 8.08 (d, *J* = 7.8 Hz, 2H), 7.95 (t, *J* = 7.6 Hz, 2H), 7.89 (t, *J* = 7.6 Hz, 2H), 7.73 (d, *J* = 8.2 Hz, 2H), 7.50 (t, *J* = 7.8 Hz, 1H), 7.46 (dd, *J* = 13.2, 7.2 Hz, 1H), 7.33 (d, *J* = 8.7 Hz, 2H), 7.32 – 7.27 (m, 2H). ¹⁹F NMR (565 MHz, DMSO-d₆) δ -118.04 (s, 1F), -148.12 (s, 1F), -148.18 (s, 3F). ¹³C NMR (151 MHz, DMSO-d₆) δ 159.0 (d, *J* = 247.2 Hz), 139.3 (s), 135.7 (s), 135.5 (s), 134.1 (s), 131.0 (d, *J* = 8.3 Hz), 130.8 (d, *J* = 2.0 Hz), 130.7 (d, *J* = 2.6 Hz), 130.4 (s), 129.7 (s), 128.4 (s), 126.0 (d, *J* = 13.0 Hz), 125.2 (d, *J* = 3.3 Hz), 124.2 (s), 119.1 (s), 116.4 (s), 116.2 (s). HRMS-ESI (m/z): Calculated for C₂₄H₁₆FS₂: [M – BF₄]⁺ 387.0672; Found, 387.0667. IR: 3091, 2700, 2326, 2081, 1614, 1570, 1474, 1450, 1392, 1288, 1260, 1214, 1043, 823, 755, 703, 659 cm⁻¹.

3-Bromo-4'-chloro-1,1'-biphenyl derived thianthrenium salt **2j**

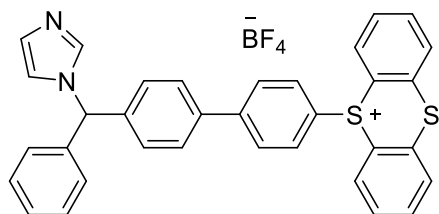


Following the general procedure A afforded the product as a yellow solid (465 mg, 41% yield, 2 mmol); Chromatography column, DCM /MeOH = 20:1.

¹H NMR (600 MHz, DMSO-d₆) δ 8.60 (dd, *J* = 8.1, 1.2 Hz, 2H), 8.25 (d, *J* = 2.0 Hz, 1H), 8.12 (dd, *J* = 8.0, 1.1 Hz, 2H), 7.93 (td, *J* = 7.8, 1.3 Hz, 2H), 7.88 – 7.83 (m, 2H), 7.80 (dd, *J* = 8.7, 2.0 Hz, 1H), 7.72 (d, *J* = 8.7 Hz, 2H), 7.51 (d, *J* = 8.7 Hz, 2H), 7.07 (d, *J* = 8.6 Hz, 1H). ¹⁹F NMR (565 MHz, DMSO-d₆) δ -148.16 (s, 1F), -148.21 (s, 3F). ¹³C NMR (151 MHz, DMSO-d₆) δ 145.2 (s), 136.8 (s), 136.5 (s), 135.1 (s), 134.8 (s),

134.5 (s), 133.5 (s), 131.8 (s), 130.7 (s), 129.5 (s), 129.2 (s), 129.1 (s), 126.7 (s), 124.0 (s), 121.9 (s), 117.8 (s). HRMS-ESI (m/z): Calculated for C₂₄H₁₅BrClS₂: [M – BF₄]⁺ 480.9482; Found, 480.9476. IR: 3632, 3084, 2329, 1726, 1620, 1576, 1448, 1370, 1287, 1259, 1170, 1048, 815, 758, 701, 658 cm⁻¹.

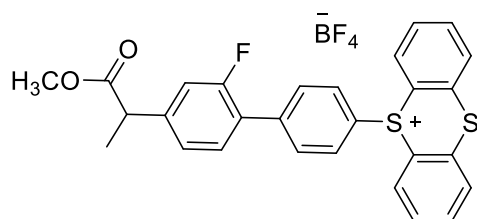
Bifonazole derived thianthrenium salt **2k**



Under ambient atmosphere, a 50 mL round-bottom flask equipped with a magnetic stir bar was charged with bifonazole (2 mmol, 1.0 equiv.), thianthrene S-oxide (450 mg, 1.94 mmol, 0.97 equiv.), thianthrene (14 mg, 0.06 mmol, 0.03 equiv.) and CH₃CN (5 mL). After cooling to -40°C, Trifluoroacetic anhydride (0.56 mL, 840mg, 4 mmol, 2.0 equiv.) was added in one portion, followed by the addition of HBF₄·OEt₂ (0.6 mL, 712 mg, 4.4 mmol, 2.2 equiv.) in one portion. The mixture was stirred at -40 °C for 1 h, then at ambient temperature for 6 h. The reaction mixture was concentrated under reduced pressure, and subsequently diluted with DCM (14 mL). The solution was poured onto a saturated aqueous NaHCO₃ solution (14 mL), and the layers were separated. The organic phase was washed with aqueous NaBF₄ solution (2 × 14 mL, 10%), and with water (2 × 14 mL). The organic phase was dried over Na₂SO₄, and the solvent was removed under reduced pressure. The residue was purified by chromatography on silica gel eluting with DCM / MeOH (10:1 (v/v)) to afford **2k** as a yellow solid (887 mg, 72% yield, 2 mmol).

¹H NMR (400 MHz, DMSO-d₆) δ 8.62 (d, *J* = 7.7 Hz, 2H), 8.08 (d, *J* = 7.7 Hz, 2H), 7.98 – 7.82 (m, 6H), 7.76 (d, *J* = 8.3 Hz, 1H), 7.68 (d, *J* = 8.2 Hz, 2H), 7.43 – 7.33 (m, 3H), 7.30 (d, *J* = 8.6 Hz, 2H), 7.24 (d, *J* = 8.1 Hz, 2H), 7.18 (d, *J* = 7.2 Hz, 3H), 7.02 (s, 1H), 6.94 (s, 1H). ¹⁹F NMR (376 MHz, DMSO-d₆) δ -148.20 (s, 1F), -148.26 (s, 3F). ¹³C NMR (101 MHz, DMSO-d₆) δ 143.5 (s), 140.5 (s), 139.5 (s), 137.3 (s), 135.7 (s), 135.4 (s), 134.8 (s), 130.3 (s), 129.7 (s), 128.8 (s), 128.7 (s), 128.6 (s), 128.1 (s), 127.9 (s), 127.5 (s), 123.7 (s), 119.2 (s), 63.1 (s). HRMS-ESI (m/z): Calculated for C₃₄H₂₅N₂S₂: [M – BF₄]⁺ 525.1454; Found, 525.1446. IR: 3746, 3394, 3086, 2165, 1831, 1605, 1572, 1447, 1379, 1320, 1252, 1172, 1055, 959, 870, 845, 763 cm⁻¹.

Flurbiprofen derived thianthrenium salt **2l**

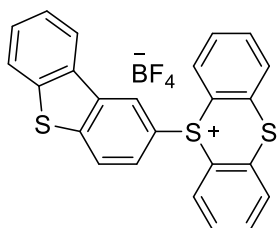


Following the general procedure A afforded the product as a white solid (878 mg, 78%

yield, 2 mmol); Chromatography column, DCM /MeOH = 10:1.

^1H NMR (400 MHz, DMSO- d_6) δ 8.62 (d, J = 7.8 Hz, 2H), 8.09 (d, J = 7.8 Hz, 2H), 7.95 (t, J = 7.5 Hz, 2H), 7.88 (t, J = 7.6 Hz, 2H), 7.73 (d, J = 8.1 Hz, 2H), 7.47 (t, J = 8.1 Hz, 1H), 7.32 (d, J = 8.6 Hz, 2H), 7.24 (dd, J = 14.0, 10.3 Hz, 2H), 3.90 (q, J = 7.1 Hz, 1H), 3.60 (s, 3H), 1.41 (d, J = 7.1 Hz, 3H). ^{19}F NMR (376 MHz, DMSO- d_6) δ -117.85 (s, 1F), -148.24 (s, 1F), -148.30 (s, 3F). ^{13}C NMR (101 MHz, DMSO- d_6) δ 173.6 (s), 158.8 (d, J = 247.7 Hz), 144.0 (d, J = 8.0 Hz), 139.0 (s), 135.7 (s), 135.5 (s), 134.9 (s), 130.9 (d, J = 2.8 Hz), 130.6 (d, J = 2.6 Hz), 130.3 (s), 129.7 (s), 128.4 (s), 124.6 (d, J = 12.9 Hz), 124.3 (d, J = 2.7 Hz), 124.1 (s), 119.1 (s), 115.5 (s), 115.3 (s), 52.0 (s), 43.8 (s), 18.3 (s). HRMS-ESI (m/z): Calculated for $\text{C}_{28}\text{H}_{22}\text{FO}_2\text{S}_2$: $[\text{M} - \text{BF}_4]^+$ 473.1040; Found, 473.1030. IR: 3088, 2945, 2322, 1821, 1734, 1619, 1568, 1431, 1392, 1283, 1196, 1128, 1095, 1050, 919, 835, 764, 703, 658 cm^{-1} .

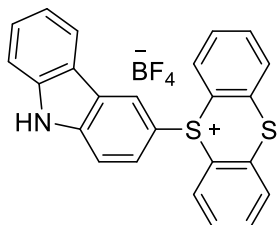
Dibenzothiophene derived thianthrenium salt **2n**



Following the general procedure A afforded the product as a white solid (650 mg, 67% yield, 2 mmol); Chromatography column, DCM /MeOH = 20:1.

^1H NMR (600 MHz, DMSO- d_6) δ 9.14 (d, J = 1.9 Hz, 1H), 8.56 (d, J = 7.8 Hz, 2H), 8.48 – 8.42 (m, 1H), 8.38 (d, J = 8.0 Hz, 2H), 8.20 (d, J = 8.7 Hz, 1H), 8.16 – 8.09 (m, 1H), 7.98 (t, J = 7.6 Hz, 2H), 7.76 (t, J = 8.1 Hz, 2H), 7.70 – 7.55 (m, 2H), 7.14 (dd, J = 8.7, 2.0 Hz, 1H). ^{19}F NMR (565 MHz, DMSO- d_6) δ -148.21 (s, 1F), -148.26 (s, 3F). ^{13}C NMR (151 MHz, DMSO- d_6) δ 144.6 (s), 139.5 (s), 139.2 (s), 136.2 (s), 133.9 (s), 133.4 (s), 131.3 (s), 128.6 (s), 128.2 (s), 126.2 (s), 126.1 (s), 125.6 (s), 124.9 (s), 124.6 (s), 124.2 (s), 123.5 (s), 122.6 (s). HRMS-ESI (m/z): Calculated for $\text{C}_{24}\text{H}_{15}\text{S}_3$: $[\text{M} - \text{BF}_4]^+$ 399.0330; Found, 399.0326. IR: 3087, 2322, 2157, 1986, 1904, 1568, 1430, 1289, 1230, 1048, 911, 874, 808, 759, 727 cm^{-1} .

Carbazole derived thianthrenium salt **2o**

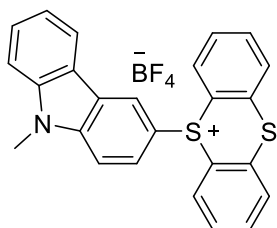


Following the general procedure A afforded the product as a white solid (572 mg, 61% yield, 2 mmol); Chromatography column, DCM /MeOH = 10:1.

^1H NMR (600 MHz, DMSO- d_6) δ 11.97 (s, 1H), 8.44 (d, J = 7.9 Hz, 2H), 8.37 (d, J = 1.9 Hz, 1H), 8.13 (d, J = 7.9 Hz, 1H), 8.07 (d, J = 7.9 Hz, 2H), 7.90 (td, J = 7.8, 1.1 Hz,

2H), 7.83 (t, $J = 7.7$ Hz, 2H), 7.70 (d, $J = 8.8$ Hz, 1H), 7.57 (d, $J = 8.1$ Hz, 1H), 7.50 (t, $J = 7.6$ Hz, 1H), 7.39 (dd, $J = 8.8, 2.1$ Hz, 1H), 7.25 (t, $J = 7.5$ Hz, 1H). ^{19}F NMR (565 MHz, DMSO- d_6) δ -148.15 (s, 1F), -148.21 (s, 3F). ^{13}C NMR (151 MHz, DMSO- d_6) δ 141.8 (s), 140.5 (s), 134.6 (s), 134.3 (s), 134.1 (s), 130.2 (s), 129.8 (s), 127.5 (s), 125.4 (s), 123.6 (s), 122.6 (s), 121.2 (s), 121.1 (s), 120.9 (s), 120.1 (s), 113.4 (s), 111.9 (s), 111.8 (s). HRMS-ESI (m/z): Calculated for $\text{C}_{24}\text{H}_{16}\text{NS}_2$: $[\text{M} - \text{BF}_4]^+$ 382.0719; Found, 382.0713. IR: 3356, 3085, 2164, 1603, 1570, 1496, 1329, 1286, 1251, 1069, 1005, 961, 808, 761, 709, 658 cm^{-1} .

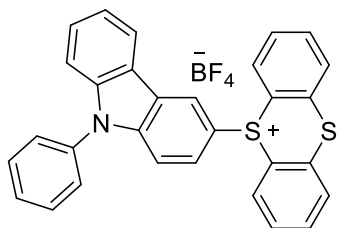
9-Methylcarbazole derived thianthrenium salt **2p**



Following the general procedure A afforded the product as a pale green solid (653 mg, 67% yield, 2 mmol); Chromatography column, DCM /MeOH = 10:1.

^1H NMR (600 MHz, DMSO- d_6) δ 8.45 (dd, $J = 8.0, 1.1$ Hz, 2H), 8.36 (d, $J = 2.1$ Hz, 1H), 8.15 (d, $J = 7.8$ Hz, 1H), 8.07 (dd, $J = 7.9, 1.0$ Hz, 2H), 7.90 (td, $J = 7.8, 1.3$ Hz, 2H), 7.86 – 7.79 (m, 3H), 7.67 (d, $J = 8.2$ Hz, 1H), 7.57 (t, $J = 7.3$ Hz, 1H), 7.46 (dd, $J = 8.9, 2.1$ Hz, 1H), 7.29 (t, $J = 7.5$ Hz, 1H), 3.89 (s, 3H). ^{19}F NMR (565 MHz, DMSO- d_6) δ -148.14 (s, 1F), -148.19 (s, 3F). ^{13}C NMR (151 MHz, DMSO- d_6) δ 142.4 (s), 141.4 (s), 134.6 (s), 134.3 (s), 134.1 (s), 130.2 (s), 129.8 (s), 127.6 (s), 125.5 (s), 123.1 (s), 122.4 (s), 121.1 (s), 120.9 (s), 120.7 (s), 120.4 (s), 112.0 (s), 111.7 (s), 110.1 (s), 29.4 (s). HRMS-ESI (m/z): Calculated for $\text{C}_{25}\text{H}_{18}\text{NS}_2$: $[\text{M} - \text{BF}_4]^+$ 396.0875; Found, 396.0870. IR: 3635, 3078, 2329, 2014, 1737, 1585, 1502, 1458, 1429, 1323, 1288, 1254, 1155, 1049, 911, 888, 751, 700, 657 cm^{-1} .

9-Phenylcarbazole derived thianthrenium salt **2q**

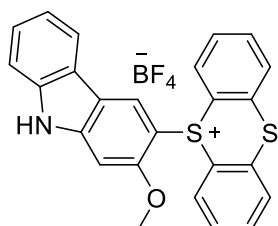


Following the general procedure A afforded the product as a pale green solid (795 mg, 82% yield, 2 mmol); Chromatography column, DCM /MeOH = 10:1.

^1H NMR (600 MHz, DMSO- d_6) δ 8.52 (d, $J = 7.6$ Hz, 2H), 8.47 (d, $J = 1.7$ Hz, 1H), 8.21 (d, $J = 7.9$ Hz, 1H), 8.01 (d, $J = 7.6$ Hz, 2H), 7.89 (t, $J = 7.2$ Hz, 2H), 7.84 (t, $J = 7.4$ Hz, 2H), 7.60 (t, $J = 7.7$ Hz, 2H), 7.52 (t, $J = 7.4$ Hz, 1H), 7.45 (d, $J = 7.6$ Hz, 2H), 7.43 – 7.40 (m, 1H), 7.39 (d, $J = 9.0$ Hz, 1H), 7.36 (dd, $J = 9.0, 1.8$ Hz, 1H), 7.30 (t, $J = 7.5$ Hz, 1H), 7.21 (d, $J = 8.2$ Hz, 1H). ^{19}F NMR (565 MHz, DMSO- d_6) δ -147.89 (s,

1F), -147.94 (s, 3F). ^{13}C NMR (151 MHz, DMSO- d_6) δ 142.0 (s), 141.0 (s), 135.4 (s), 134.8 (s), 134.5 (s), 134.4 (s), 130.3 (s), 130.2 (s), 129.9 (s), 128.6 (s), 128.0 (s), 126.7 (s), 125.8 (s), 124.0 (s), 122.5 (s), 121.4 (s), 121.3 (s), 120.4 (s), 113.9 (s), 111.9 (s), 110.2 (s). HRMS-ESI (m/z): Calculated for $\text{C}_{30}\text{H}_{20}\text{NS}_2$: $[\text{M} - \text{BF}_4]^+$ 458.1032; Found, 458.1022. IR: 3948, 3628, 3069, 2806, 2167, 1833, 1590, 1499, 1449, 1284, 1237, 1172, 1048, 801, 754, 698, 661 cm^{-1} .

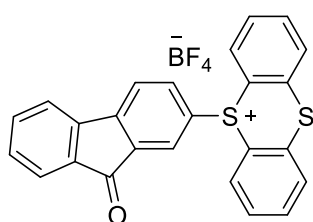
2-Methoxycarbazole derived thianthrenium salt **2r**



Following the general procedure A afforded the product as a yellow solid (437 mg, 44% yield, 2 mmol); Chromatography column, DCM /MeOH = 10:1.

^1H NMR (600 MHz, DMSO- d_6) δ 11.82 (s, 1H), 8.32 (d, J = 8.0 Hz, 2H), 8.06 (d, J = 7.9 Hz, 2H), 7.94 (d, J = 7.8 Hz, 1H), 7.88 (t, J = 7.7 Hz, 2H), 7.80 (t, J = 7.7 Hz, 2H), 7.60 (s, 1H), 7.51 (d, J = 8.1 Hz, 1H), 7.40 (t, J = 7.6 Hz, 1H), 7.32 (s, 1H), 7.17 (t, J = 7.5 Hz, 1H), 4.02 (s, 3H). ^{19}F NMR (565 MHz, DMSO- d_6) δ -148.18 (s, 1F), -148.24 (s, 3F). ^{13}C NMR (151 MHz, DMSO- d_6) δ 156.1 (s), 144.2 (s), 140.2 (s), 135.6 (s), 134.1 (s), 134.1 (s), 130.3 (s), 129.5 (s), 126.2 (s), 122.6 (s), 121.5 (s), 120.1 (s), 120.1 (s), 119.2 (s), 116.9 (s), 111.5 (s), 98.9 (s), 95.9 (s), 57.1 (s). HRMS-ESI (m/z): Calculated for $\text{C}_{25}\text{H}_{18}\text{ONS}_2$: $[\text{M} - \text{BF}_4]^+$ 412.0824; Found, 412.0812. IR: 3746, 3394, 3086, 2165, 1831, 1605, 1572, 1447, 1379, 1320, 1252, 1172, 1055, 959, 870, 845, 763 cm^{-1} .

9-Fluorenone derived thianthrenium salt **2t**



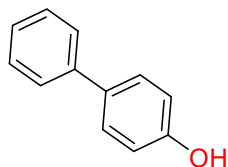
Following the general procedure A afforded the product as a yellow solid (795 mg, 82% yield, 2 mmol); Chromatography column, DCM /MeOH = 10:1.

^1H NMR (400 MHz, DMSO- d_6) δ 8.64 (d, J = 7.7 Hz, 2H), 8.10 (d, J = 7.8 Hz, 2H), 8.00 – 7.83 (m, 6H), 7.66 (dd, J = 13.0, 7.6 Hz, 2H), 7.46 (t, J = 7.5 Hz, 1H), 7.41 (dd, J = 8.1, 1.8 Hz, 1H), 7.35 (s, 1H). ^{19}F NMR (376 MHz, DMSO- d_6) δ -148.23 (s, 1F), -148.29 (s, 3F). ^{13}C NMR (101 MHz, DMSO- d_6) δ 190.7 (s), 147.3 (s), 142.0 (s), 136.0 (s), 135.9 (s), 135.4 (s), 134.9 (s), 134.3 (s), 133.3 (s), 131.1 (s), 130.3 (s), 129.7 (s), 125.8 (s), 124.6 (s), 123.1 (s), 122.7 (s), 122.5 (s), 119.4 (s). HRMS-ESI (m/z): Calculated for $\text{C}_{25}\text{H}_{15}\text{OS}_2$: $[\text{M} - \text{BF}_4]^+$ 395.0559; Found, 395.0554. IR: 3585, 3094, 2925, 2322, 1873, 1718, 1600, 1567, 1446, 1287, 1267, 1190, 1156, 1025, 961, 938,

834, 748, 705, 662 cm^{-1} .

3.2 Characterization for hydroxylation of simple arenes

4-Phenylphenol **3a**

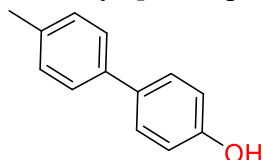


Following the general procedure B afforded the product as a pale yellow solid (56 mg, 82% yield); Chromatography column, pentane / EA = 5:1.

Scale-up experiment: 4mmol scale: Under N_2 atmosphere, a 50 mL flat-bottom quartz vial equipped with a magnetic stir bar was charged with aryl thianthrenium salts **2a** (4 mmol, 1.0 equiv.), 4-Oxo-TEMPO (32 mmol, 8 equiv.) and DMF (10 mL). The tube was sealed, and the mixture was stirred at room temperature under UV-light (254 nm, 144 W) for 24 h before quenching with aqueous saturated NaHCO_3 and dilution with EtOAc. The organic layer was washed with brine, dried using Na_2SO_4 , filtered, and concentrated *in vacuo*, to give the crude product **4a**, which was purified by column chromatography on silica gel. pentane / EA = 5:1. (446 mg, 66% yield)

^1H NMR (600 MHz, CDCl_3) δ 7.55 (d, $J = 7.4$ Hz, 2H), 7.49 (d, $J = 8.5$ Hz, 2H), 7.42 (t, $J = 7.7$ Hz, 2H), 7.31 (t, $J = 7.4$ Hz, 1H), 6.91 (d, $J = 8.5$ Hz, 2H), 4.88 (s, 1H). ^{13}C NMR (151 MHz, CDCl_3) δ 155.2 (s), 140.9 (s), 134.2 (s), 128.9 (s), 128.5 (s), 126.9 (s), 126.8 (s), 115.8 (s). The characterization of this compound is in accordance with the literature.⁴

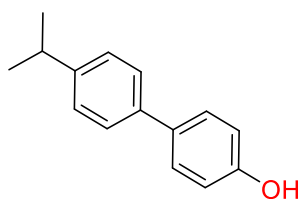
4'-methyl-[1,1'-biphenyl]-4-ol **3b**



Following the general procedure B afforded the product as a yellow solid (73 mg, 82% yield); Chromatography column, pentane / EA = 4:1.

^1H NMR (600 MHz, CDCl_3) δ 7.47 (d, $J = 8.5$ Hz, 2H), 7.45 (d, $J = 8.0$ Hz, 2H), 7.24 (d, $J = 7.9$ Hz, 2H), 6.90 (d, $J = 8.5$ Hz, 2H), 4.99 (s, 1H), 2.40 (s, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 155.0 (s), 138.0 (s), 136.5 (s), 134.1 (s), 129.6 (s), 128.3 (s), 126.7 (s), 115.7 (s), 21.2 (s). The characterization of this compound is in accordance with the literature.⁵

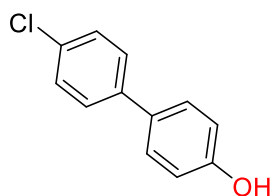
4'-isopropyl-[1,1'-biphenyl]-4-ol **3c**



Following the general procedure B afforded the product as a yellow solid (60 mg, 71% yield); Chromatography column, pentane / EA = 4:1.

^1H NMR (600 MHz, CDCl_3) δ 7.48 (d, $J = 3.9$ Hz, 2H), 7.47 (d, $J = 4.2$ Hz, 2H), 7.29 (d, $J = 8.1$ Hz, 2H), 6.90 (d, $J = 8.5$ Hz, 2H), 4.92 (s, 1H), 2.95 (hept, $J = 6.9$ Hz, 1H), 1.30 (d, $J = 6.9$ Hz, 6H). ^{13}C NMR (151 MHz, CDCl_3) δ 155.0 (s), 147.6 (s), 138.4 (s), 134.2 (s), 128.4 (s), 126.9 (s), 126.8 (s), 115.7 (s), 33.9 (s), 24.2 (s). HRMS-APCI (m/z): Calculated for $\text{C}_{15}\text{H}_{16}\text{O}$: [M] 212.1196; Found, 212.1202. IR: 3376, 2962, 2872, 1716, 1609, 1497, 1447, 1367, 1226, 1173, 1108, 1024, 819, 921, 753, 720, 685 cm^{-1} .

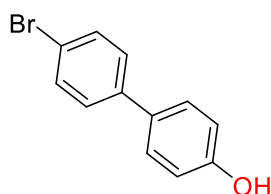
4'-chloro-[1,1'-biphenyl]-4-ol **3d**



Following the general procedure B afforded the product as a yellow solid (51 mg, 62% yield); Chromatography column, pentane / EA = 4:1.

^1H NMR (600 MHz, CDCl_3) δ 7.46 (d, $J = 8.4$ Hz, 4H), 7.38 (d, $J = 8.2$ Hz, 2H), 6.92 (s, 2H). ^{13}C NMR (151 MHz, CDCl_3) δ 155.3 (s), 139.3 (s), 132.8 (s), 132.8 (s), 128.9 (s), 128.4 (s), 128.0 (s), 116.2 (s). The characterization of this compound is in accordance with the literature.⁶

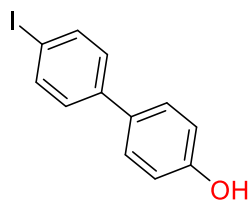
4'-bromo-[1,1'-biphenyl]-4-ol **3e**



Following the general procedure B afforded the product as a yellow solid (58 mg, 58% yield); Chromatography column, pentane / EA = 5:1.

^1H NMR (600 MHz, CDCl_3) δ 7.53 (d, $J = 8.5$ Hz, 2H), 7.44 (d, $J = 8.5$ Hz, 2H), 7.40 (d, $J = 8.5$ Hz, 2H), 6.91 (d, $J = 8.5$ Hz, 2H), 5.02 (s, 1H). ^{13}C NMR (151 MHz, CDCl_3) δ 155.5 (s), 139.8 (s), 132.9 (s), 131.9 (s), 128.4 (s), 128.4 (s), 121.0 (s), 115.9 (s). The characterization of this compound is in accordance with the literature.⁷

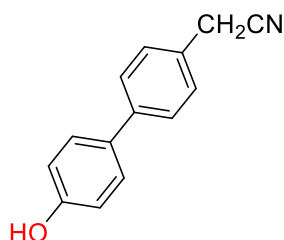
4'-iodo-[1,1'-biphenyl]-4-ol **3f**



Following the general procedure B afforded the product as a white solid (28 mg, 24% yield); Chromatography column, pentane / EA = 4:1.

^1H NMR (600 MHz, CDCl_3) δ 7.73 (d, J = 8.5 Hz, 2H), 7.43 (d, J = 8.6 Hz, 2H), 7.27 (d, J = 8.5 Hz, 2H), 6.90 (d, J = 8.6 Hz, 2H), 4.88 (s, 1H). ^{13}C NMR (151 MHz, CDCl_3) δ 137.8 (s), 128.6 (s), 128.2 (s), 115.8 (s). The characterization of this compound is in accordance with the literature.⁸

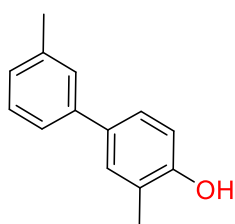
2-(4'-hydroxy-[1,1'-biphenyl]-4-yl) acetonitrile **3g**



Following the general procedure B afforded the product as a pale yellow solid (54 mg, 64% yield); Chromatography column, pentane / EA = 2:1.

^1H NMR (600 MHz, CD_3OD) δ 7.56 (d, J = 8.2 Hz, 2H), 7.45 (d, J = 8.6 Hz, 2H), 7.37 (d, J = 8.2 Hz, 2H), 6.86 (d, J = 8.6 Hz, 2H), 3.89 (s, 2H). ^{13}C NMR (151 MHz, CD_3OD) δ 158.4 (s), 142.1 (s), 132.9 (s), 130.2 (s), 129.4 (s), 129.0 (s), 128.0 (s), 119.7 (s), 116.7 (s), 23.1 (s). The characterization of this compound is in accordance with the literature.⁹

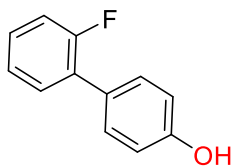
3,3'-dimethyl-[1,1'-biphenyl]-4-ol **3h**



Following the general procedure B afforded the product as a pale yellow solid (41 mg, 52% yield); Chromatography column, pentane / EA = 5:1.

^1H NMR (600 MHz, CDCl_3) δ 7.38 (s, 2H), 7.37 (d, J = 8.0 Hz, 1H), 7.32 (t, J = 7.6 Hz, 2H), 7.14 (d, J = 7.3 Hz, 1H), 6.85 (d, J = 8.2 Hz, 1H), 4.89 (s, 1H), 2.43 (s, 3H), 2.34 (s, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 153.4 (s), 141.1 (s), 134.2 (s), 123.0 (s), 128.7 (s), 127.7 (s), 127.5 (s), 125.9 (s), 124.1 (s), 124.0 (s), 115.3 (s), 21.7 (s), 16.0 (s). HRMS-APCI (m/z): Calculated for $\text{C}_{14}\text{H}_{15}\text{O}$: $[\text{M} + \text{H}]^+$ 199.1039; Found, 199.1112. IR: 3891, 3410, 3027, 2921, 2859, 2335, 1873, 1606, 1509, 1480, 1387, 1305, 1239, 1183, 1116, 881, 820, 738, 698 cm^{-1} .

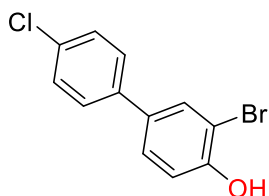
2'-fluoro-[1,1'-biphenyl]-4-ol **3i**



Following the general procedure B afforded the product as a yellow solid (55 mg, 73% yield); Chromatography column, pentane / EA = 5:1.

^1H NMR (600 MHz, CDCl_3) δ 7.45 (d, J = 7.8 Hz, 2H), 7.40 (t, J = 7.6 Hz, 1H), 7.28 (d, J = 6.0 Hz, 1H), 7.19 (t, J = 7.4 Hz, 1H), 7.16 – 7.10 (m, 1H), 6.92 (d, J = 8.2 Hz, 2H), 4.95 (s, 1H). ^{19}F NMR (565 MHz, CDCl_3) δ -118.25 (s, 1F). ^{13}C NMR (151 MHz, CDCl_3) δ 159.9 (d, J = 247.0 Hz), 155.3 (s), 130.6 (d, J = 3.5 Hz), 130.5 (d, J = 3.2 Hz), 128.8 (d, J = 13.3 Hz), 128.6 (d, J = 8.5 Hz), 124.4 (d, J = 3.6 Hz), 116.19 (d, J = 22.6 Hz), 115.5 (s). (At least one line overlapped). HRMS-APCI (m/z): Calculated for $\text{C}_{12}\text{H}_9\text{OF}$: [M] 188.0632; Found, 188.0634. IR: 3425, 2925, 1709, 1606, 1517, 1478, 1448, 1363, 1228, 1102, 1035, 1007, 942, 820, 753 cm^{-1} .

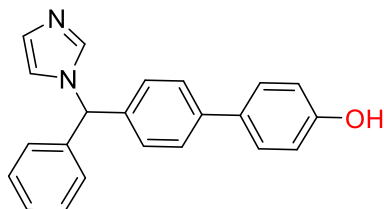
3-bromo-4'-chloro-[1,1'-biphenyl]-4-ol **3j**



Following the general procedure B afforded the product as a yellow solid (58 mg, 51% yield); Chromatography column, pentane / EA = 4:1.

^1H NMR (600 MHz, CDCl_3) δ 7.66 (d, J = 2.0 Hz, 1H), 7.43 (d, J = 8.5 Hz, 2H), 7.41 (dd, J = 8.5, 2.0 Hz, 1H), 7.38 (d, J = 8.5 Hz, 2H), 7.09 (d, J = 8.4 Hz, 1H), 5.56 (s, 1H). ^{13}C NMR (151 MHz, CDCl_3) δ 152.1 (s), 138.0 (s), 134.3 (s), 133.5 (s), 130.5 (s), 129.1 (s), 128.1 (s), 127.9 (s), 116.6 (s), 110.9 (s). HRMS-APCI (m/z): Calculated for $\text{C}_{12}\text{H}_8\text{OBrCl}$: [M] 283.9419; Found, 283.9413. IR: 3394, 3060, 2974, 1898, 1703, 1600, 1477, 1384, 1282, 1183, 1093, 1012, 956, 884, 815, 751, 680 cm^{-1} .

4'-((1H-imidazol-1-yl) (phenyl)methyl)-[1,1'-biphenyl]-4-ol **3k**

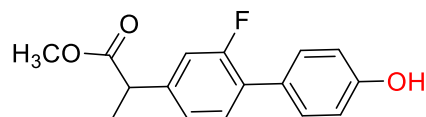


Following the general procedure B afforded the product as a white solid (81 mg, 62% yield); Chromatography column, DCM / EA = 1:1.

^1H NMR (600 MHz, DMSO-d_6) δ 9.59 (s, 1H), 7.67 (s, 1H), 7.59 (d, J = 8.3 Hz, 2H), 7.49 (d, J = 8.6 Hz, 2H), 7.40 (t, J = 7.4 Hz, 2H), 7.35 (t, J = 7.3 Hz, 1H), 7.17 (t, J = 7.3 Hz, 4H), 7.13 (s, 1H), 6.97 (s, 1H), 6.88 (s, 1H), 6.84 (d, J = 8.6 Hz, 2H). ^{13}C NMR (151 MHz, DMSO-d_6) δ 157.3 (s), 140.0 (s), 139.8 (s), 137.9 (s), 137.2 (s), 130.2 (s),

128.7 (s), 128.7 (s), 128.3 (s), 128.0 (s), 127.8 (s), 127.8 (s), 126.2 (s), 119.2 (s), 115.8 (s), 63.1 (s). HRMS-ESI (m/z): Calculated for C₂₂H₁₉ON₂: [M + H]⁺ 327.1492; Found, 327.1491. IR: 3116, 3029, 2934, 2815, 2676, 2606, 2159, 1661, 1605, 1495, 1452, 1387, 1269, 1231, 1173, 1107, 1078, 1025, 921, 826, 795, 736, 702, 660 cm⁻¹.

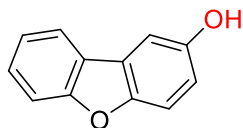
Methyl 2-(2-fluoro-4'-hydroxy-[1,1'-biphenyl]-4-yl) propanoate **3l**



Following the general procedure B afforded the product as a white solid (61 mg, 56% yield); Chromatography column, pentane / EA = 4:1.

¹H NMR (600 MHz, CDCl₃) δ 7.40 (d, *J* = 7.6 Hz, 2H), 7.34 (t, *J* = 8.0 Hz, 1H), 7.11 (t, *J* = 10.1 Hz, 2H), 6.88 (d, *J* = 8.4 Hz, 2H), 3.77 (q, *J* = 7.2 Hz, 1H), 3.72 (s, 3H), 1.54 (d, *J* = 7.2 Hz, 3H). ¹⁹F NMR (565 MHz, CDCl₃) δ -117.67 (s, 1F). ¹³C NMR (151 MHz, CDCl₃) δ 175.1 (s), 159.7 (d, *J* = 247.9 Hz), 155.6 (s), 141.1 (d, *J* = 7.7 Hz), 130.6 (d, *J* = 3.7 Hz), 130.3 (d, *J* = 3.2 Hz), 127.9 (s), 127.6 (d, *J* = 13.7 Hz), 123.6 (d, *J* = 2.6 Hz), 115.6 (s), 115.3 (d, *J* = 23.9 Hz), 52.5 (s), 45.0 (s), 18.4 (s). HRMS-APCI (m/z): Calculated for C₁₆H₁₆OF: [M + H]⁺ 275.1078; Found, 275.1076. IR: 3756, 3366, 2963, 1706, 1611, 1525, 1493, 1433, 1334, 1274, 1210, 1170, 1073, 1009, 968, 918, 871, 825, 786, 715 cm⁻¹.

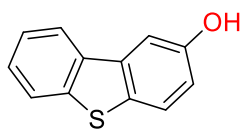
dibenzo[b,d]furan-2-ol **3m**



Following the general procedure B afforded the product as a white solid (53 mg, 72% yield); Chromatography column, pure DCM.

¹H NMR (600 MHz, CDCl₃) δ 7.88 (d, *J* = 7.6 Hz, 1H), 7.54 (d, *J* = 8.2 Hz, 1H), 7.44 (dd, *J* = 15.5, 8.1 Hz, 2H), 7.38 (s, 1H), 7.32 (t, *J* = 7.4 Hz, 1H), 6.96 (d, *J* = 10.1 Hz, 1H), 4.86 (s, 1H). ¹³C NMR (151 MHz, CDCl₃) δ 157.1 (s), 151.6 (s), 151.1 (s), 127.4 (s), 125.2 (s), 124.3 (s), 122.6 (s), 120.8 (s), 115.4 (s), 112.2 (s), 111.9 (s), 106.4 (s). The characterization of this compound is in accordance with the literature.¹⁰

dibenzo[b,d]thiophen-2-ol **3n**

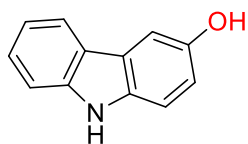


Following the general procedure B afforded the product as a white solid (51 mg, 64% yield); Chromatography column, pentane / EA = 4:1.

¹H NMR (600 MHz, CDCl₃) δ 8.05 (dd, *J* = 6.7, 2.0 Hz, 1H), 7.83 (dd, *J* = 6.8, 1.8 Hz, 1H), 7.69 (d, *J* = 8.5 Hz, 1H), 7.59 (d, *J* = 2.4 Hz, 1H), 7.48 – 7.37 (m, 2H), 7.02 (dd, *J* = 8.5, 2.4 Hz, 1H), 5.02 (s, 1H). ¹³C NMR (151 MHz, CDCl₃) δ 153.5 (s), 140.8 (s),

137.0 (s), 135.3 (s), 131.6 (s), 127.0 (s), 124.3 (s), 123.7 (s), 123.1 (s), 121.8 (s), 116.1 (s), 107.7 (s). HRMS-APCI (m/z): Calculated for C₁₂H₈OS: [M] 200.0290; Found, 200.0285. IR: 3797, 3485, 3388, 3282, 2923, 2854, 1708, 1602, 1466, 1427, 1330, 1183, 1068, 1020, 892, 850, 807, 756, 725, 658 cm⁻¹.

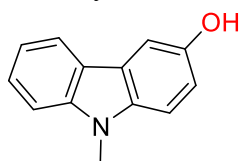
9H-carbazol-3-ol **3o**



Following the general procedure B afforded the product as a white solid (48 mg, 66% yield); Chromatography column, pentane / EA = 2:1.

¹H NMR (600 MHz, DMSO-d₆) δ 10.86 (s, 1H), 8.89 (s, 1H), 7.97 (d, *J* = 7.7 Hz, 1H), 7.41 (d, *J* = 2.2 Hz, 1H), 7.39 (d, *J* = 8.1 Hz, 1H), 7.33 – 7.29 (m, 1H), 7.27 (d, *J* = 8.6 Hz, 1H), 7.06 (t, *J* = 7.8 Hz, 1H), 6.88 (dd, *J* = 8.6, 2.3 Hz, 1H). ¹³C NMR (151 MHz, DMSO-d₆) δ 150.4 (s), 140.4 (s), 133.7 (s), 125.2 (s), 123.0 (s), 122.3 (s), 120.1 (s), 117.7 (s), 115.0 (s), 111.3 (s), 110.8 (s), 104.8 (s). The characterization of this compound is in accordance with the literature.¹¹

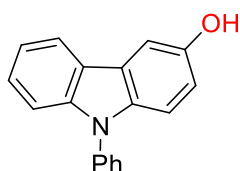
9-methyl-9H-carbazol-3-ol **3p**



Following the general procedure B afforded the product as a yellow solid (47 mg, 60% yield); Chromatography column, pentane / EA = 2:1.

¹H NMR (600 MHz, CDCl₃) δ 8.02 (d, *J* = 7.6 Hz, 1H), 7.58 (s, 1H), 7.49 (t, *J* = 7.5 Hz, 1H), 7.38 (d, *J* = 8.1 Hz, 1H), 7.29 (s, 1H), 7.21 (t, *J* = 7.2 Hz, 1H), 7.10 (s, 1H), 3.82 (s, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 148.0 (s), 141.0 (s), 135.7 (s), 125.2 (s), 122.9 (s), 121.8 (s), 119.8 (s), 117.7 (s), 115.4 (s), 108.6 (s), 107.9 (s), 106.8 (s), 28.6 (s). The characterization of this compound is in accordance with the literature.¹²

9-phenyl-9H-carbazol-3-ol **3q**

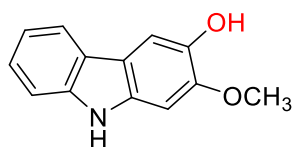


Following the general procedure B afforded the product as a yellow solid (61 mg, 59% yield); Chromatography column, pentane / EA = 2:1.

¹H NMR (600 MHz, DMSO-d₆) δ 9.17 (s, 1H), 8.12 (d, *J* = 7.6 Hz, 1H), 7.65 (t, *J* = 7.3 Hz, 2H), 7.58 (d, *J* = 7.6 Hz, 2H), 7.55 (s, 1H), 7.49 (t, *J* = 7.2 Hz, 1H), 7.42 – 7.31 (m, 2H), 7.21 (dd, *J* = 12.7, 7.6 Hz, 2H), 6.92 (d, *J* = 20.5 Hz, 1H). ¹³C NMR (151 MHz, DMSO-d₆) δ 151.6 (s), 140.4 (s), 137.3 (s), 134.1 (s), 130.1 (s), 127.1 (s), 126.3 (s),

126.0 (s), 123.5 (s), 122.6 (s), 120.5 (s), 119.4 (s), 115.4 (s), 110.1 (s), 109.4 (s), 105.2 (s). HRMS-APCI (m/z): Calculated for C₁₈H₁₄ON: [M + H]⁺ 260.1070; Found, 260.1073. IR: 3315, 2934, 2862, 1627, 1593, 1485, 1450, 1361, 1313, 1234, 1190, 1106, 1024, 931, 875, 803, 744, 696 cm⁻¹.

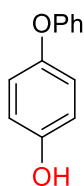
2-methoxy-9H-carbazol-3-ol **3r**



Following the general procedure B afforded the product as a yellow solid (29 mg, 34% yield); Chromatography column, pentane / EA = 2:1.

¹H NMR (600 MHz, DMSO-d₆) δ 10.80 (s, 1H), 8.43 (s, 1H), 7.87 (d, *J* = 7.7 Hz, 1H), 7.42 (s, 1H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.22 (t, *J* = 7.5 Hz, 1H), 7.03 (t, *J* = 7.4 Hz, 1H), 6.97 (s, 1H), 3.86 (s, 3H). ¹³C NMR (151 MHz, DMSO-d₆) δ 148.1 (s), 140.7 (s), 139.6 (s), 134.0 (s), 123.5 (s), 122.7 (s), 119.1 (s), 117.8 (s), 114.8 (s), 110.5 (s), 105.5 (s), 94.4 (s), 55.7 (s). HRMS-APCI (m/z): Calculated for C₁₃H₁₂O₂N: [M + H]⁺ 214.0863; Found, 214.0870. IR: 3530, 3391, 2923, 2853, 1715, 1613, 1489, 1453, 1342, 1307, 1178, 1147, 1025, 921, 862, 820, 743, 692 cm⁻¹.

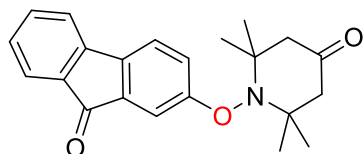
4-Phenoxyphenol **3s**



Following the general procedure B afforded the product as a brown solid (32 mg, 43% yield); Chromatography column, pentane / EA = 4:1.

¹H NMR (600 MHz, CDCl₃) δ 7.30 (dd, *J* = 8.5, 7.5 Hz, 2H), 7.05 (t, *J* = 7.4 Hz, 1H), 6.95 (t, *J* = 8.9 Hz, 4H), 6.82 (d, *J* = 8.9 Hz, 2H), 4.79 (s, 1H). ¹³C NMR (151 MHz, CDCl₃) δ 158.4 (s), 151.5 (s), 150.2 (s), 129.6 (s), 122.4 (s), 121.0 (s), 117.5 (s), 117.0 (s). The characterization of this compound is in accordance with the literature.¹³

2,2,6,6-tetramethyl-1-((9-oxo-9H-fluoren-2-yl) oxy) piperidin-4-one **3t**



Following the general procedure B afforded the product as a yellow solid (52 mg, 37% yield); Chromatography column, pentane / EA = 4:1.

¹H NMR (600 MHz, CDCl₃) δ 7.69 (s, 1H), 7.63 (d, *J* = 7.3 Hz, 1H), 7.46 (td, *J* = 7.3, 1.0 Hz, 1H), 7.43 (d, *J* = 7.2 Hz, 1H), 7.41 (d, *J* = 8.2 Hz, 1H), 7.26 (s, 1H), 7.22 (td, *J* = 7.3, 1.2 Hz, 1H), 2.77 (d, *J* = 13.1 Hz, 2H), 2.39 (d, *J* = 13.1 Hz, 2H), 1.31 (s, 6H), 1.23 (s, 6H). ¹³C NMR (151 MHz, CDCl₃) δ 207.1 (s), 194.1 (s), 164.3 (s), 145.0 (s),

137.4 (s), 135.8 (s), 135.0 (s), 134.5 (s), 128.0 (s), 124.4 (s), 121.1 (s), 119.6 (s), 119.3 (s), 110.7 (s), 63.9 (s), 53.3 (s), 31.9 (s), 23.1 (s). HRMS-ESI (m/z): Calculated for $C_{22}H_{23}O_3NNa$: $[M + Na]^+$ 372.1570; Found, 372.1569. IR: 3417, 3067, 2975, 2927, 2319, 1713, 1600, 1451, 1368, 1298, 1223, 1130, 1072, 947, 922, 886, 850, 761, 732, 671 cm^{-1} .

4. X-ray Experiment

Crystallization of compound **3t** (C₂₂H₂₃N₁O₃) from ethyl acetate/hexane at room temperature gave monoclinic crystals of space group P21/n (14) suitable for single crystal X-ray structure determination. Cell constants $a = 8.2069(3)$, $b = 26.8100(11)$, $c = 8.7494(4)$ Å, $\alpha = \gamma = 90^\circ$, $\beta = 105.943(2)$, $Z = 4$, and a molecular weight of $M_r = 349.41$ result in a density of 1.254 g cm^{-3} and a linear absorption coefficient of $\mu = 0.083 \text{ mm}^{-1}$ for MoK α radiation ($\lambda = 0.71073$ Å). 29640 reflections covering the range $-11 \leq h \leq 10$, $-38 \leq k \leq 38$, and $-11 \leq l \leq 12$ ($\Theta_{\text{max}} = 30.7^\circ$) were collected (ϕ and ω scans) at 293 K on an Bruker APEX-II CCD diffractometer equipped with a graphite-monochromator and merged to give 5740 independent diffraction data ($R_{\text{int}} = 0.0364$) of which 3827 with $I > 2\sigma(I)$. The data set was corrected for absorption effects using the multi scan absorption correction method SADABS¹⁴ ($T_{\text{min}} = 0.6788$, $T_{\text{max}} = 0.761$). The structure was solved by intrinsic phasing using the ShelXT 2018/2 structure solution program¹⁵ and refined against F^2 on all data by full-matrix least-squares methods using ShelXL-2018/3¹⁶ and ShelXle GUI.¹⁷ 3827 reflexions were used in the final full-matrix least squares refinement including 239 parameters. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were placed at idealised positions and refined isotropically using the riding model. Refinement converged at $R1 = 0.0507$ for the observed data and $wR2 = 0.1491$ for all data ($w = 1/[\sigma^2(\text{Fo}^2) + (0.0672\text{P})^2 + 0.3135\text{P}]$ where $\text{P} = (\text{Fo}^2 + 2\text{Fc}^2)/3$), a residual electron density of $-0.198/+0.270 \text{ e}\text{\AA}^{-3}$, and a final goodness of fit of 1.026.

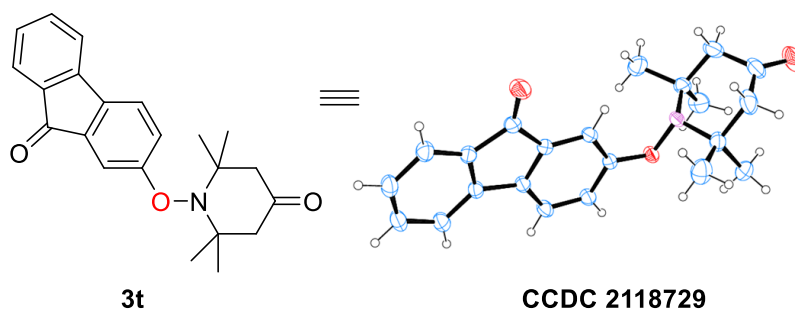


Figure S2 X-ray for compound **3t**

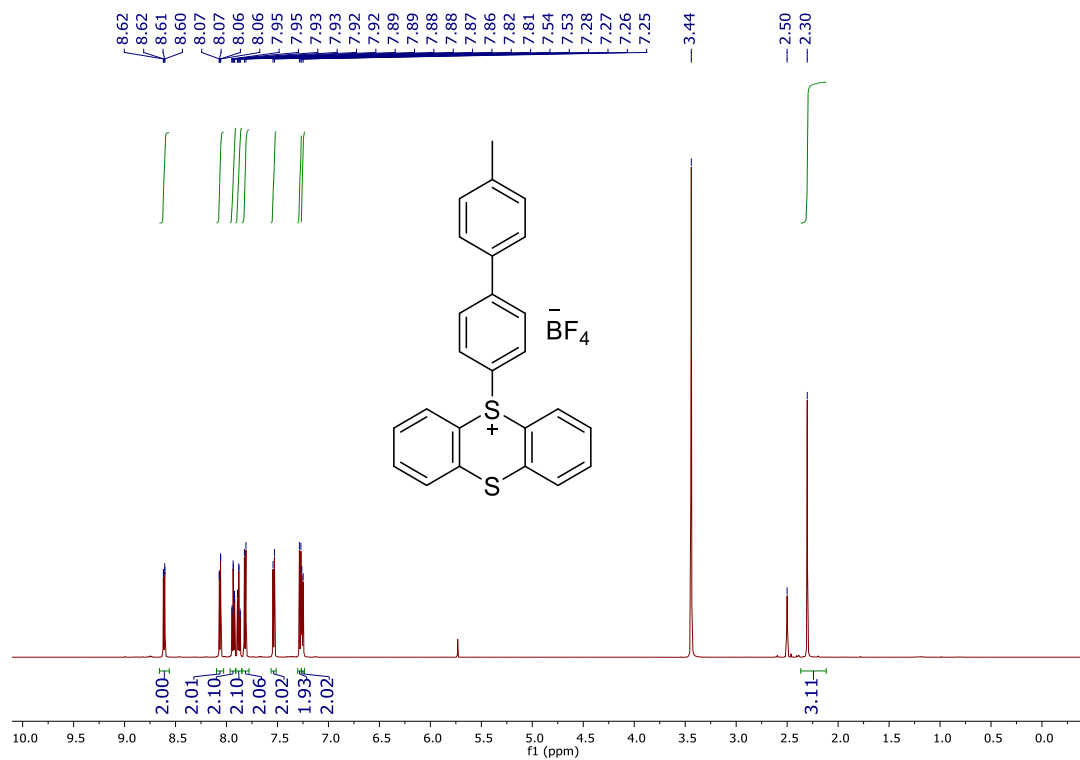
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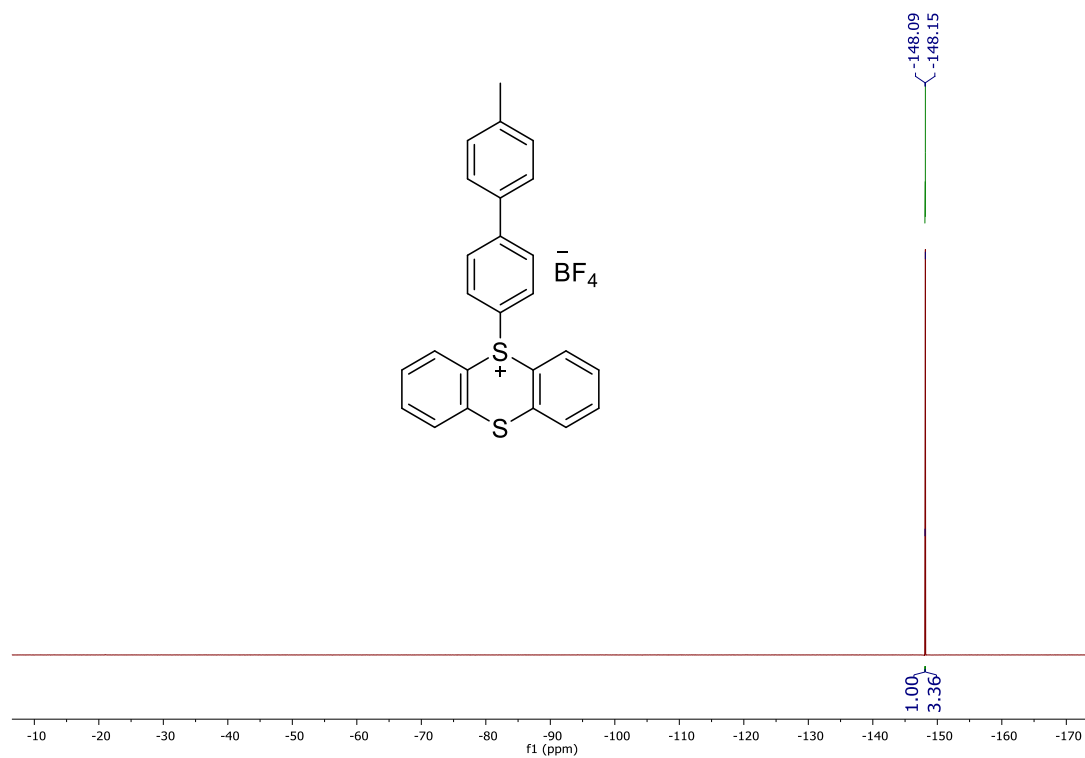
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- (17) Hübschle, C. B.; Sheldrick, G. M.; Dittrich, B. ShelXle: a Qt graphical user interface for SHELXL. *J. Appl. Cryst.* **2011**, *44*, 1281.

6. Copies of ^1H , ^{13}C and ^{19}F NMR Spectra

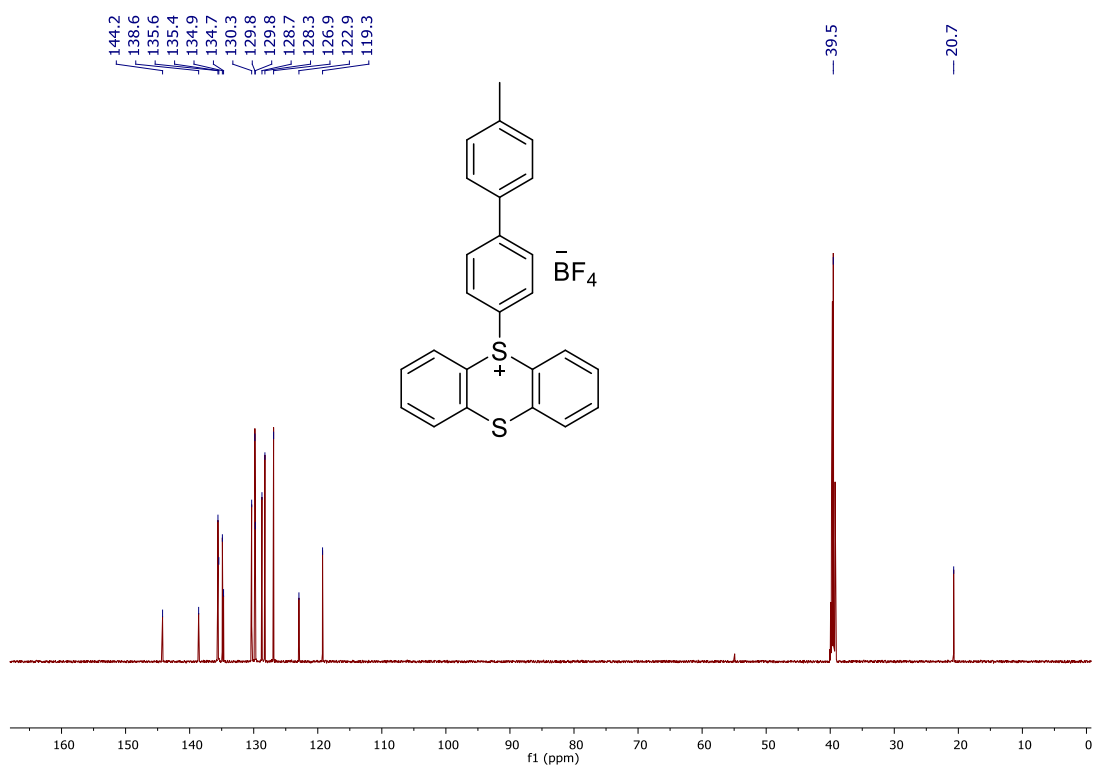
^1H NMR spectrum of **2b**



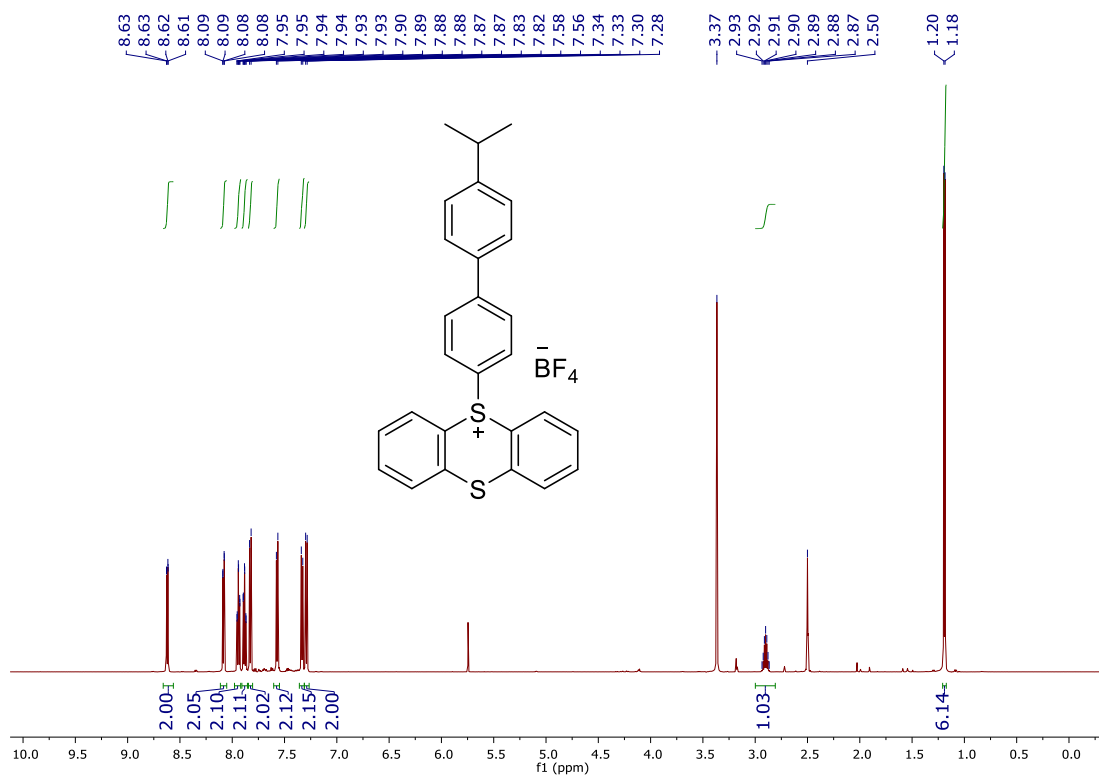
^{19}F NMR spectrum of **2b**



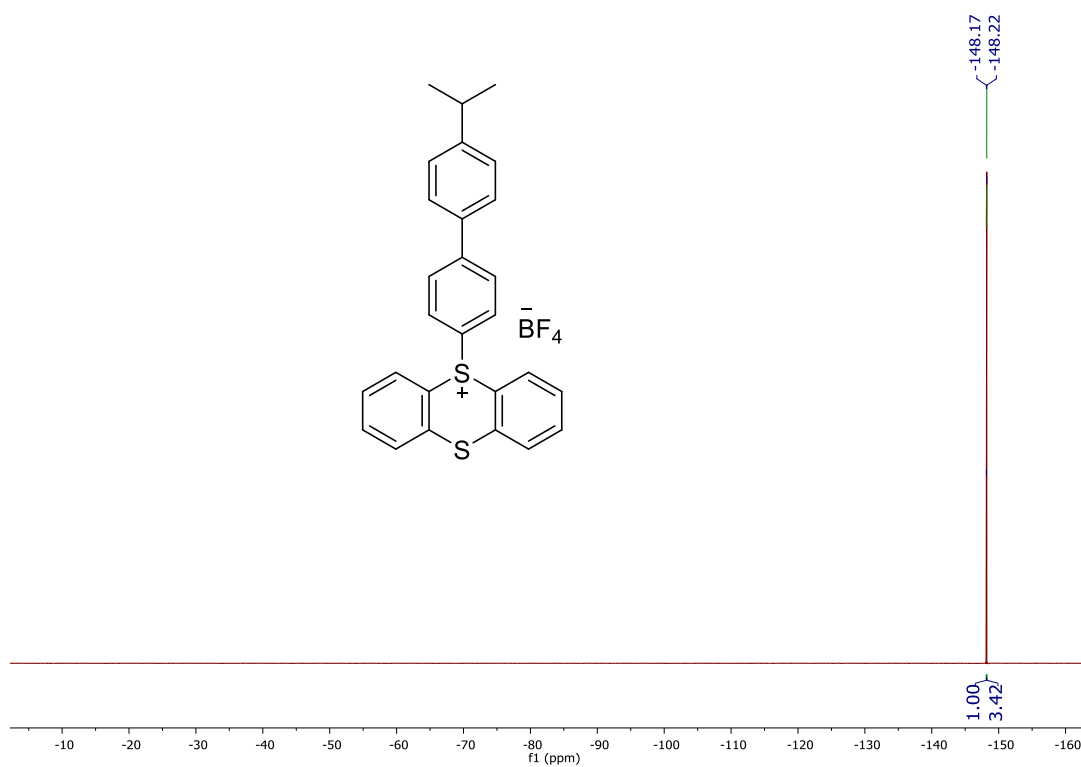
¹³C NMR spectrum of 2b



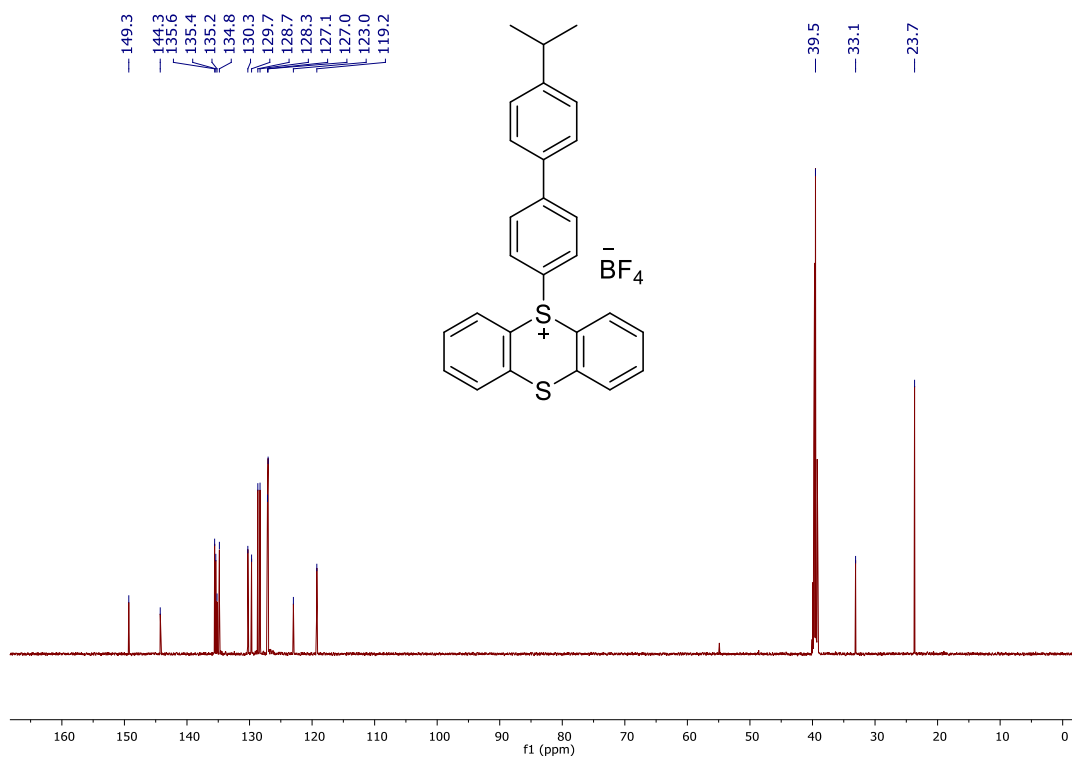
¹H NMR spectrum of 2c



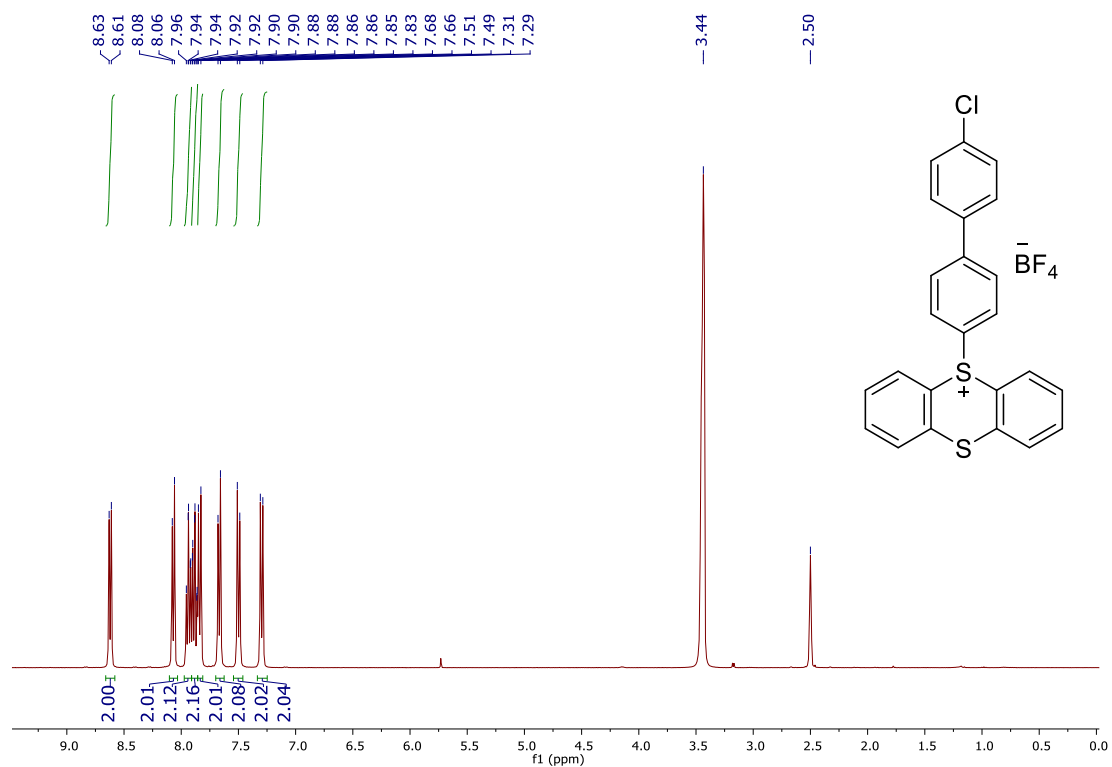
¹⁹F NMR spectrum of **2c**



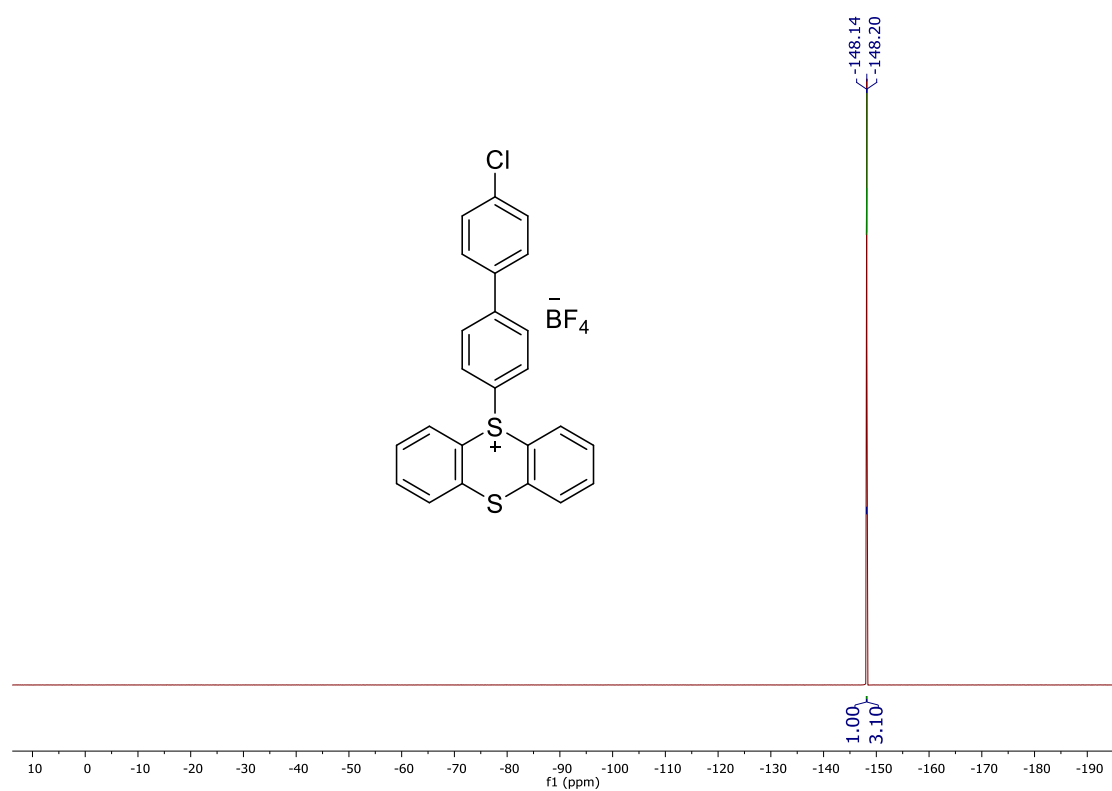
¹³C NMR spectrum of **2c**



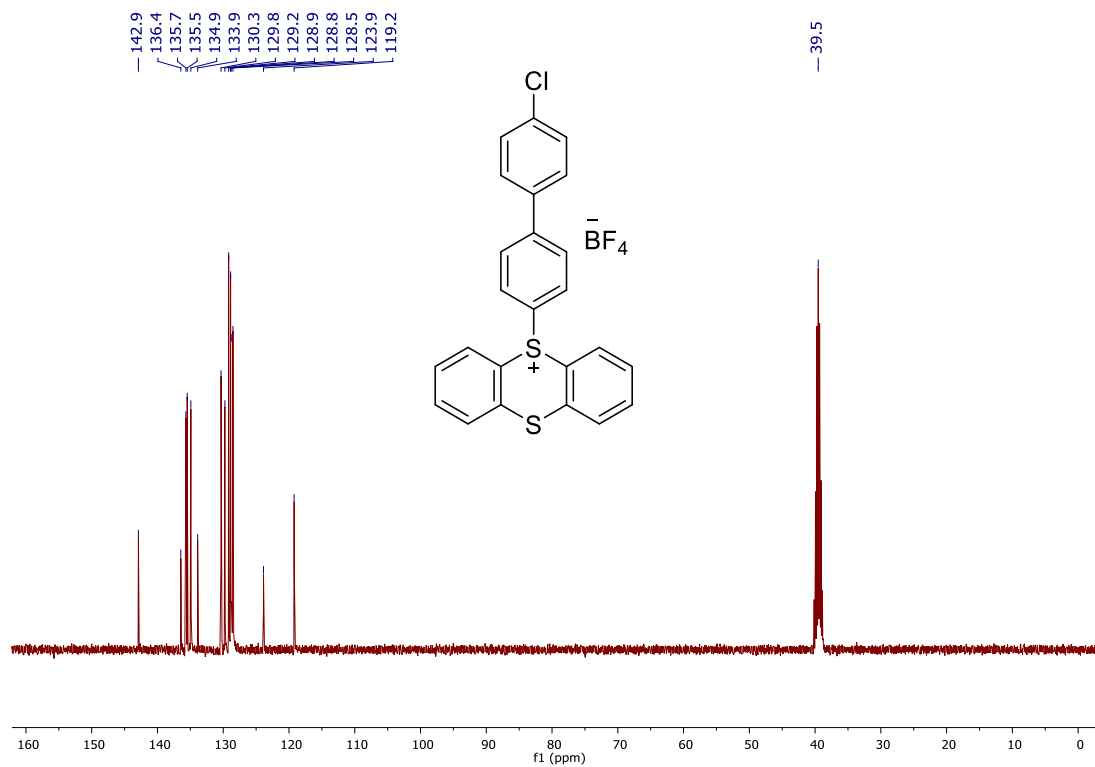
^1H NMR spectrum of **2d**



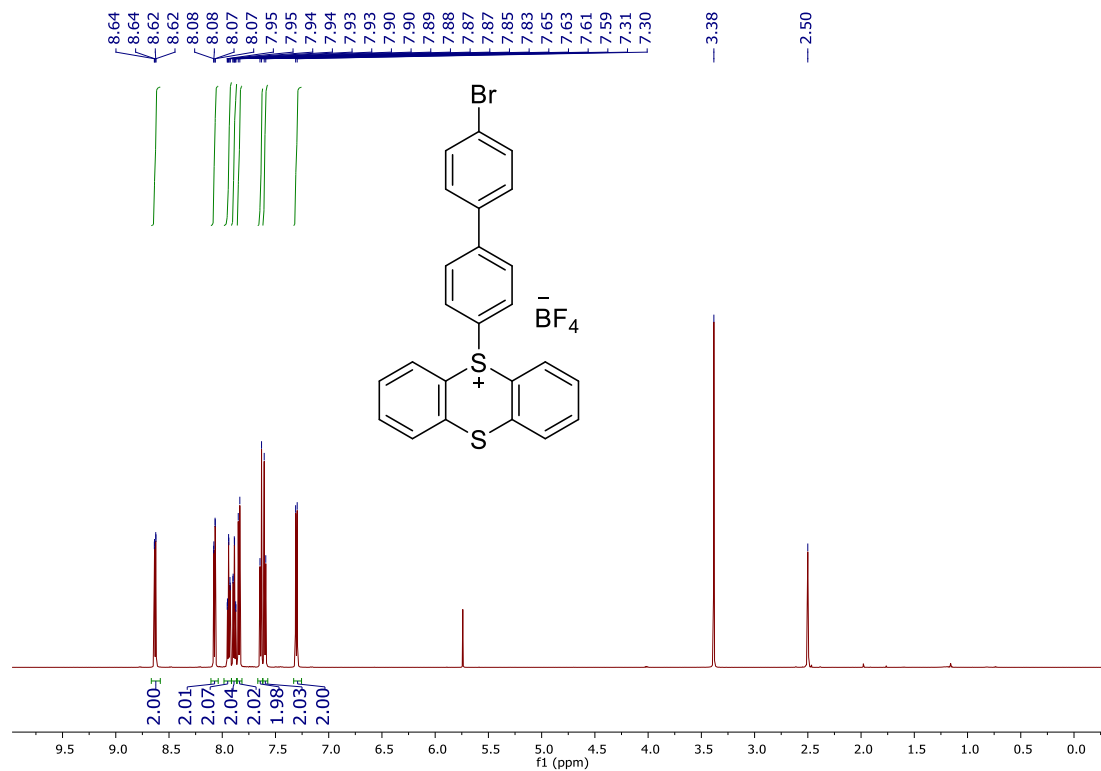
^{19}F NMR spectrum of **2d**



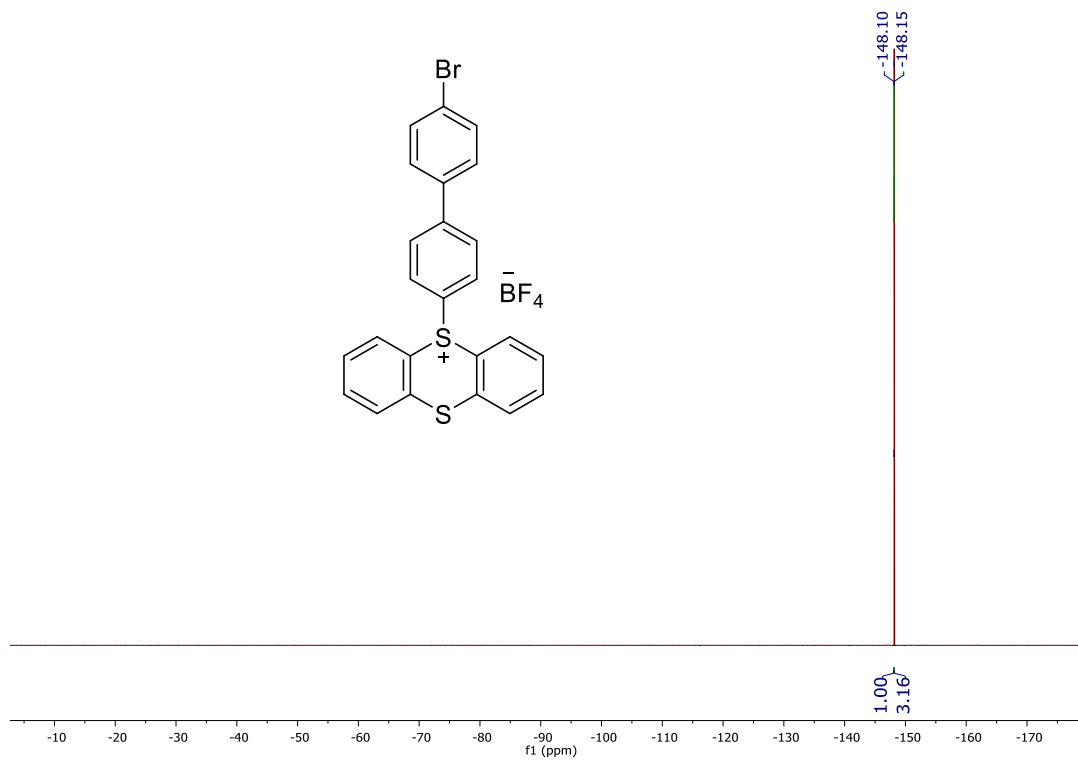
¹³C NMR spectrum of 2d



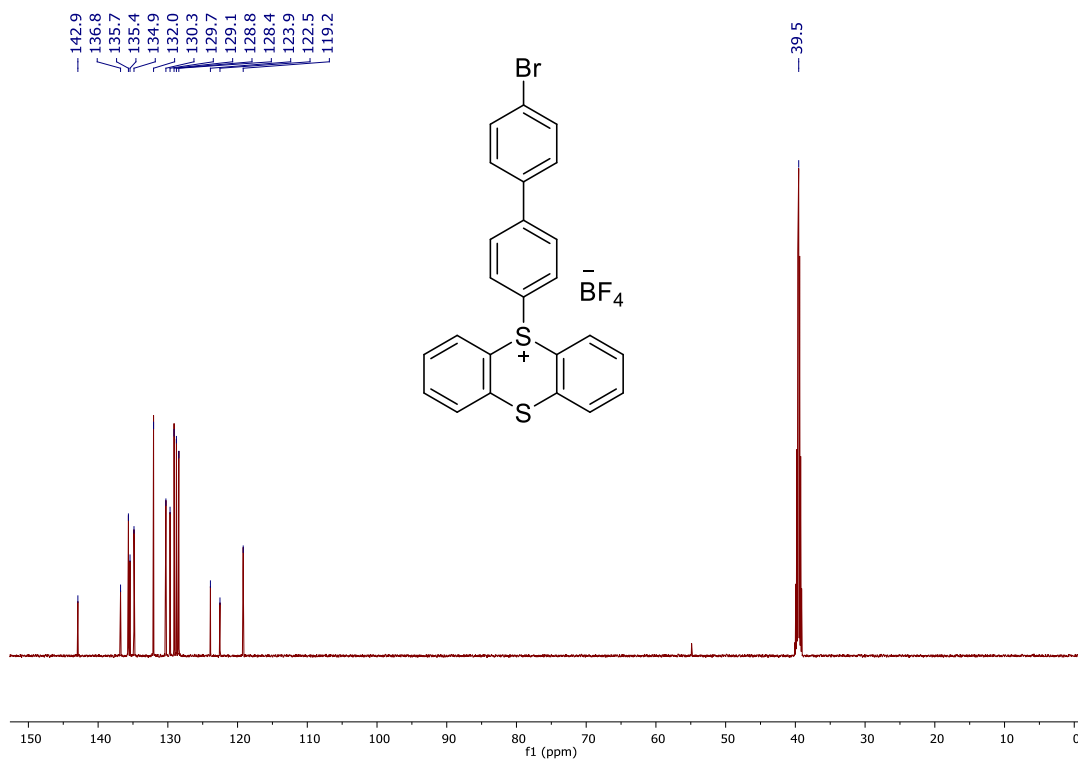
¹H NMR spectrum of 2e



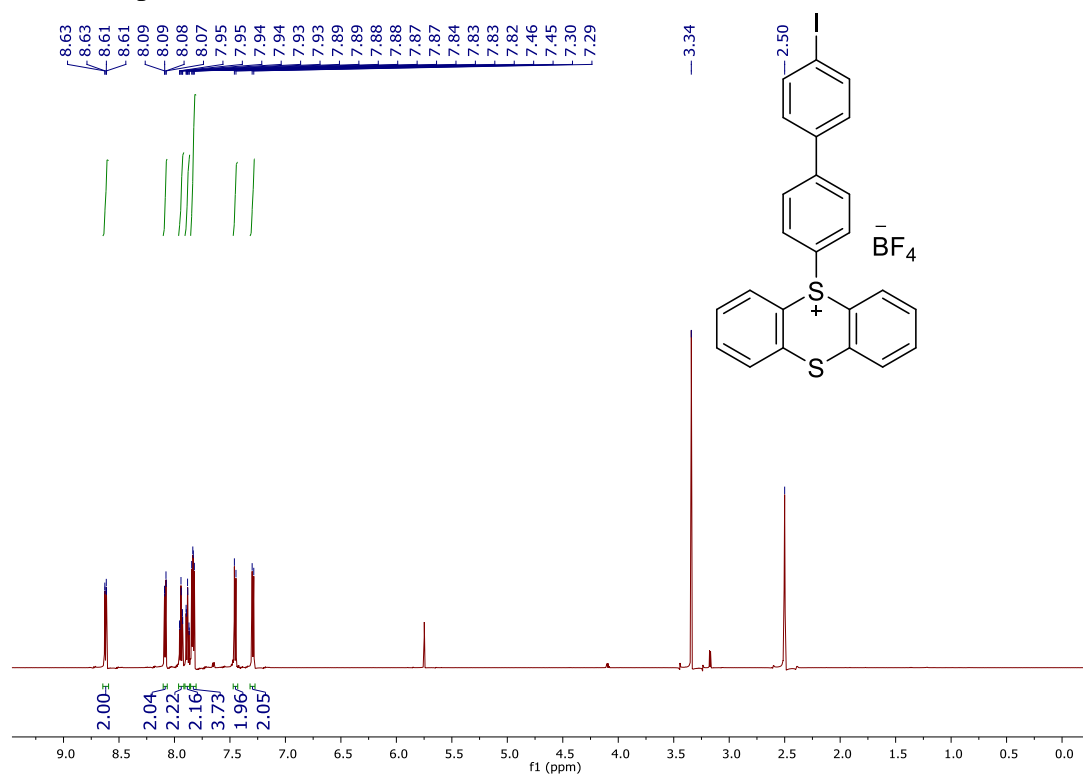
¹⁹F NMR spectrum of **2e**



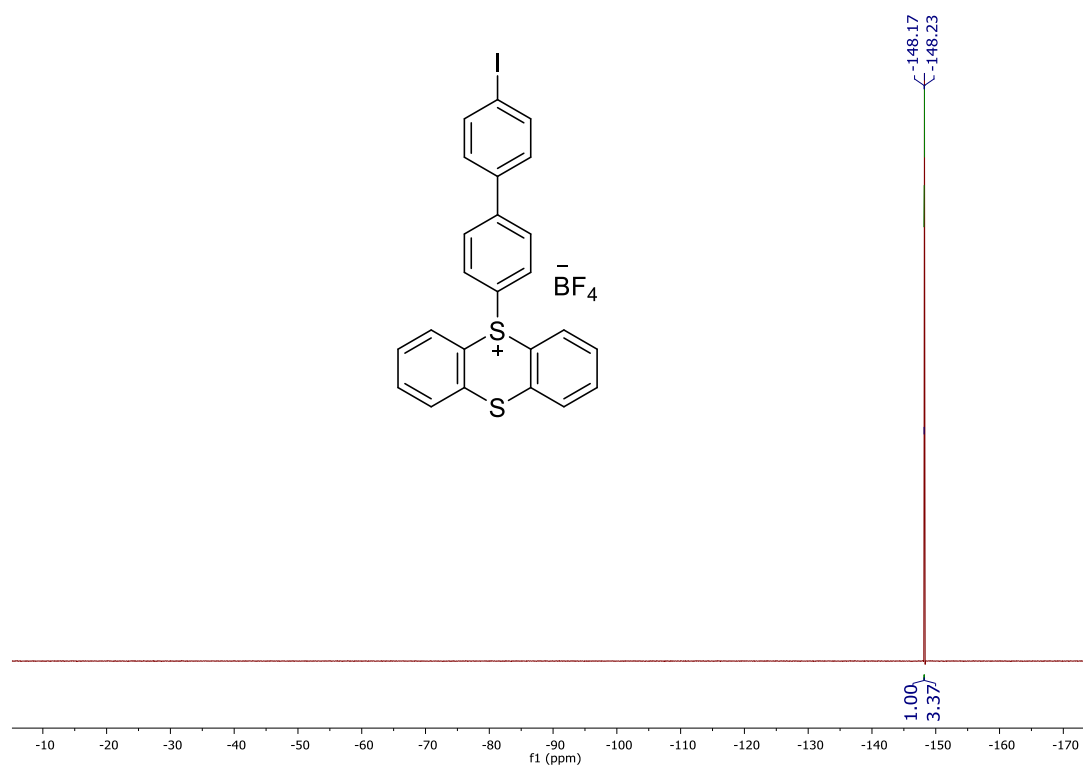
¹³C NMR spectrum of **2e**



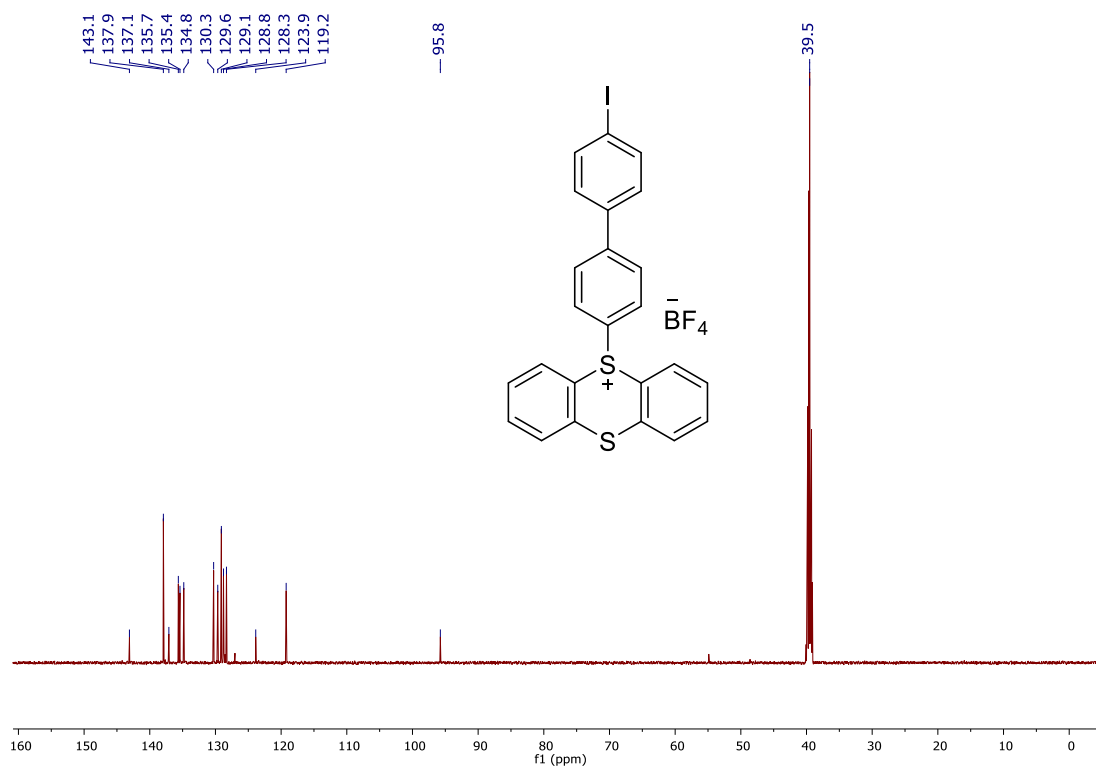
^1H NMR spectrum of **2f**



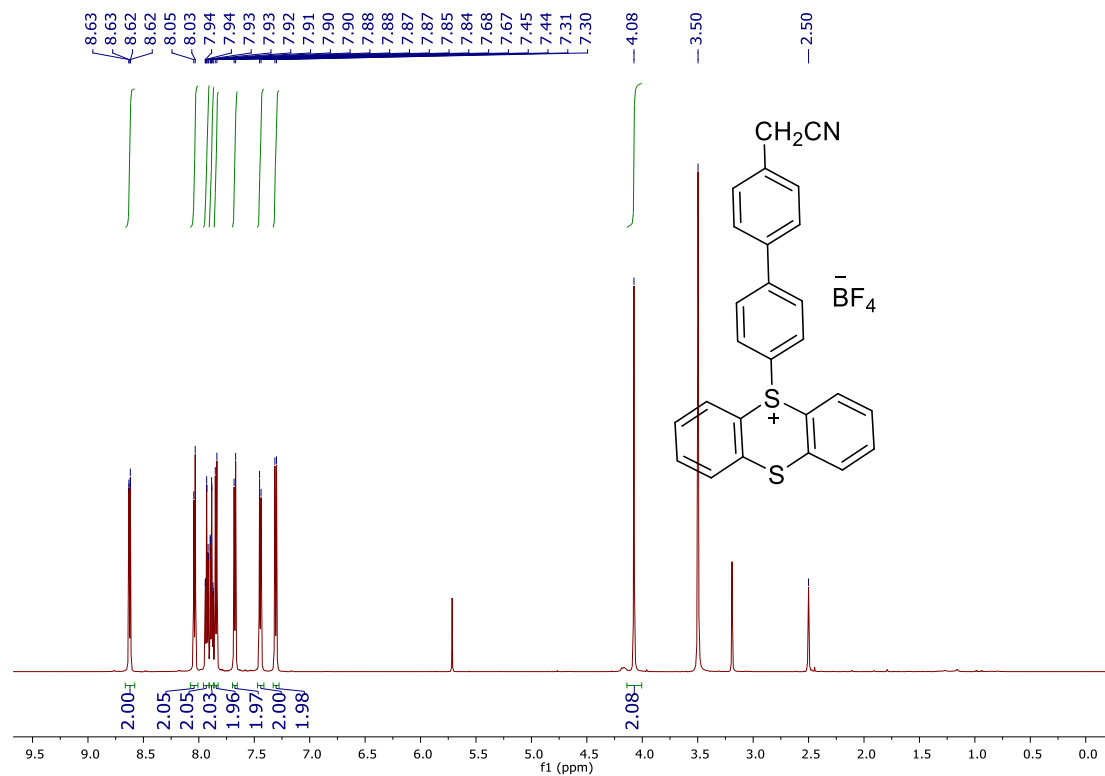
^{19}F NMR spectrum of **2f**



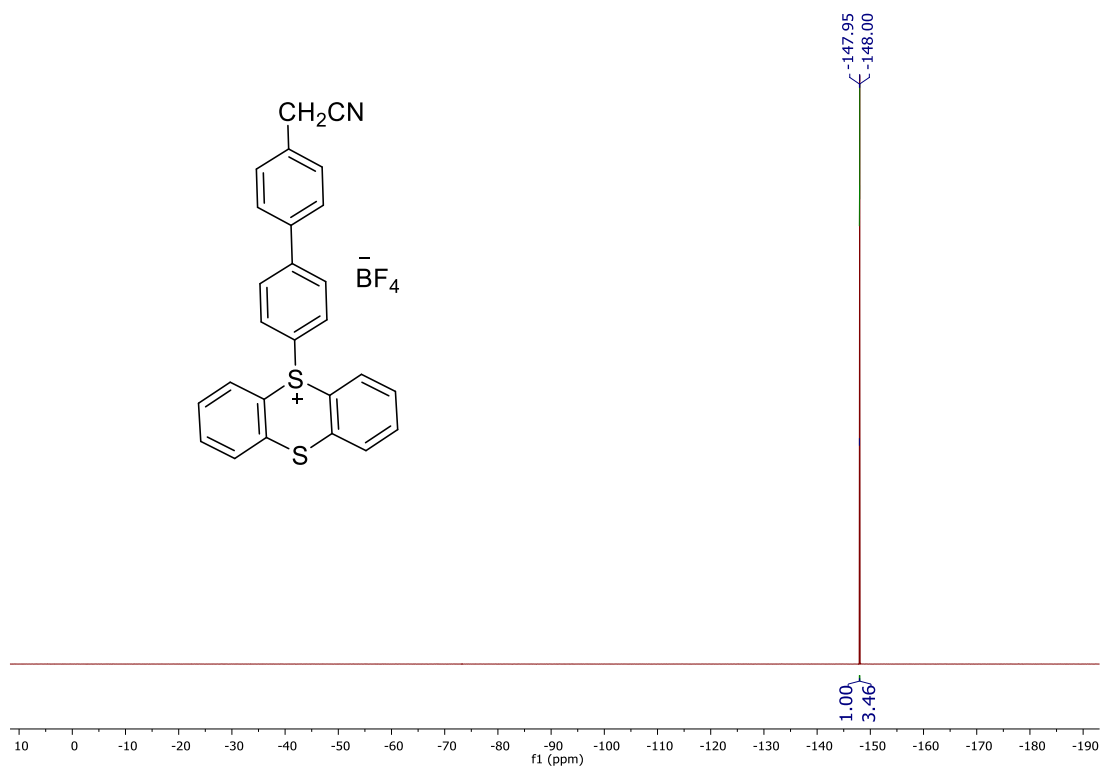
^{13}C NMR spectrum of 2f



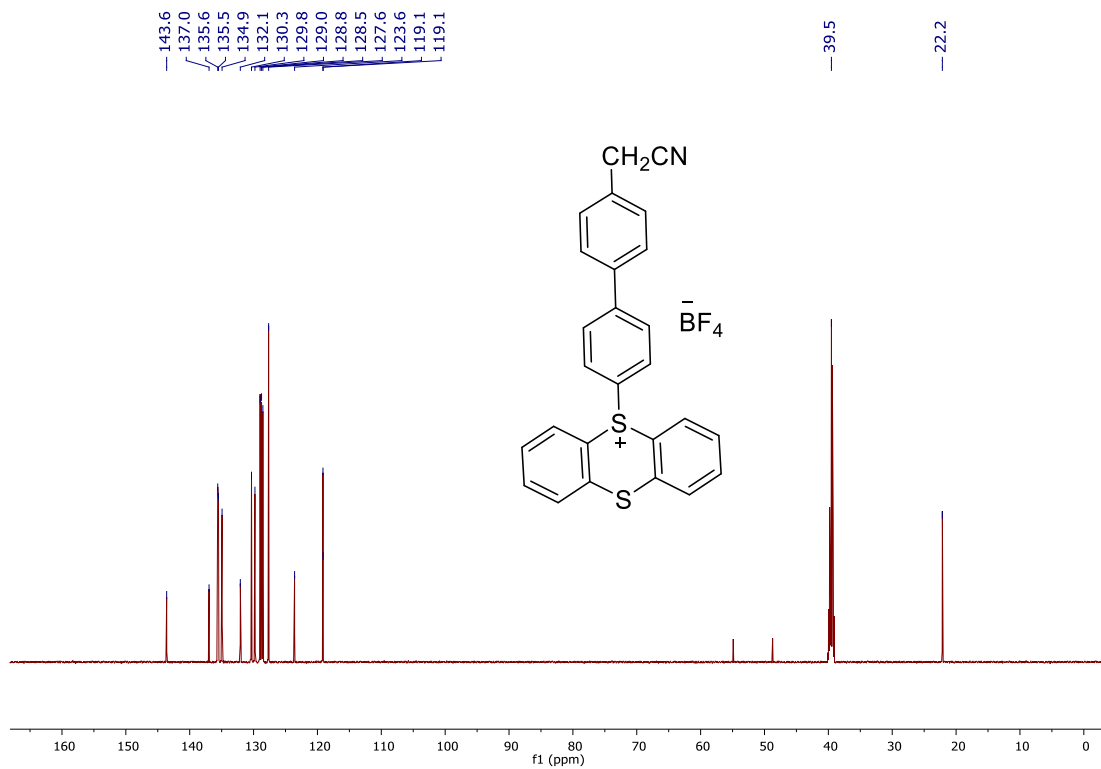
^1H NMR spectrum of 2g



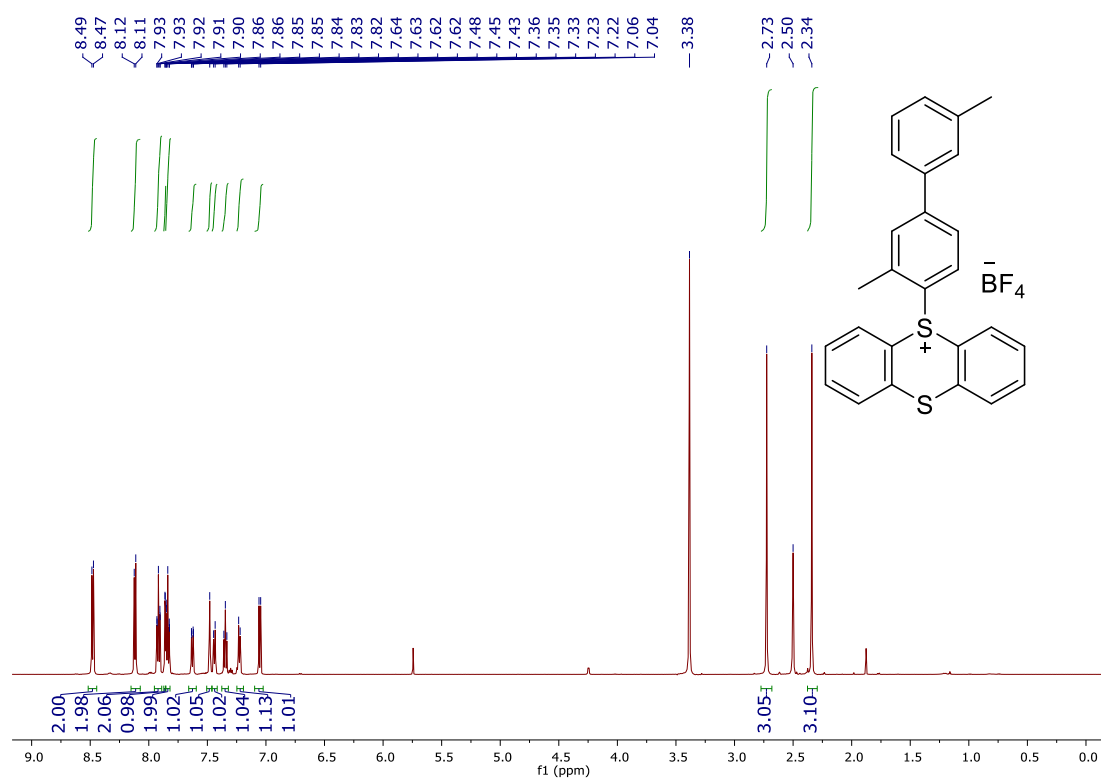
¹⁹F NMR spectrum of **2g**



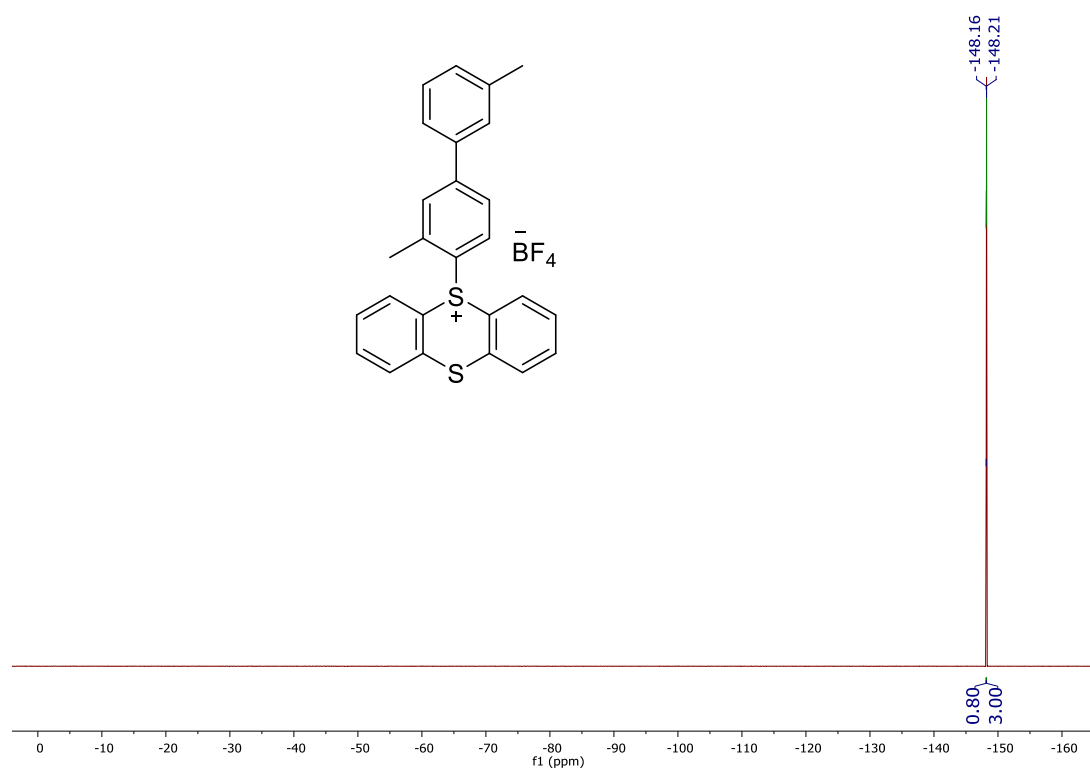
¹³C NMR spectrum of **2g**



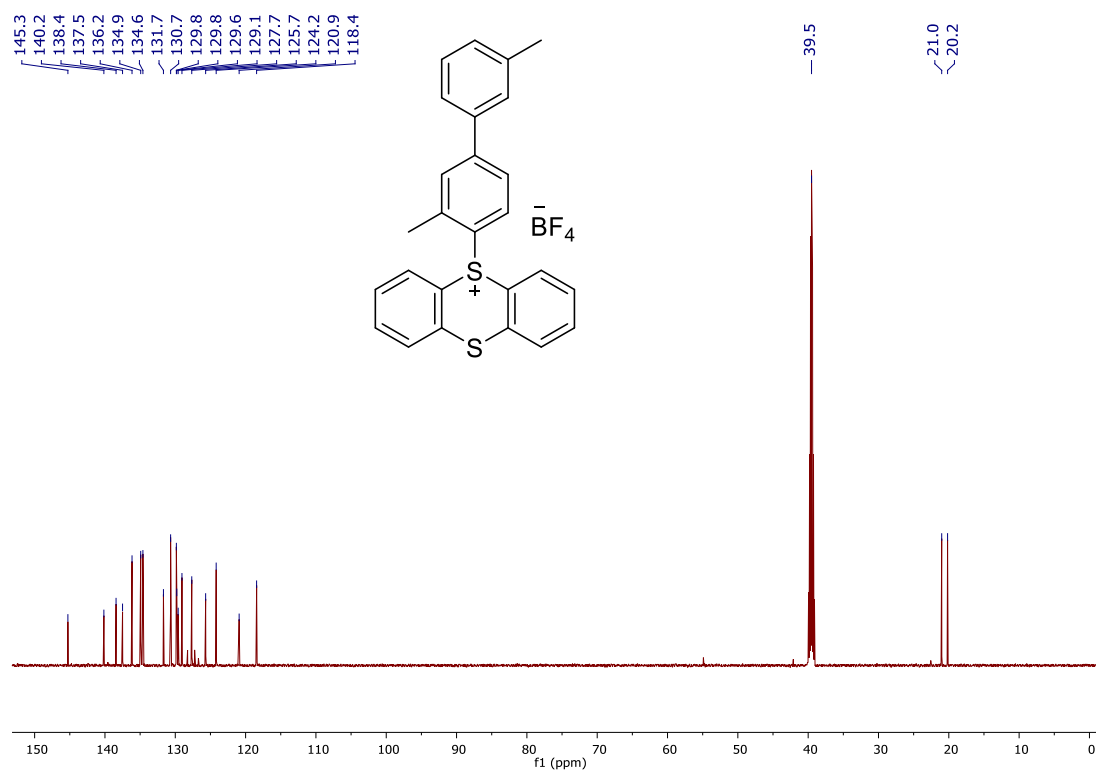
¹H NMR spectrum of 2h



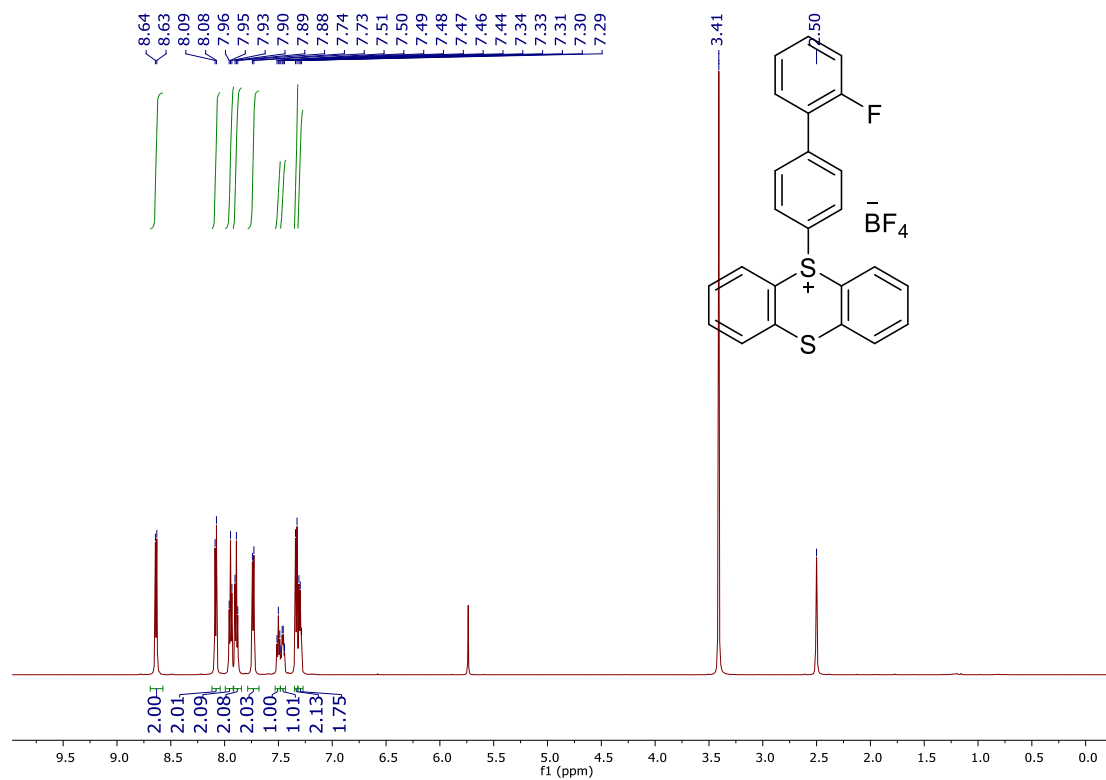
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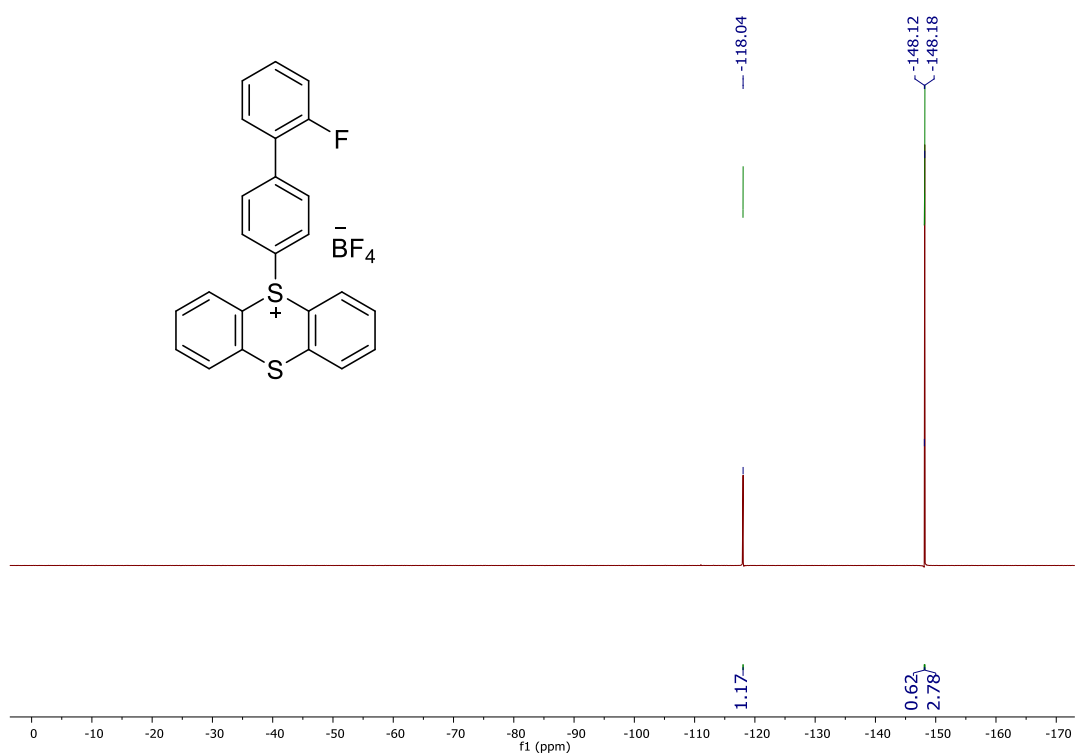
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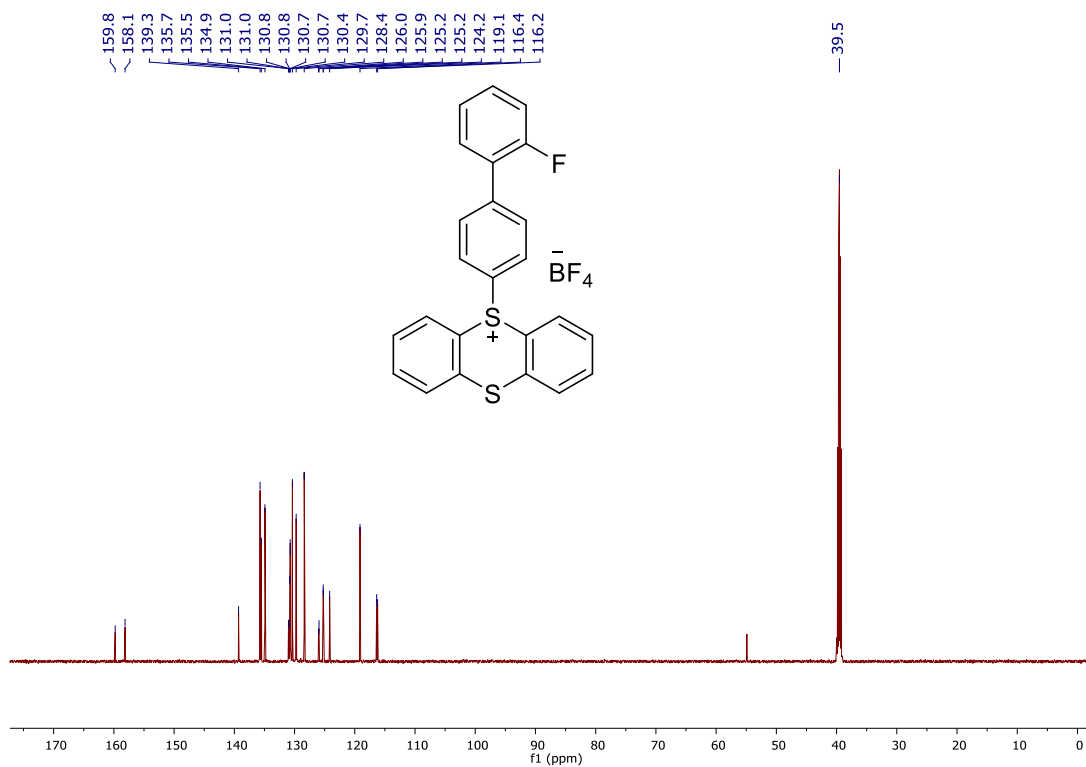
¹H NMR spectrum of 2i



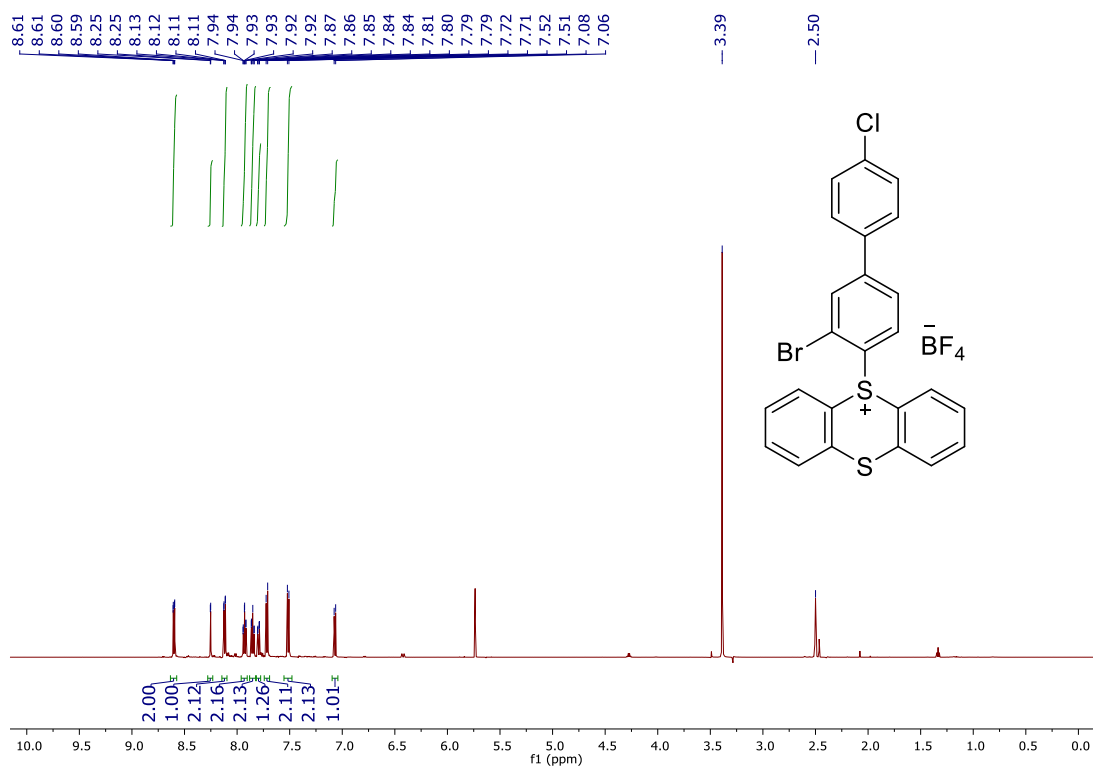
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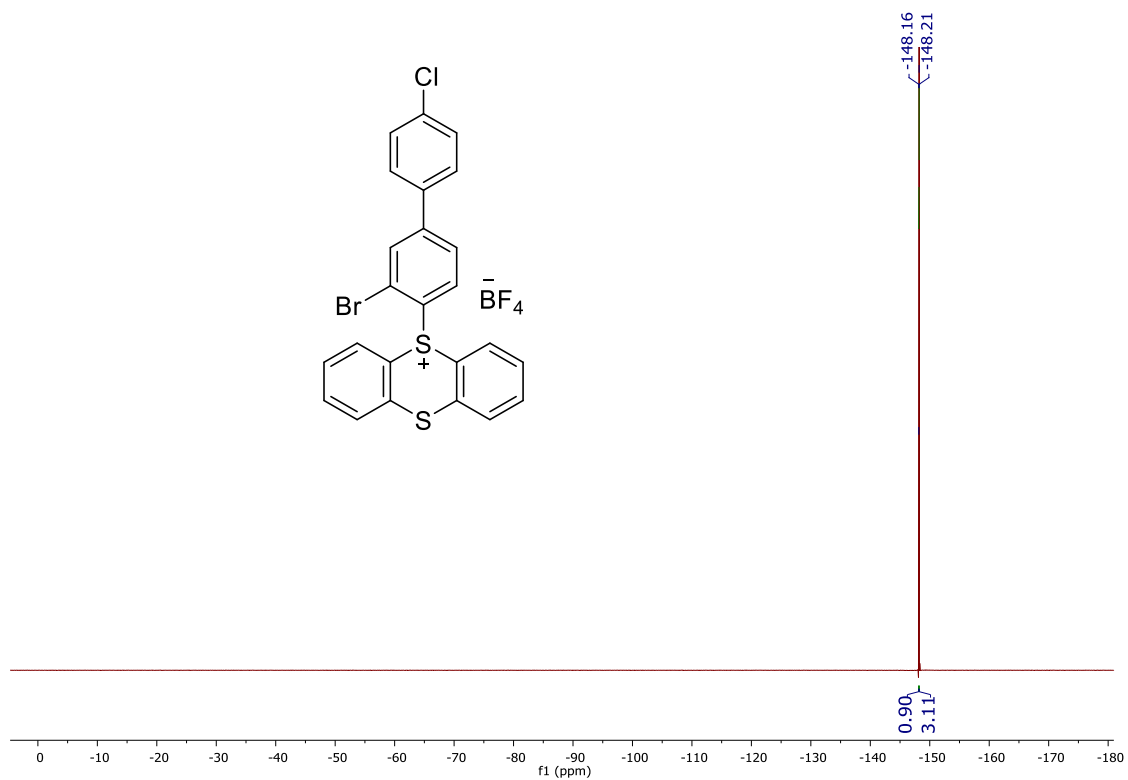
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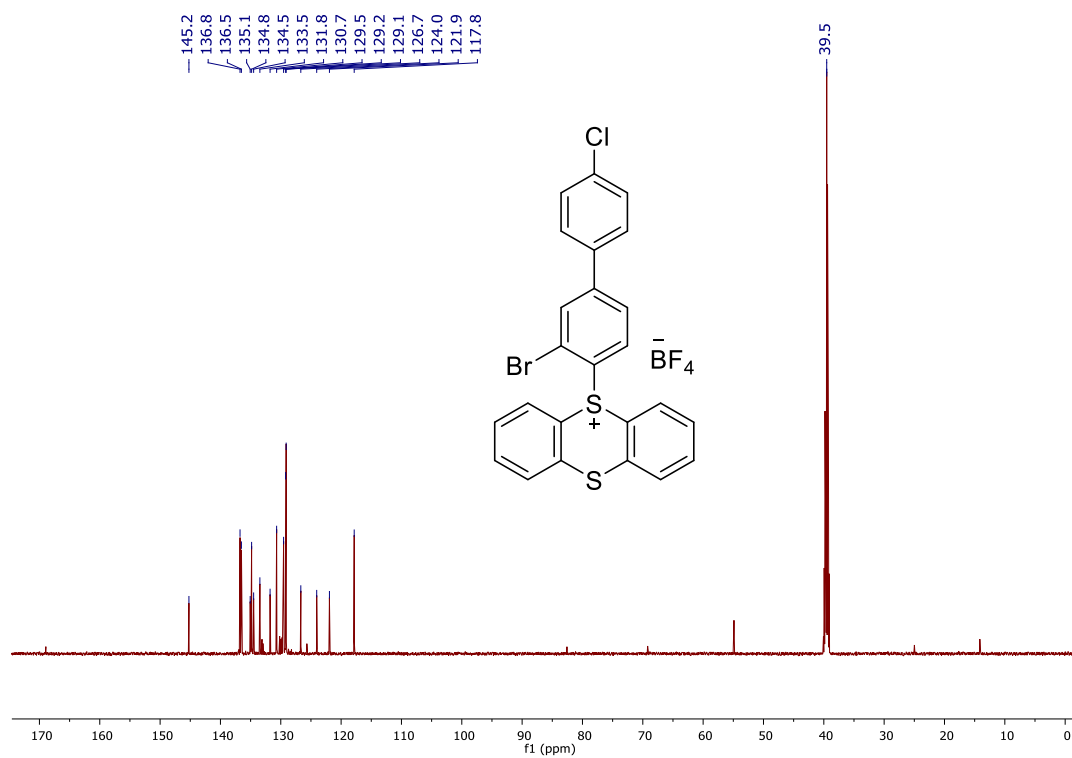
^1H NMR spectrum of 2j



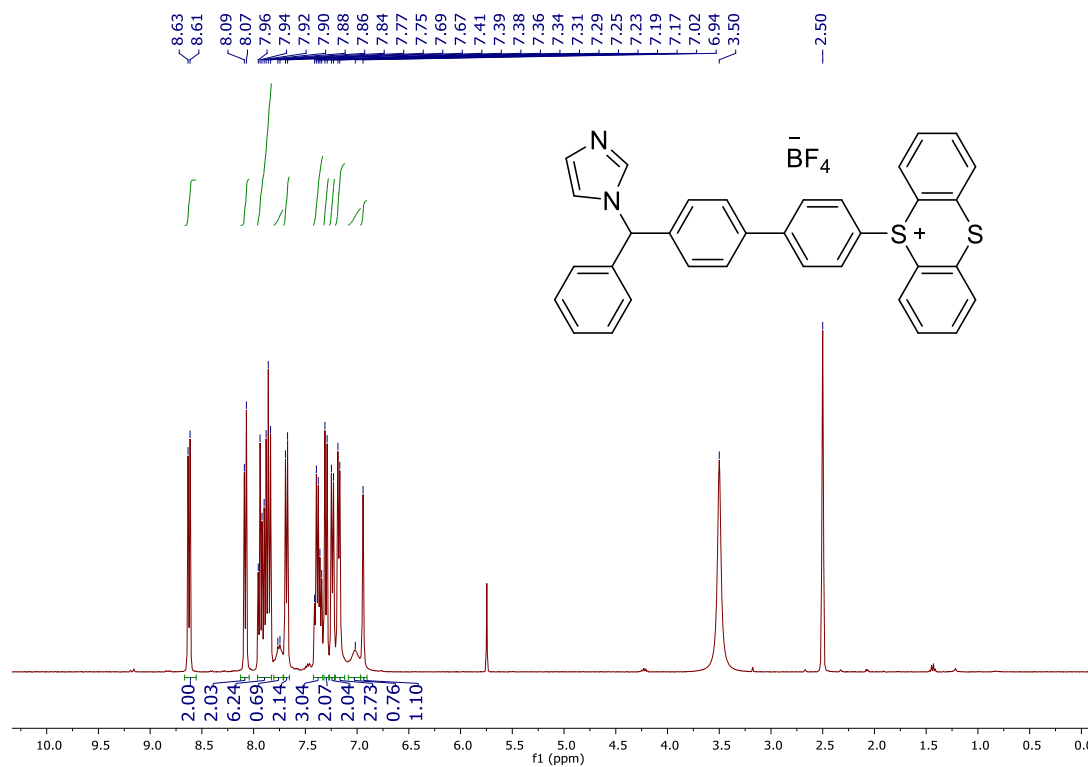
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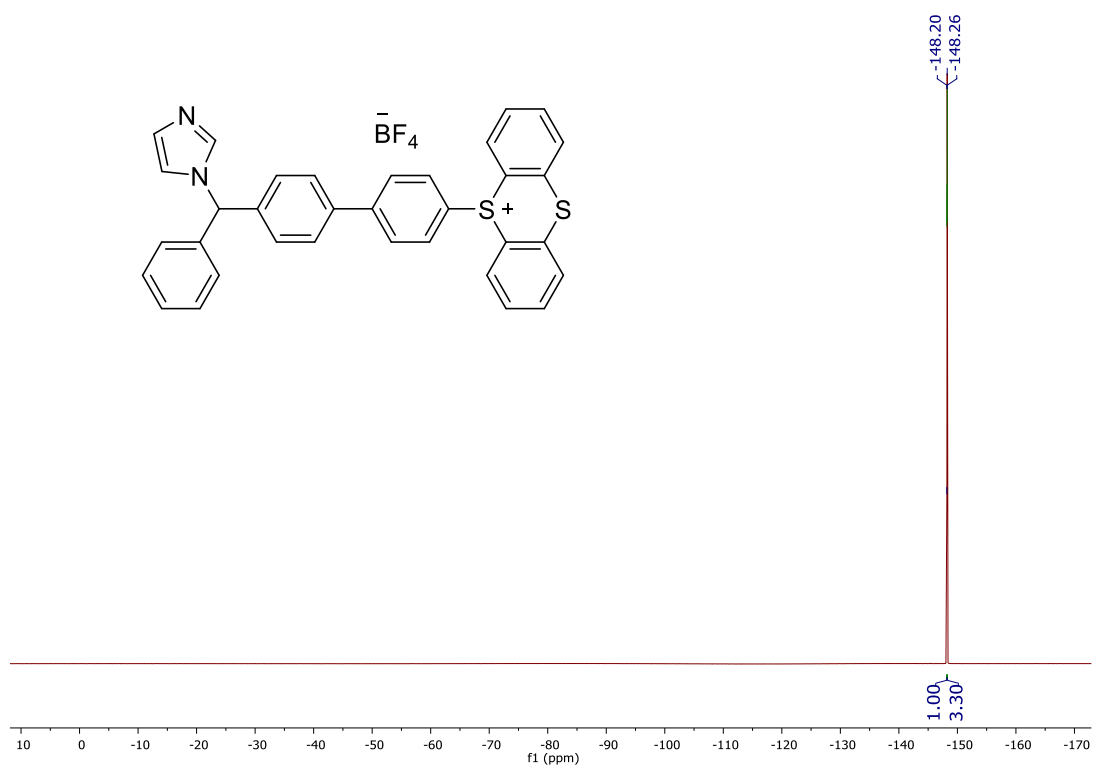
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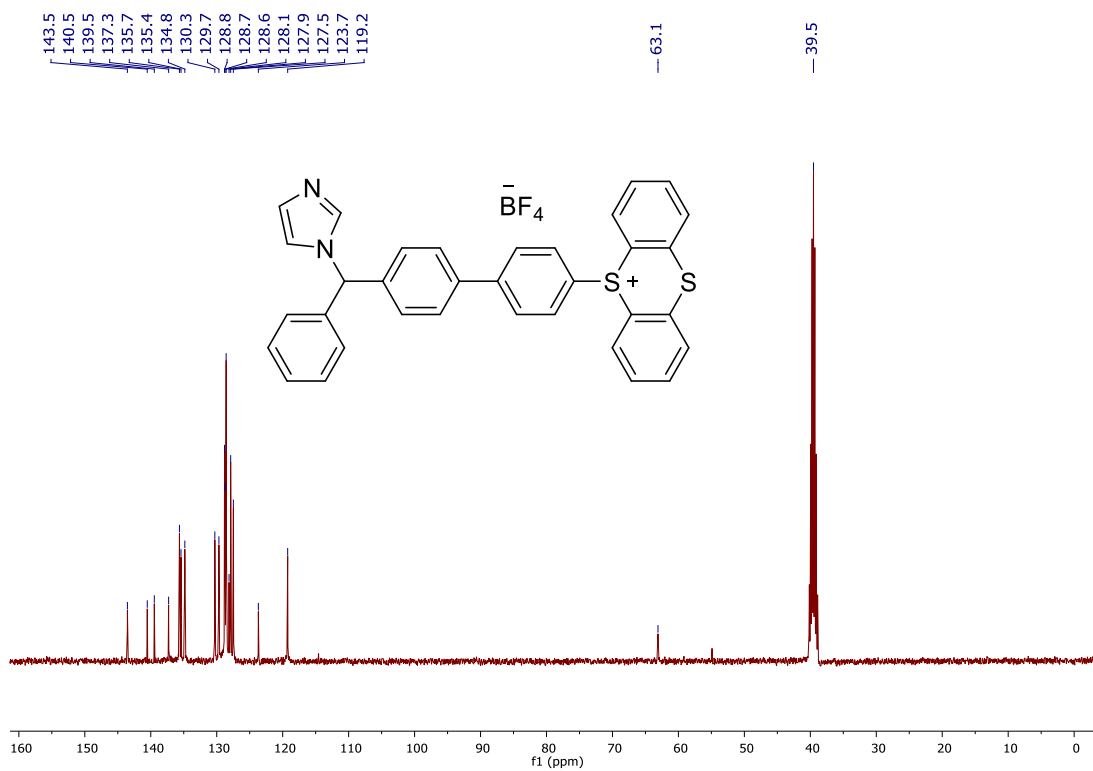
¹H NMR spectrum of **2k**



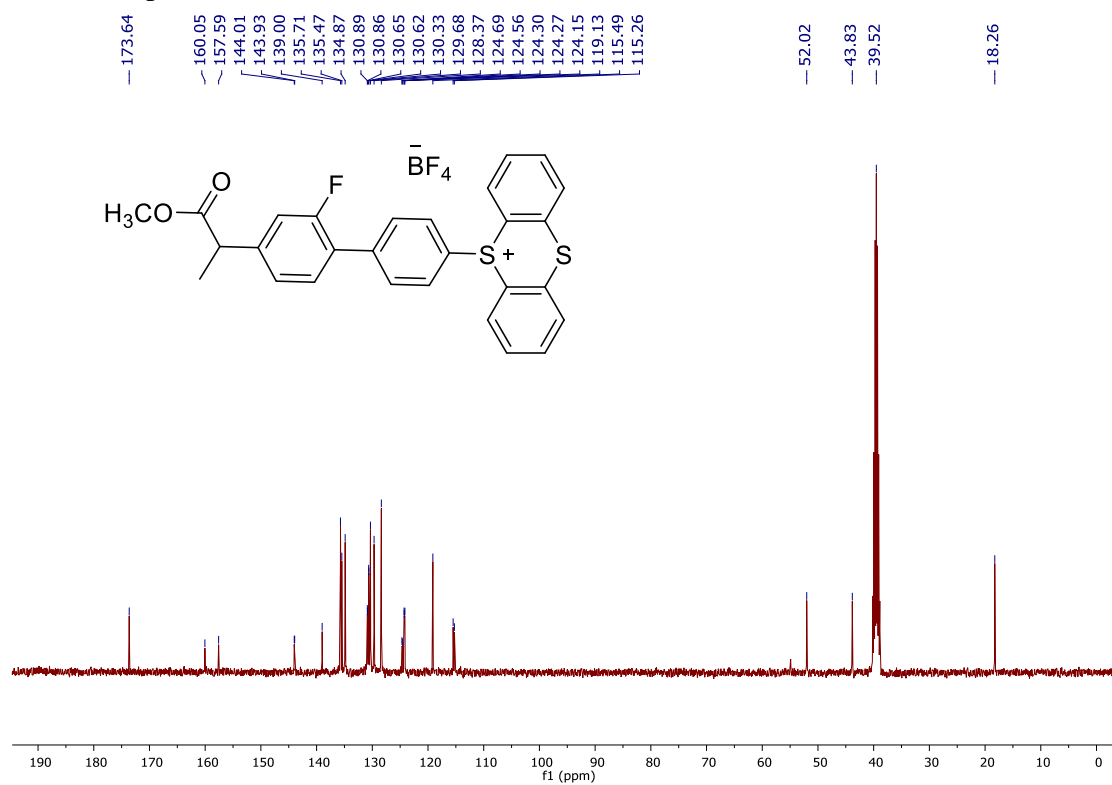
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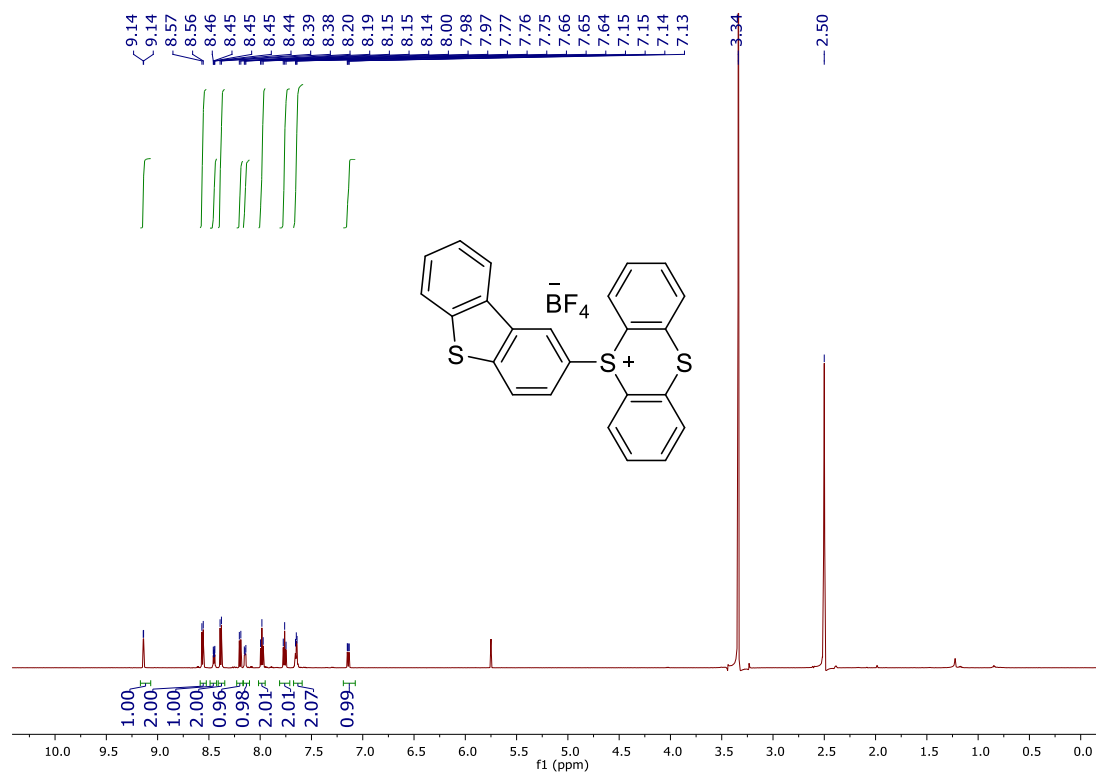
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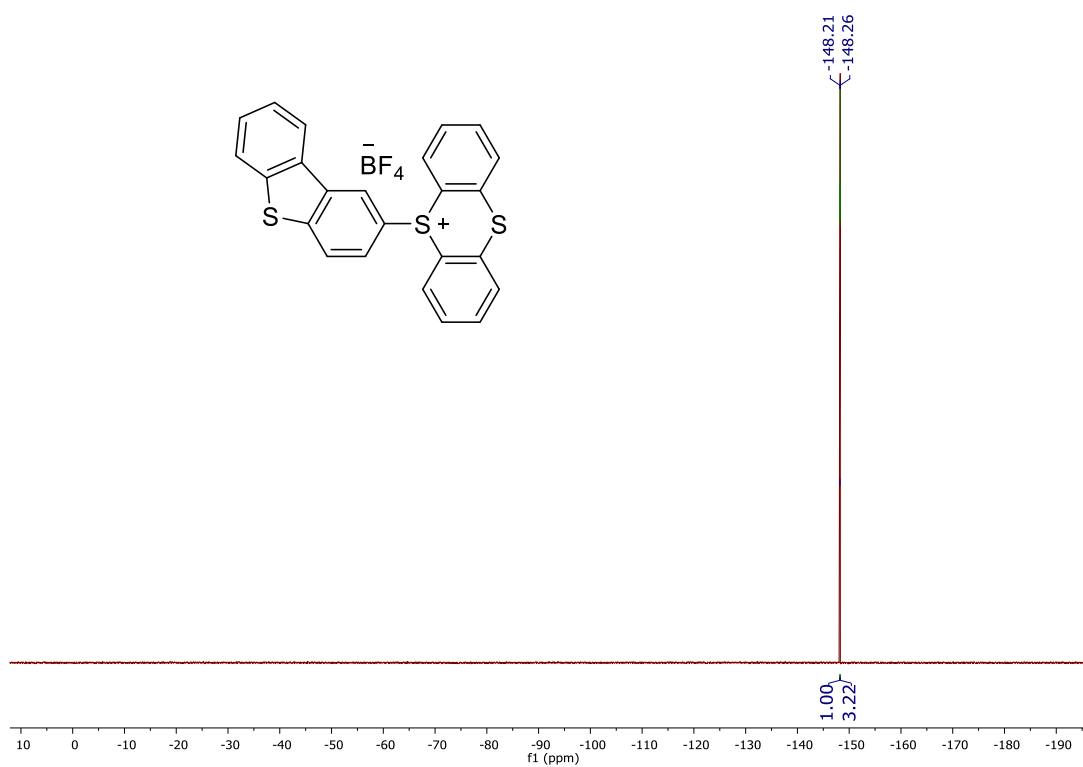
^{13}C NMR spectrum of **2l**



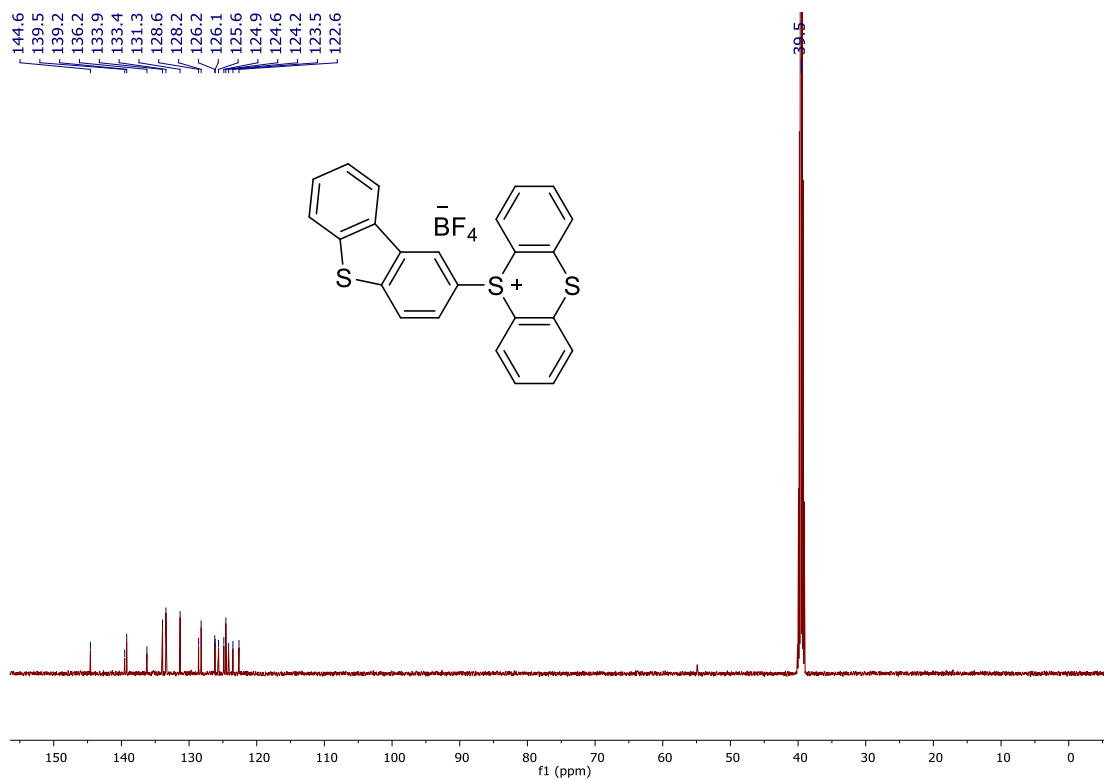
^1H NMR spectrum of **2n**



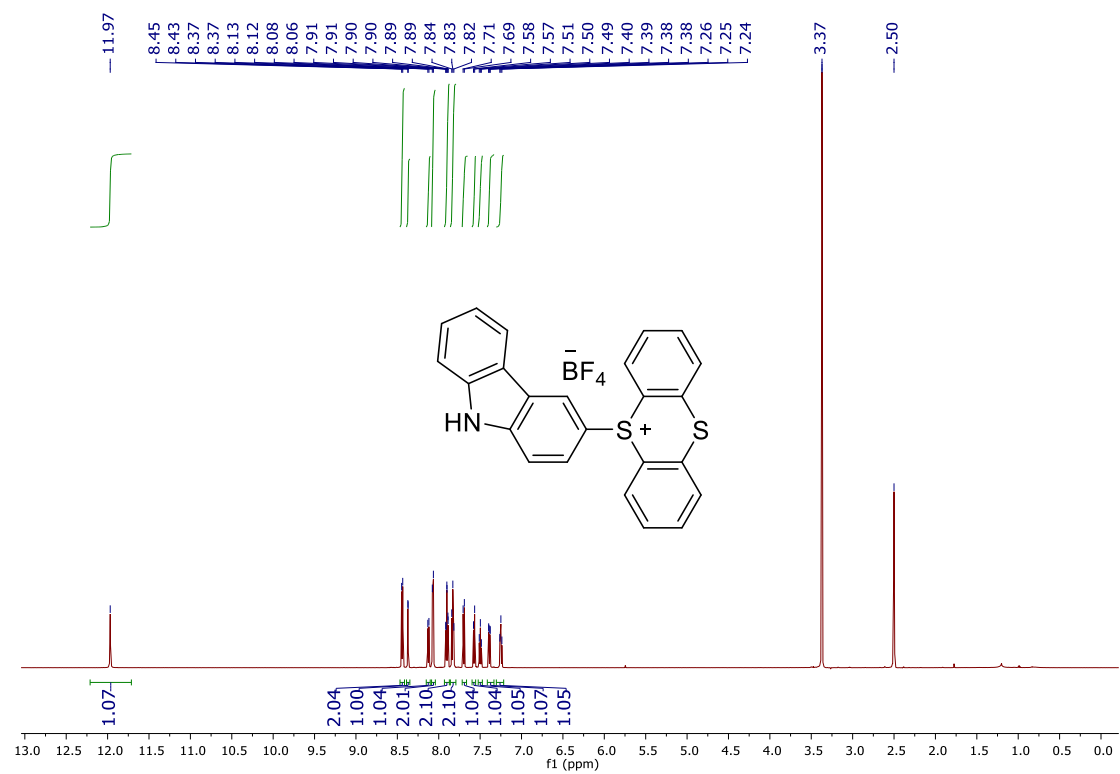
¹⁹F NMR spectrum of **2n**



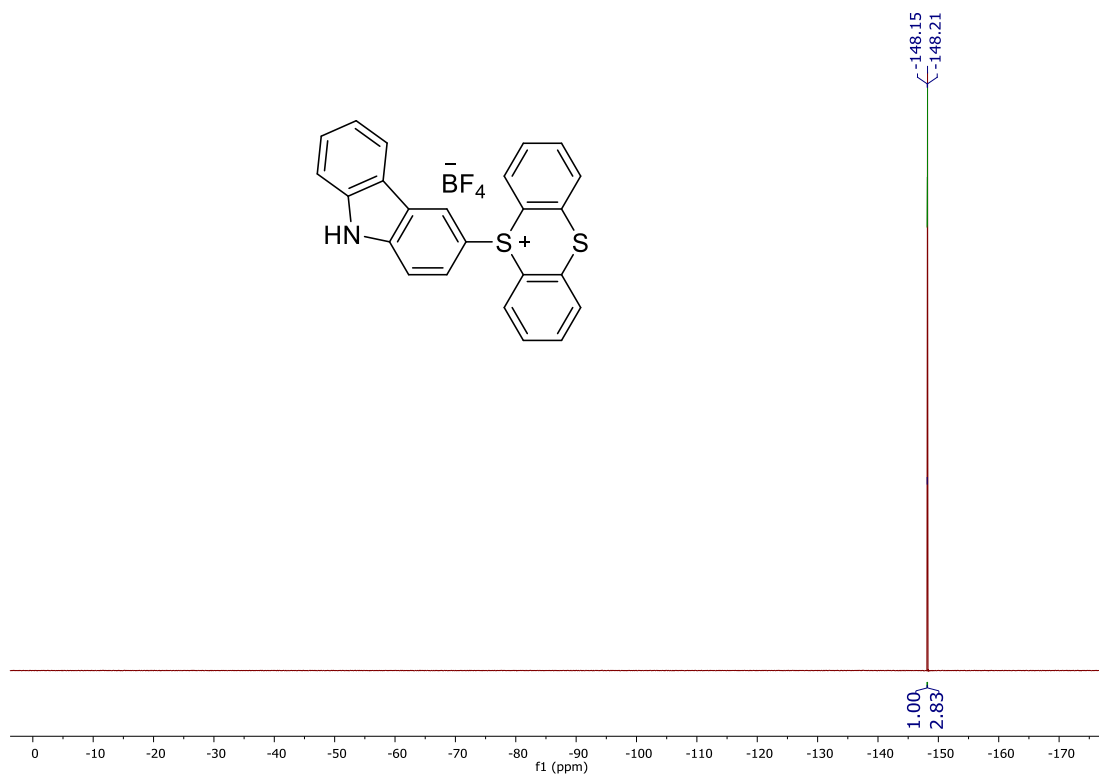
¹³C NMR spectrum of **2n**



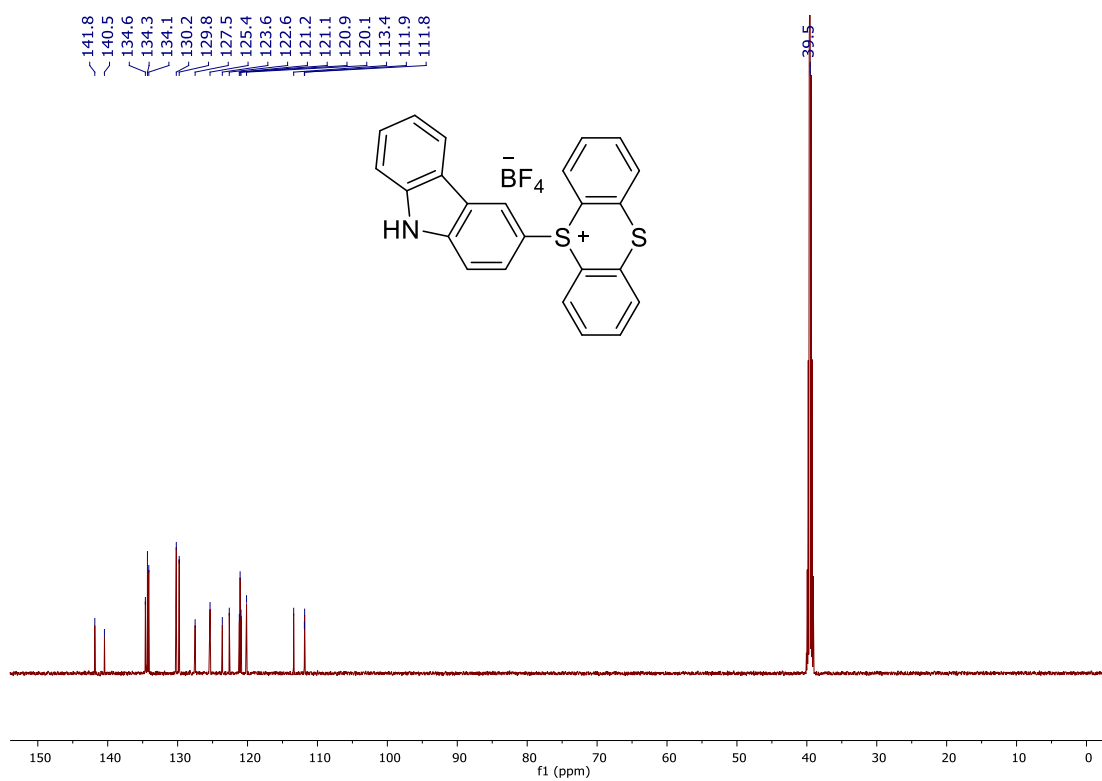
¹H NMR spectrum of **2o**



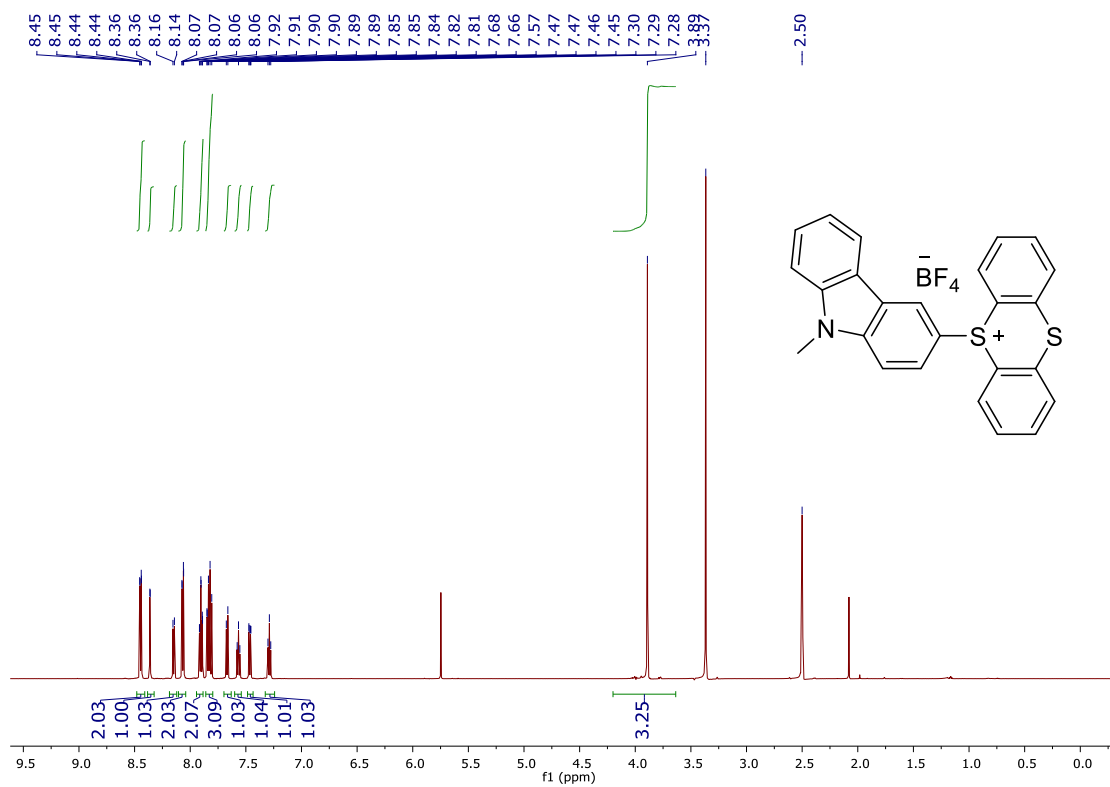
¹⁹F NMR spectrum of **2o**



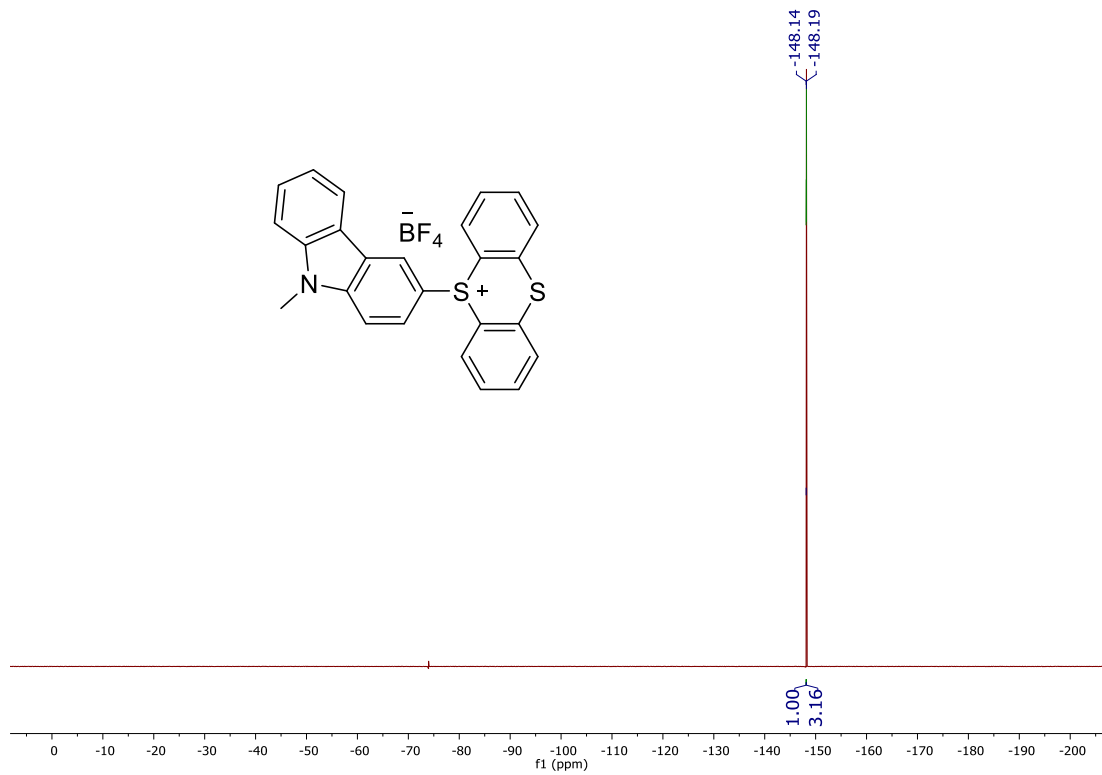
¹³C NMR spectrum of 2o



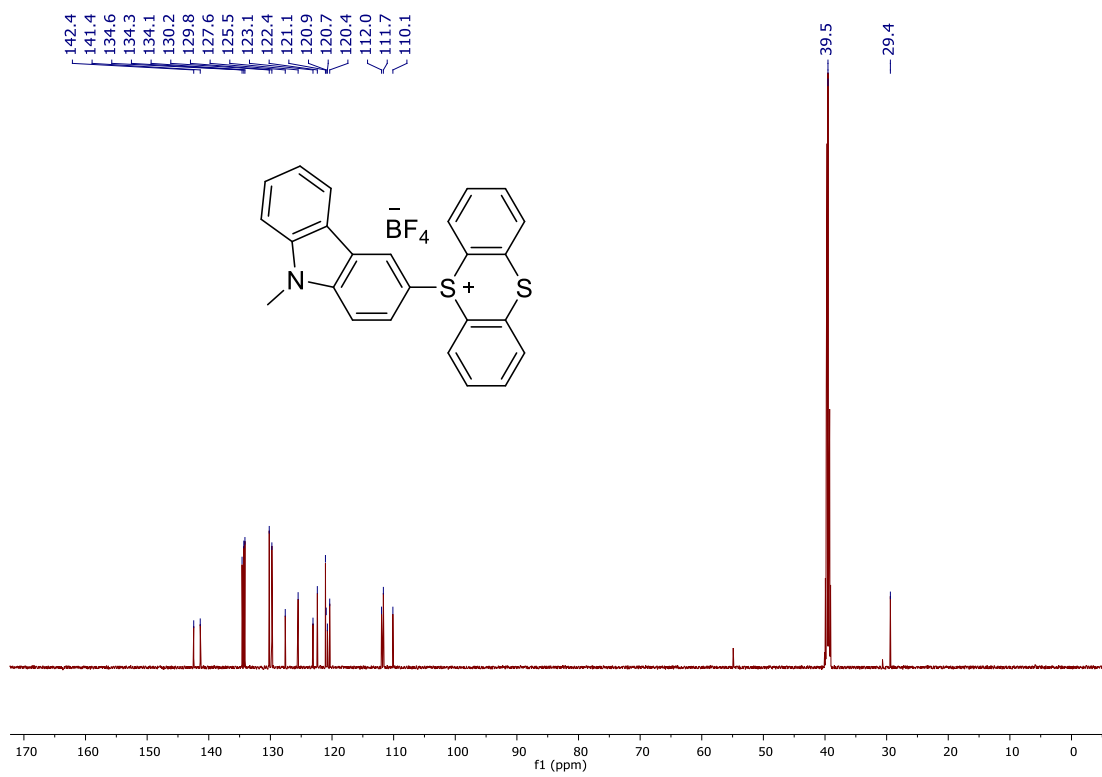
¹H NMR spectrum of 2p



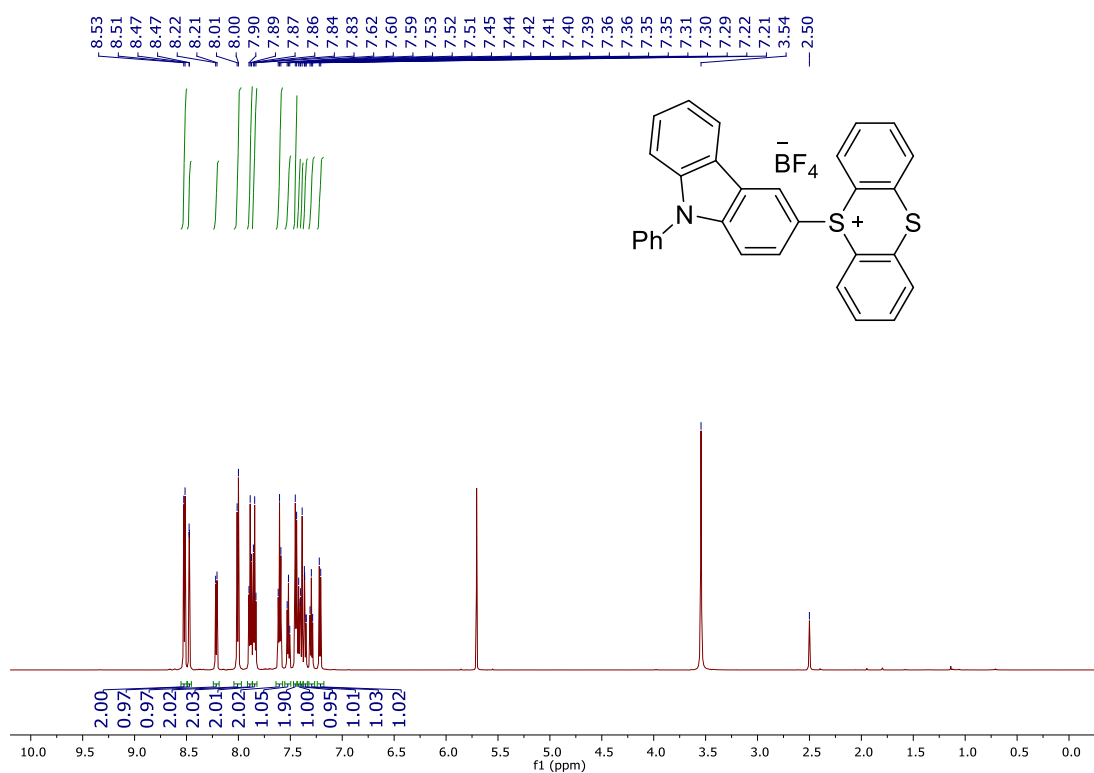
¹⁹F NMR spectrum of **2p**



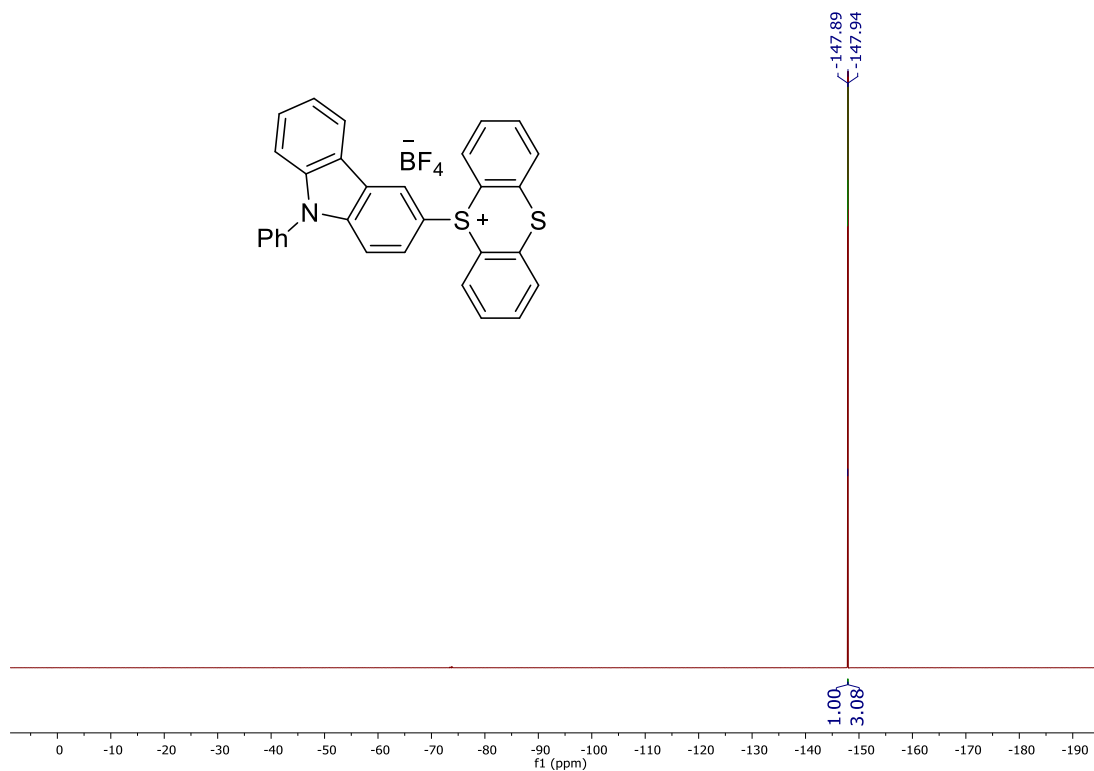
¹³C NMR spectrum of **2p**



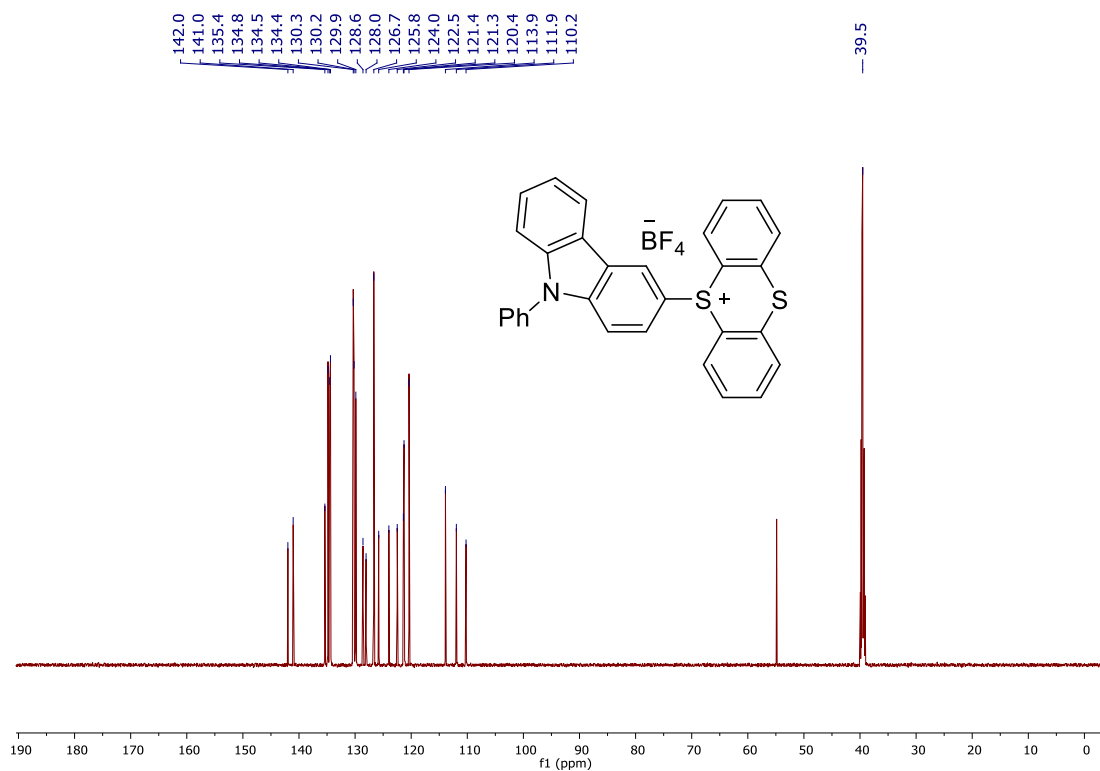
^1H NMR spectrum of **2q**



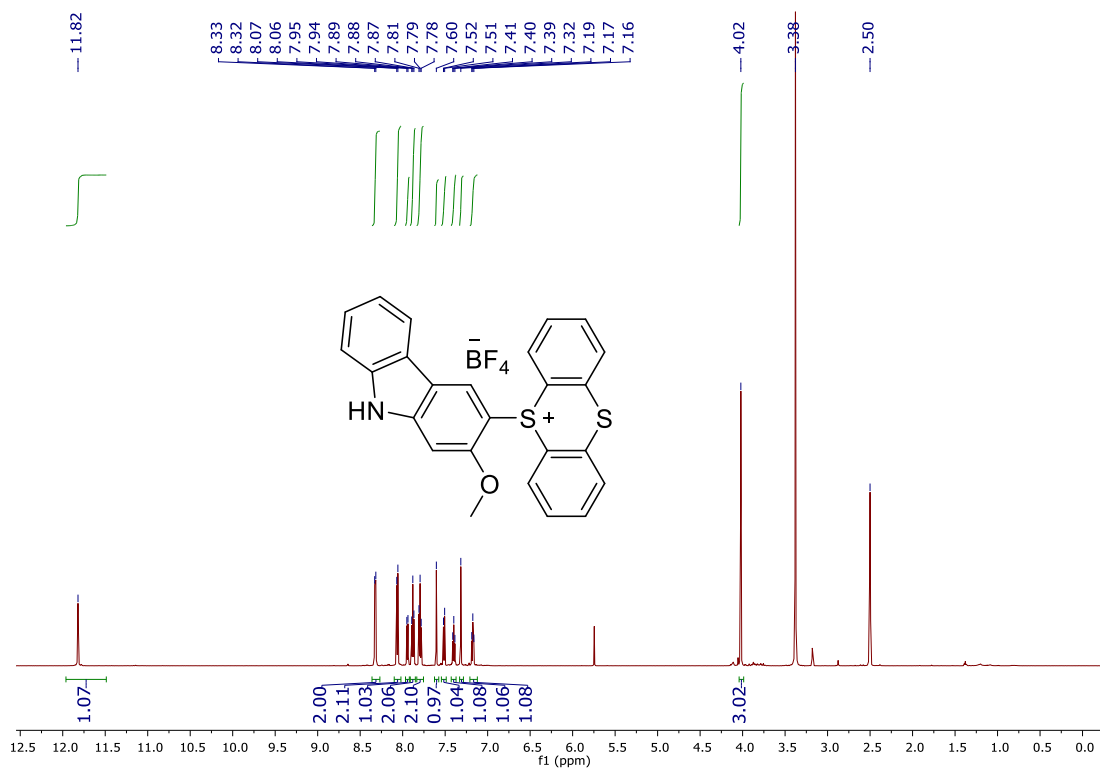
^{19}F NMR spectrum of **2q**



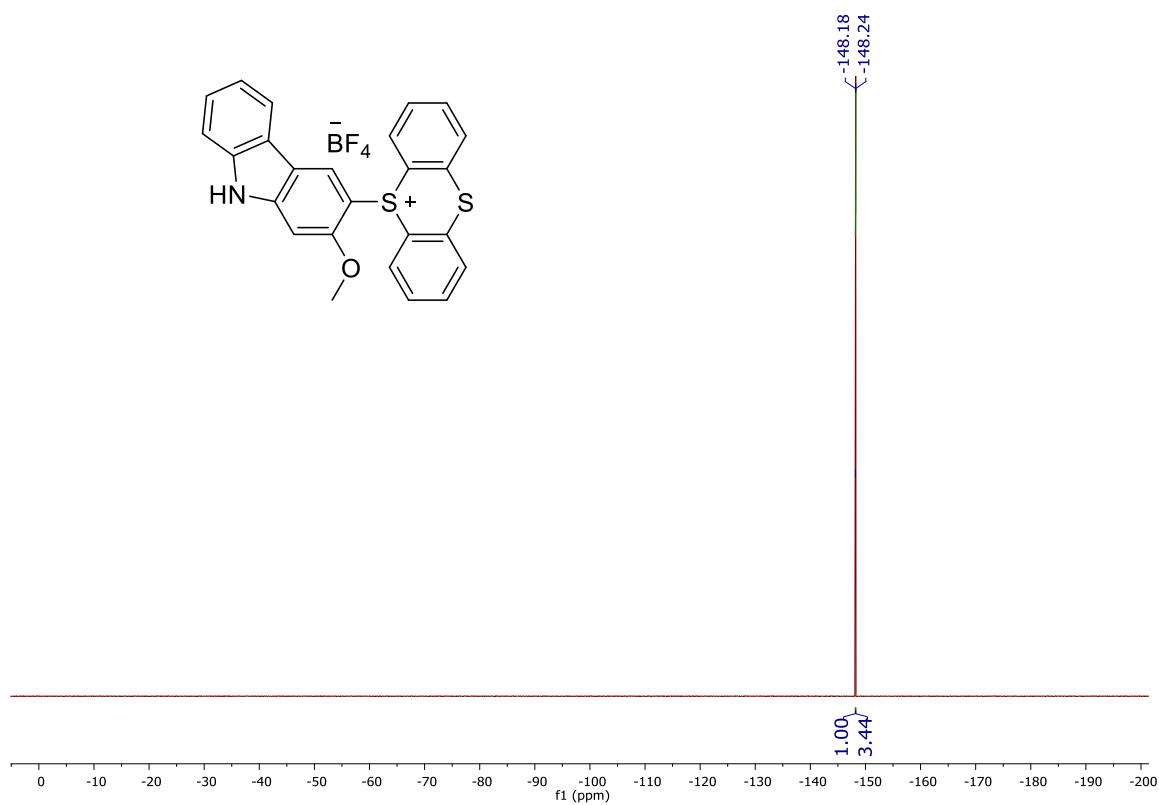
¹³C NMR spectrum of 2q



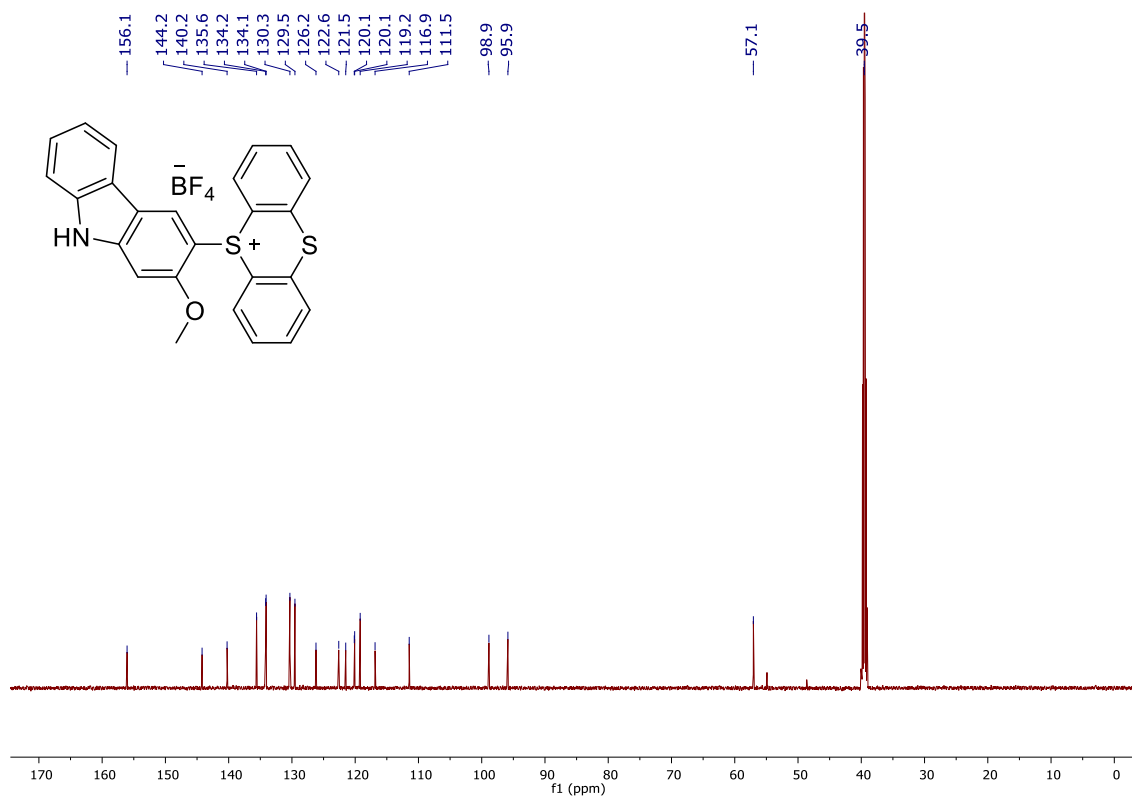
¹H NMR spectrum of 2r



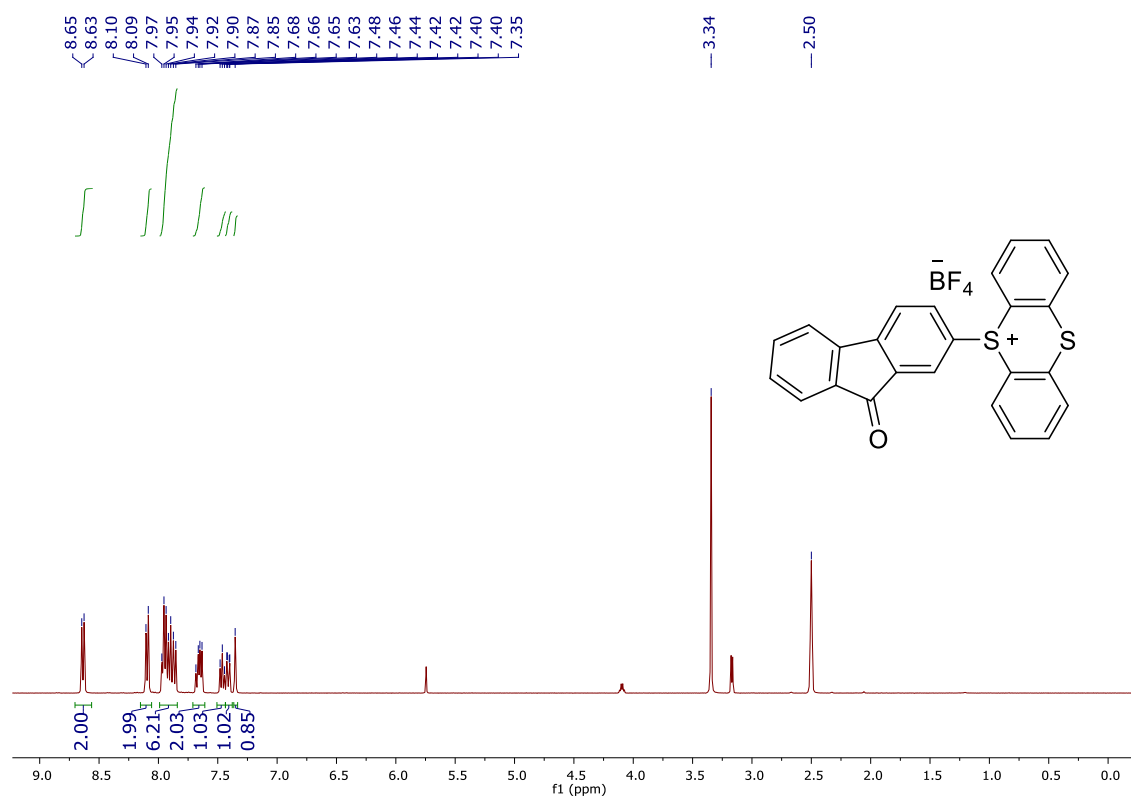
^{19}F NMR spectrum of **2r**



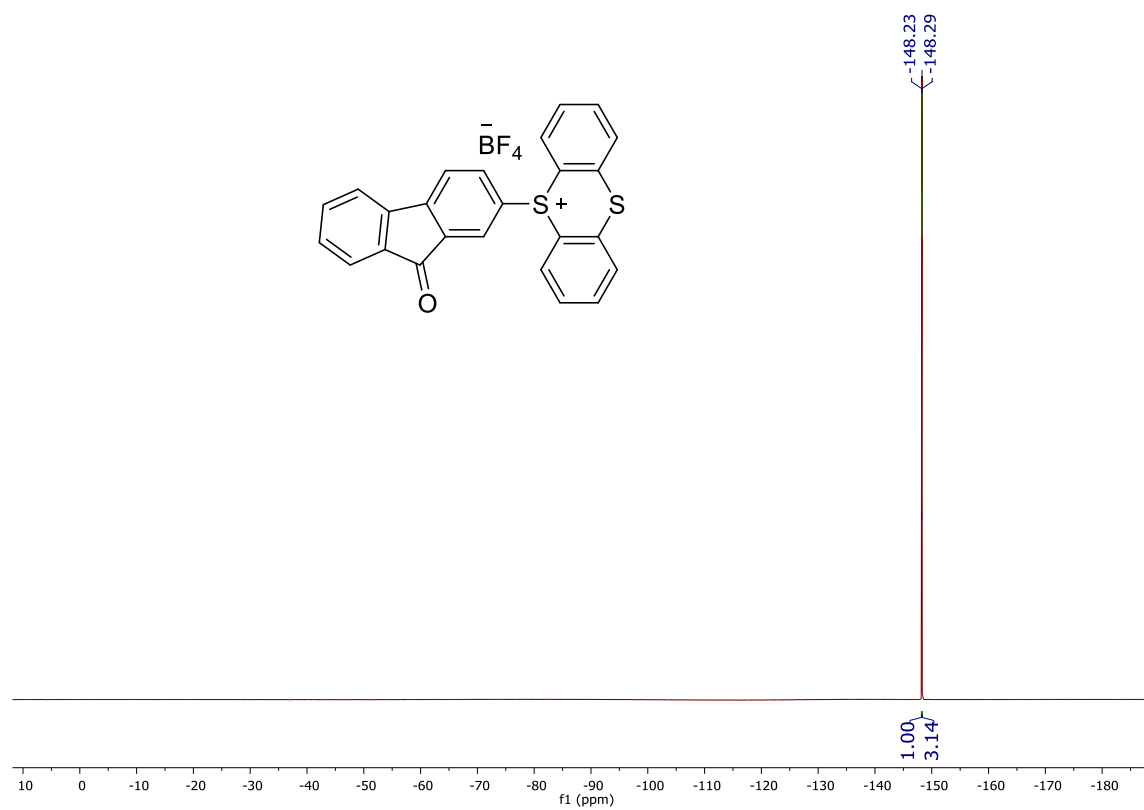
^{13}C NMR spectrum of **2r**



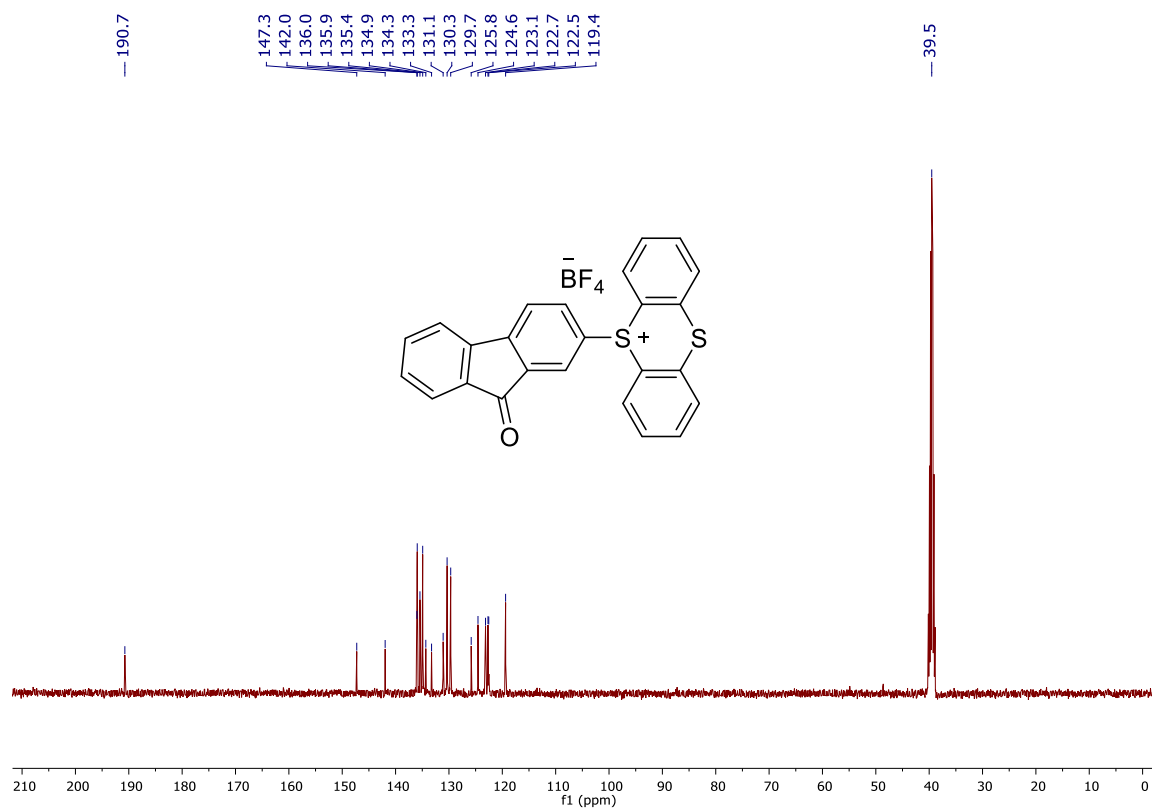
^1H NMR spectrum of **2t**



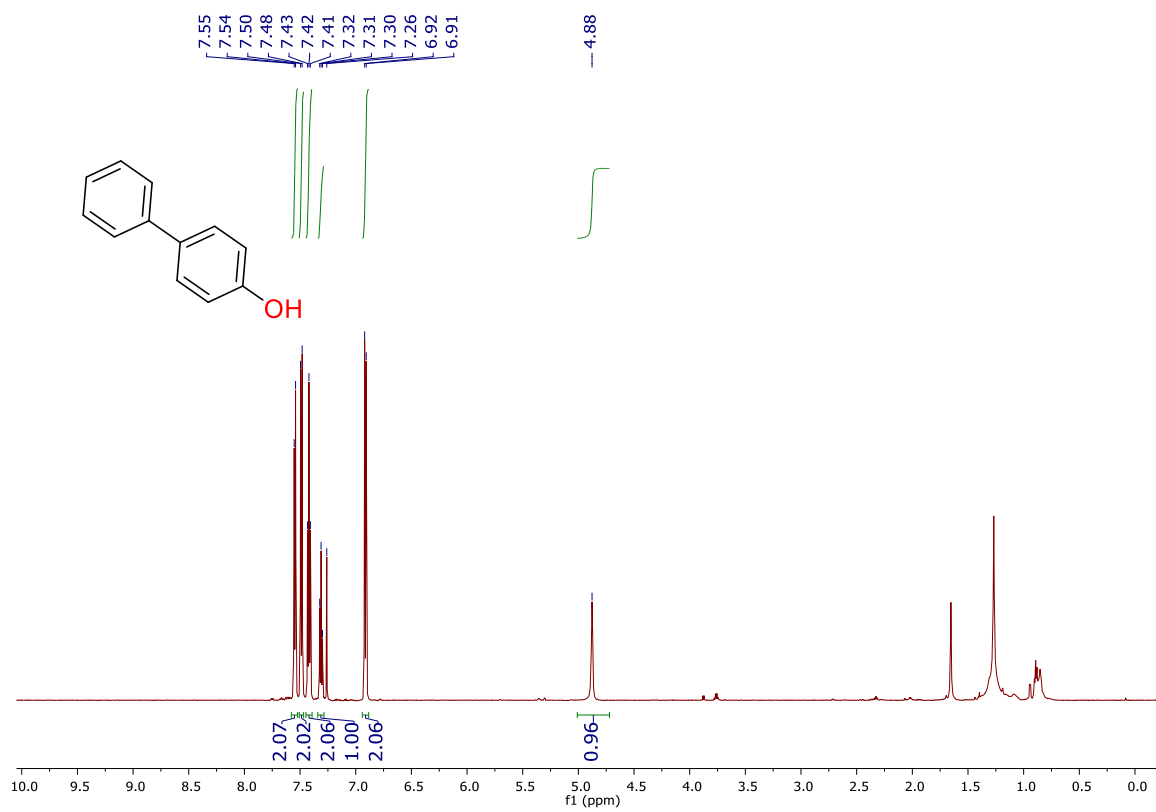
^{19}F NMR spectrum of **2t**



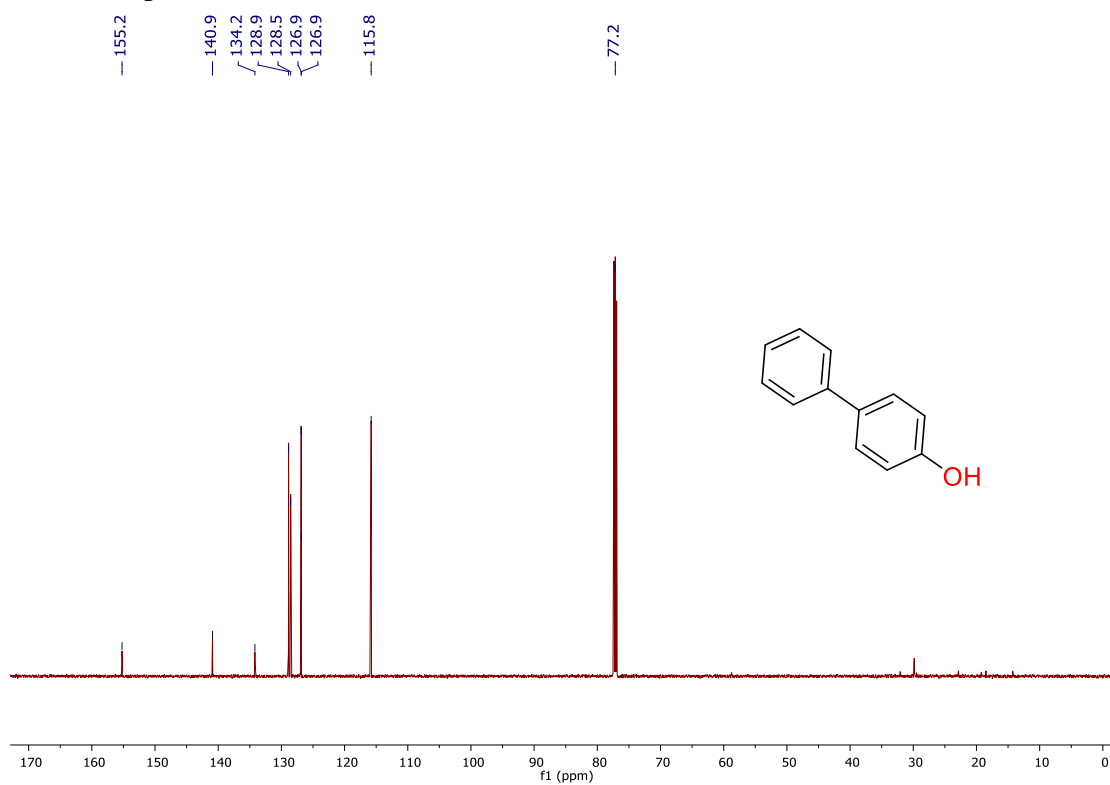
^{13}C NMR spectrum of 2t



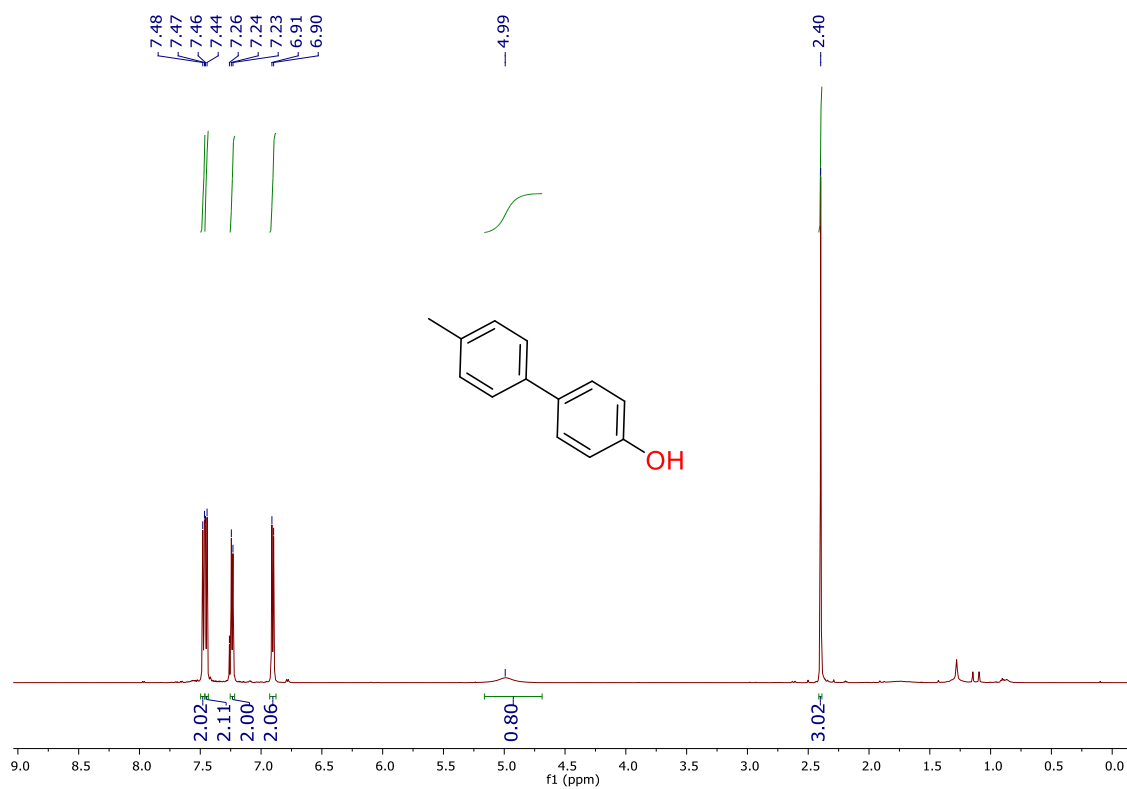
^1H NMR spectrum of 3a



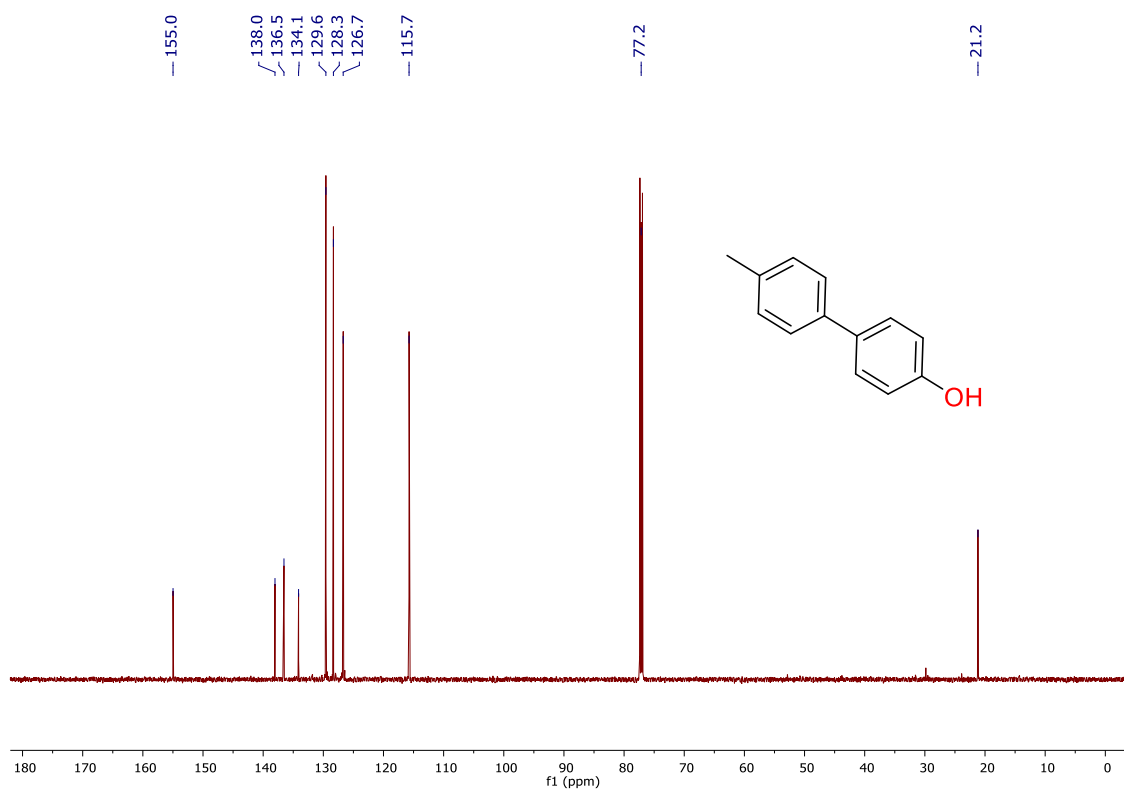
^{13}C NMR spectrum of **3a**



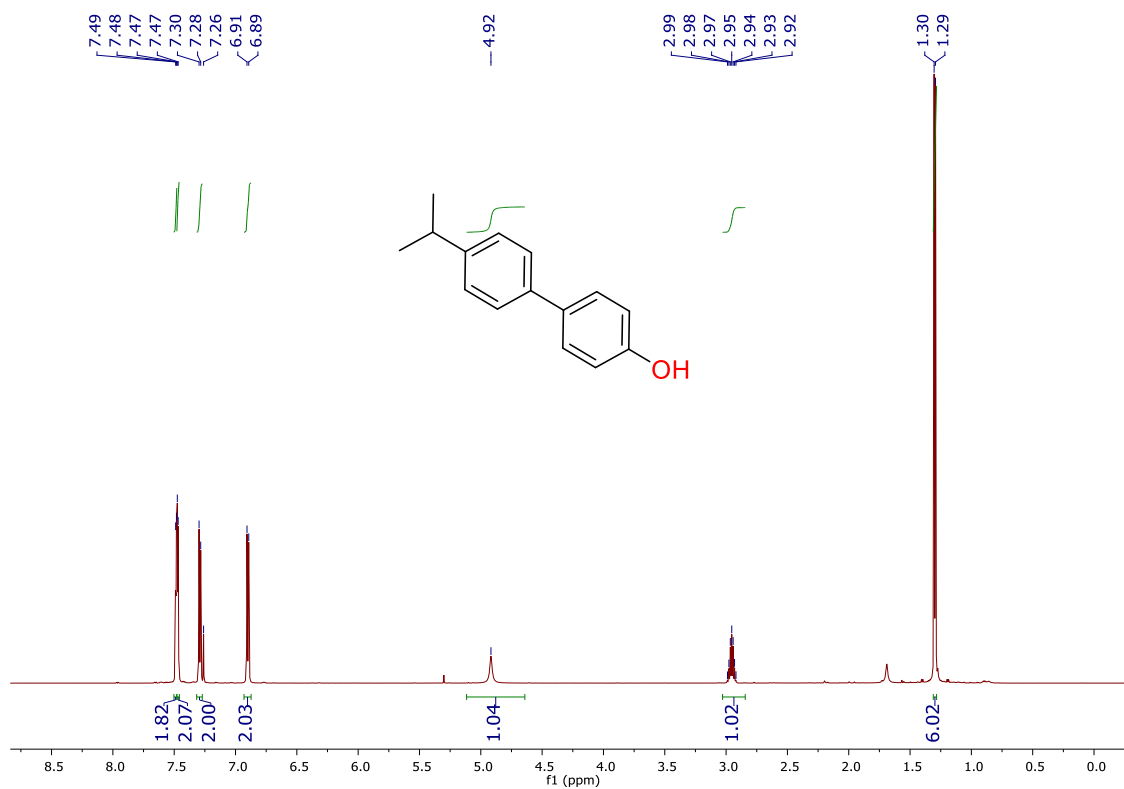
^1H NMR spectrum of **3b**



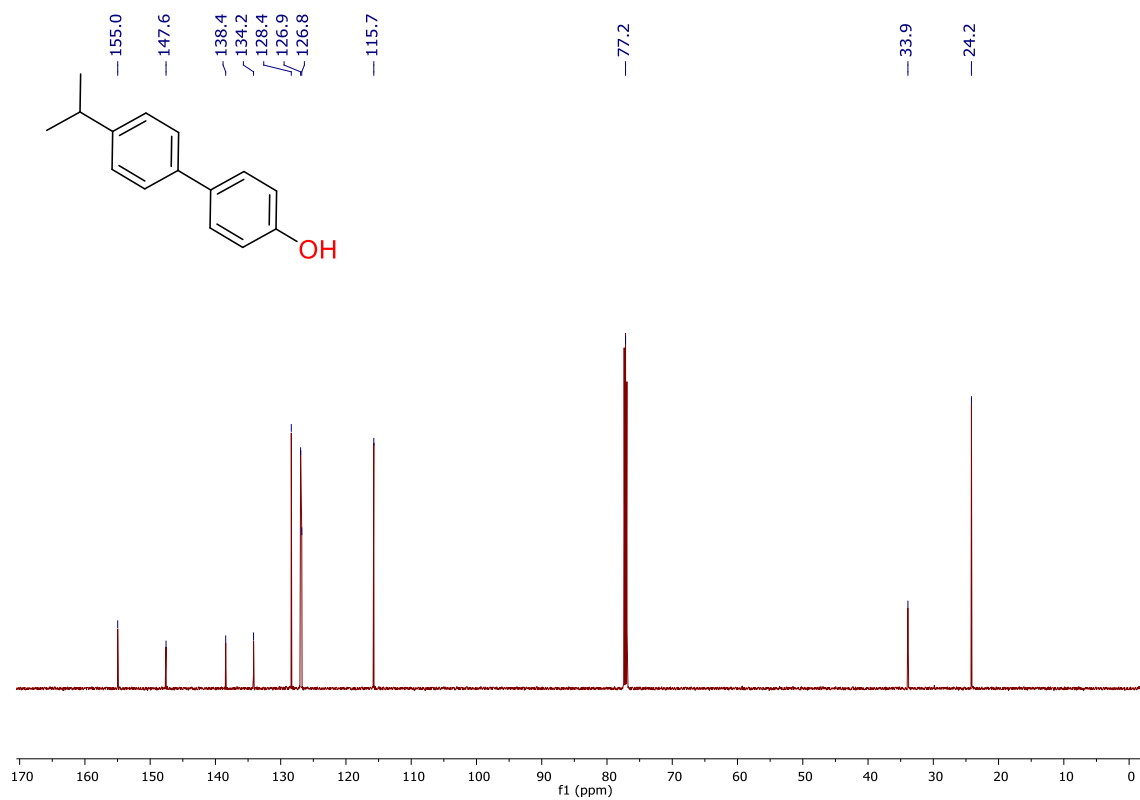
^{13}C NMR spectrum of **3b**



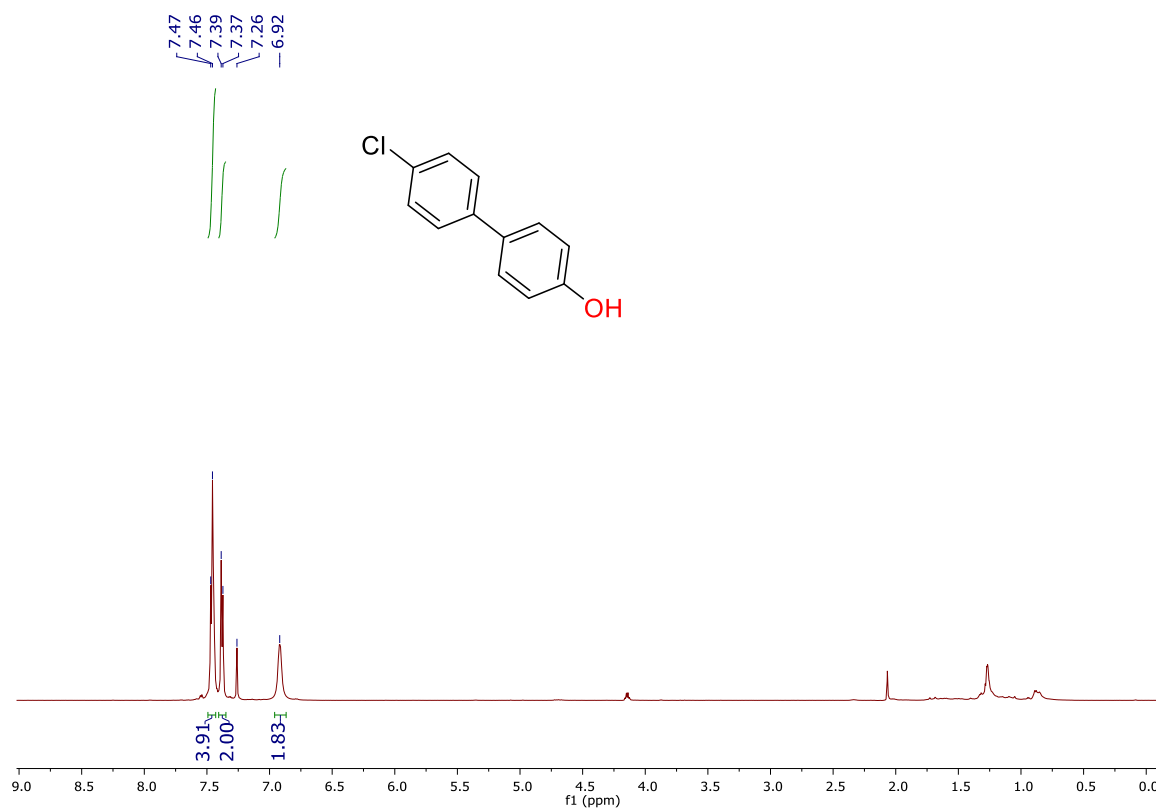
^1H NMR spectrum of **3c**



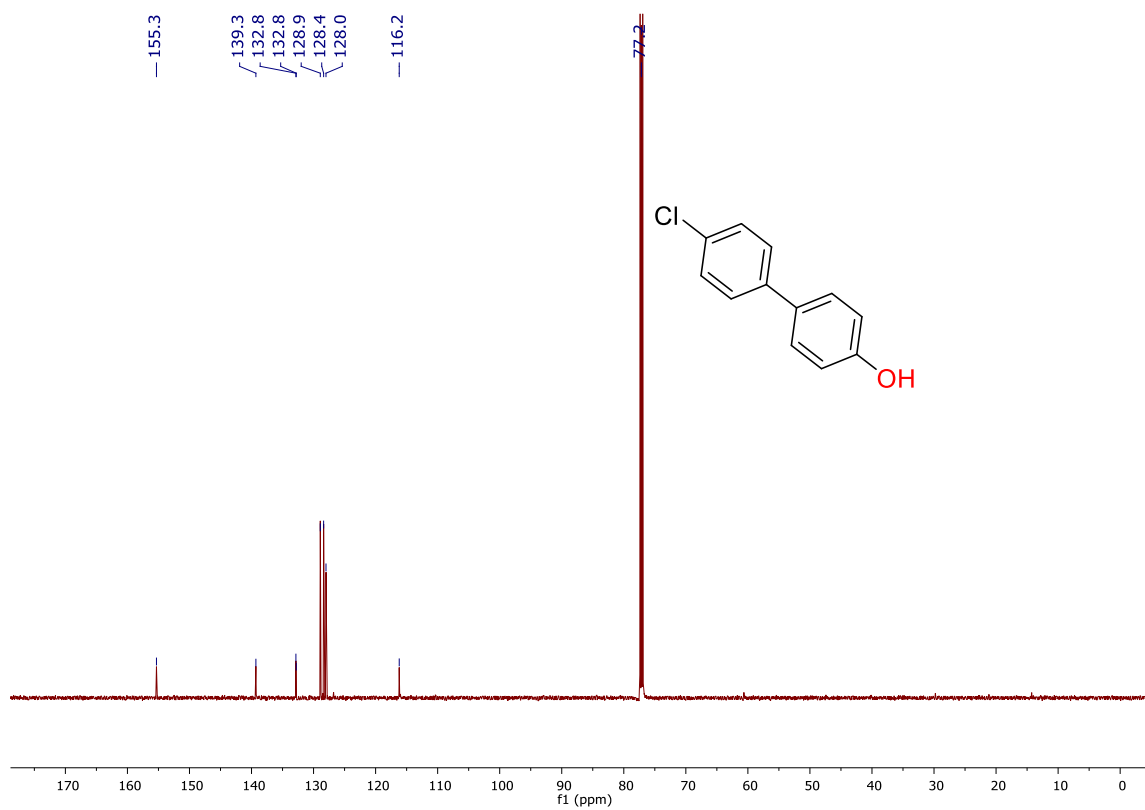
¹³C NMR spectrum of **3c**



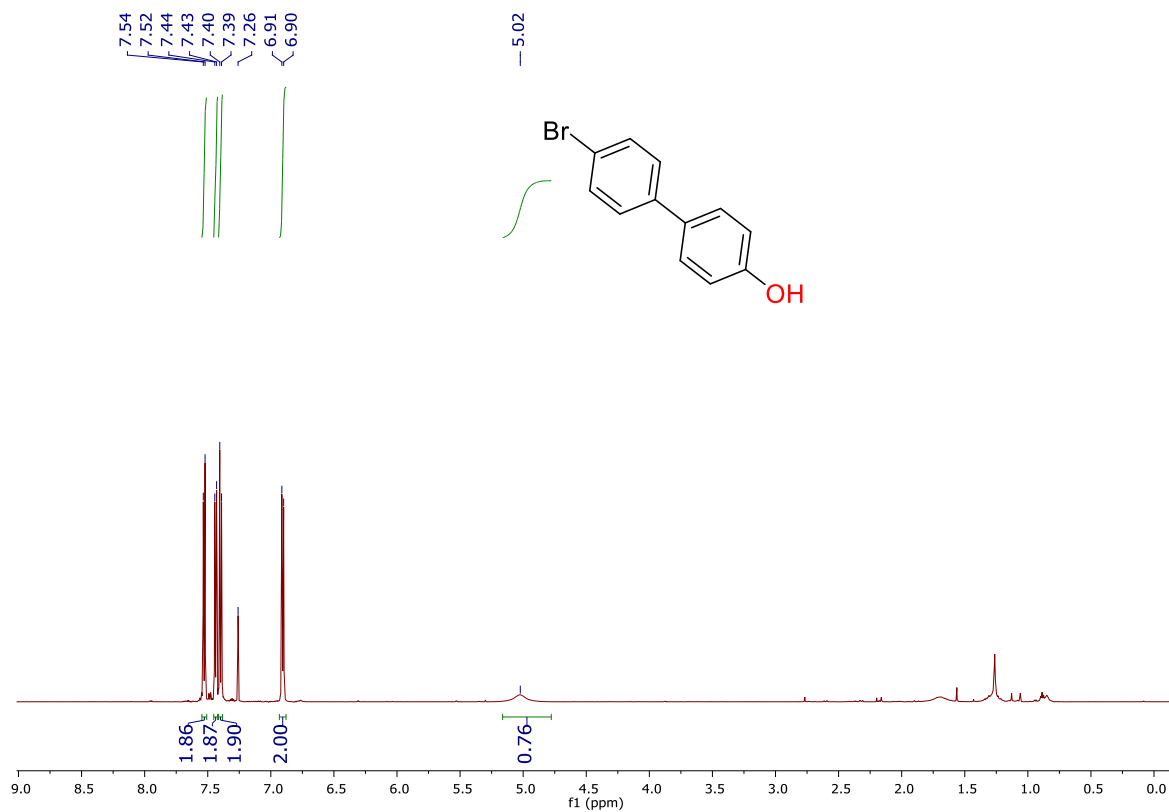
¹H NMR spectrum of **3d**



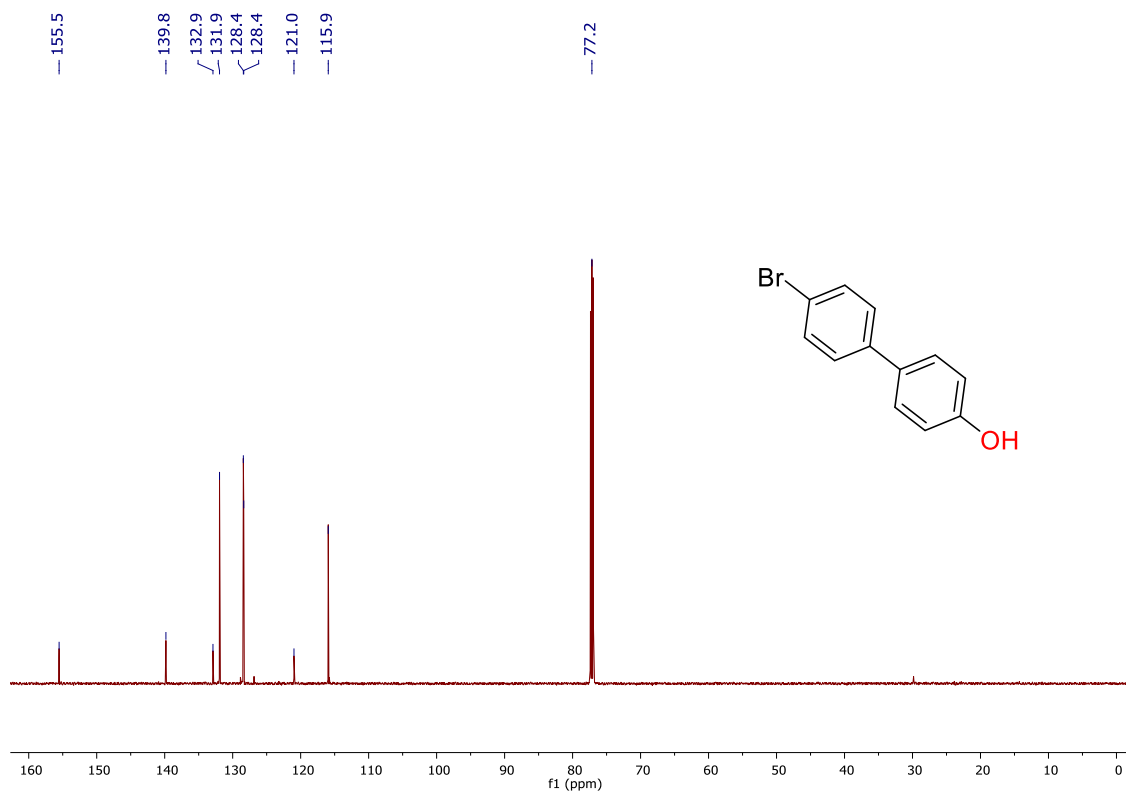
^{13}C NMR spectrum of 3d



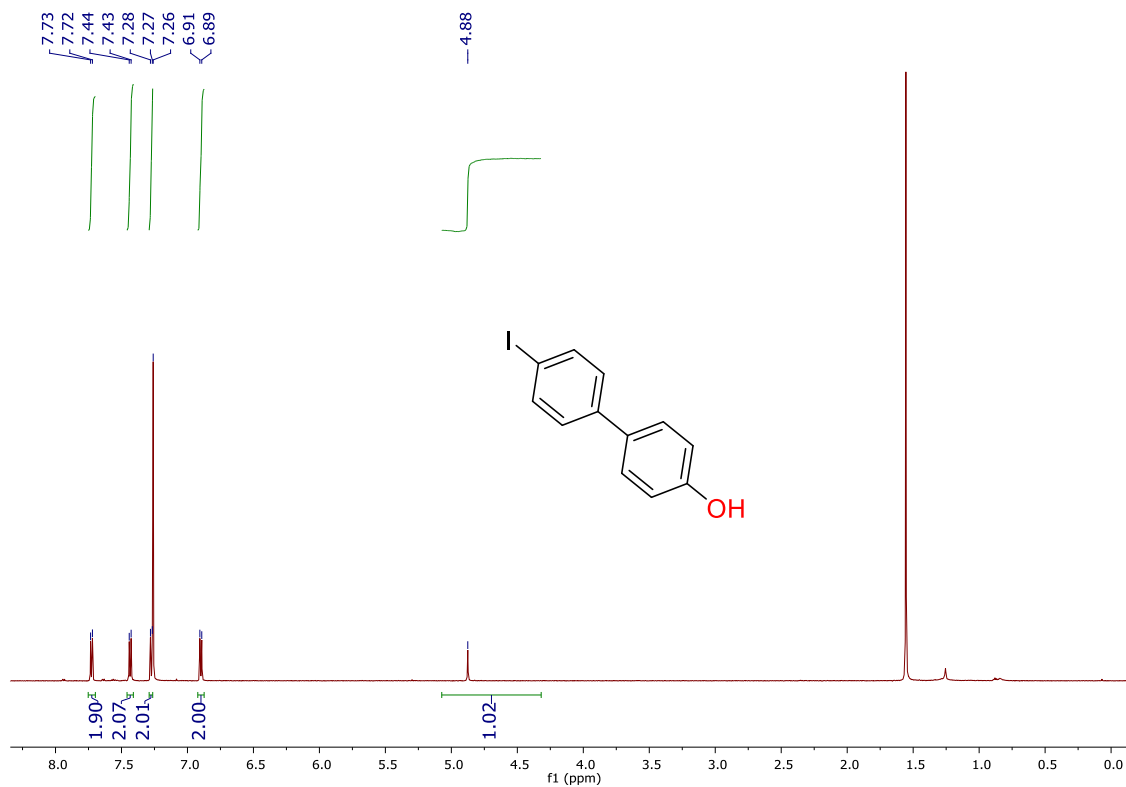
^1H NMR spectrum of 3e



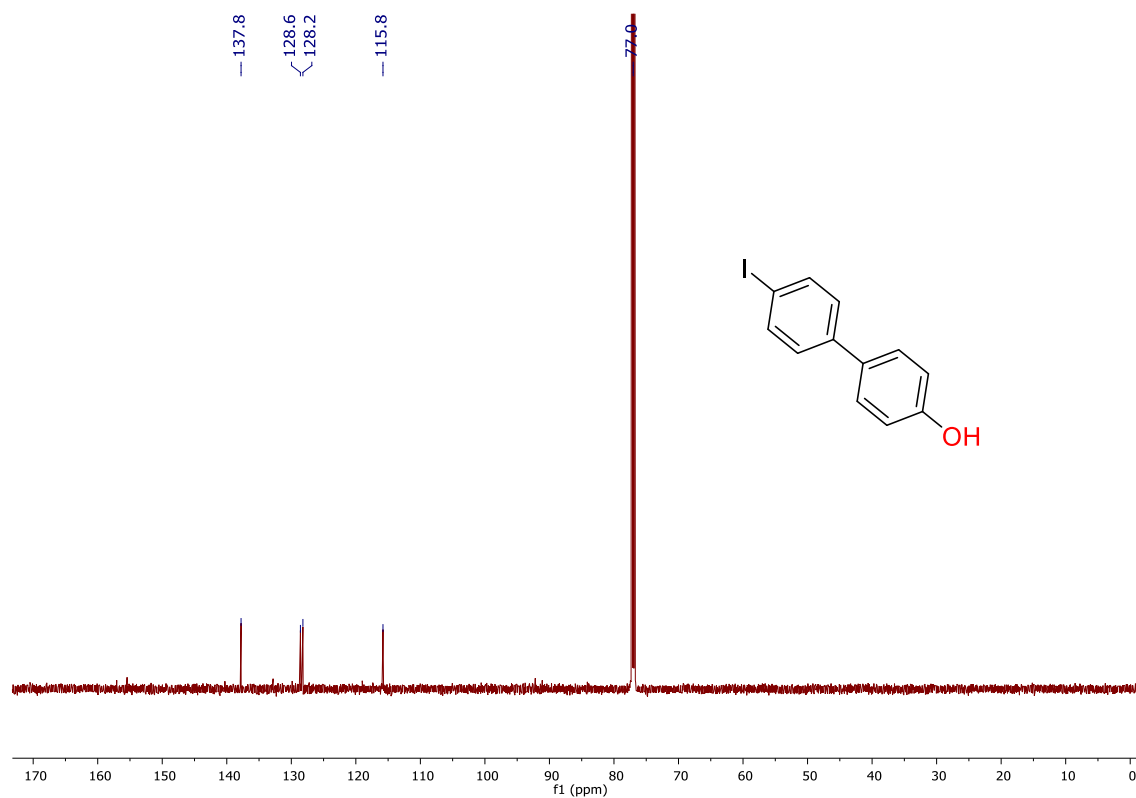
¹³C NMR spectrum of 3e



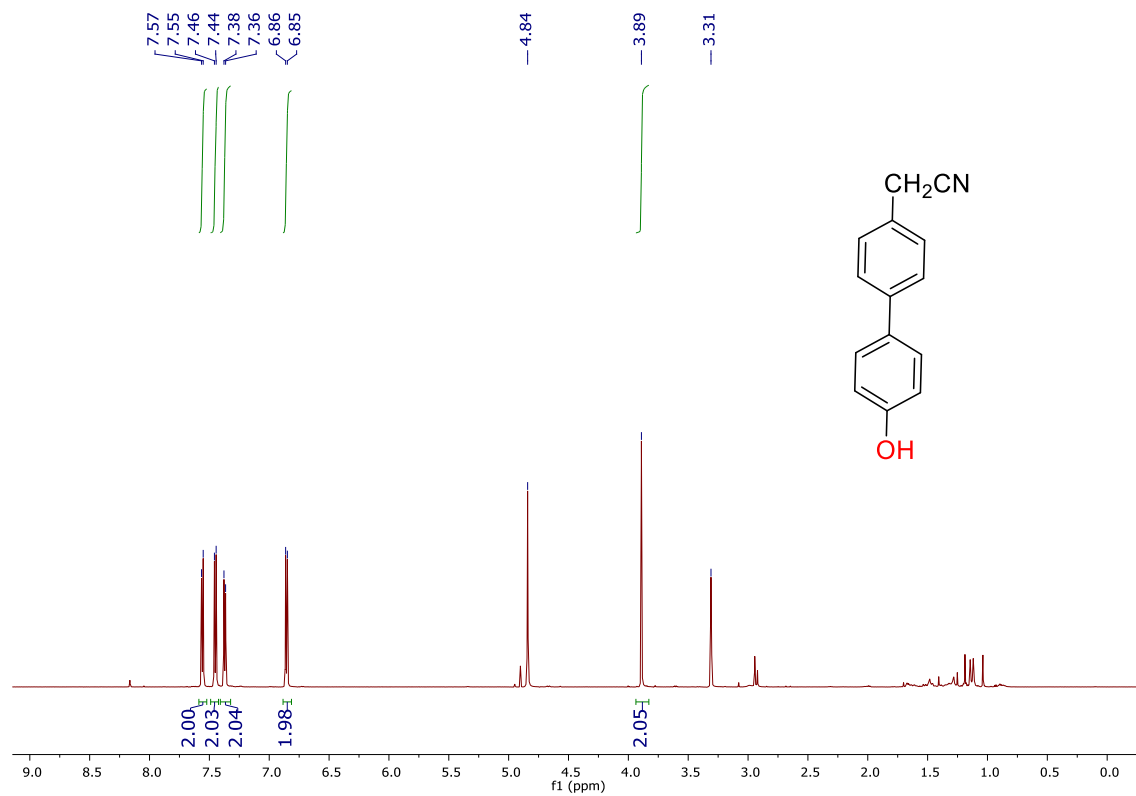
¹H NMR spectrum of 3f



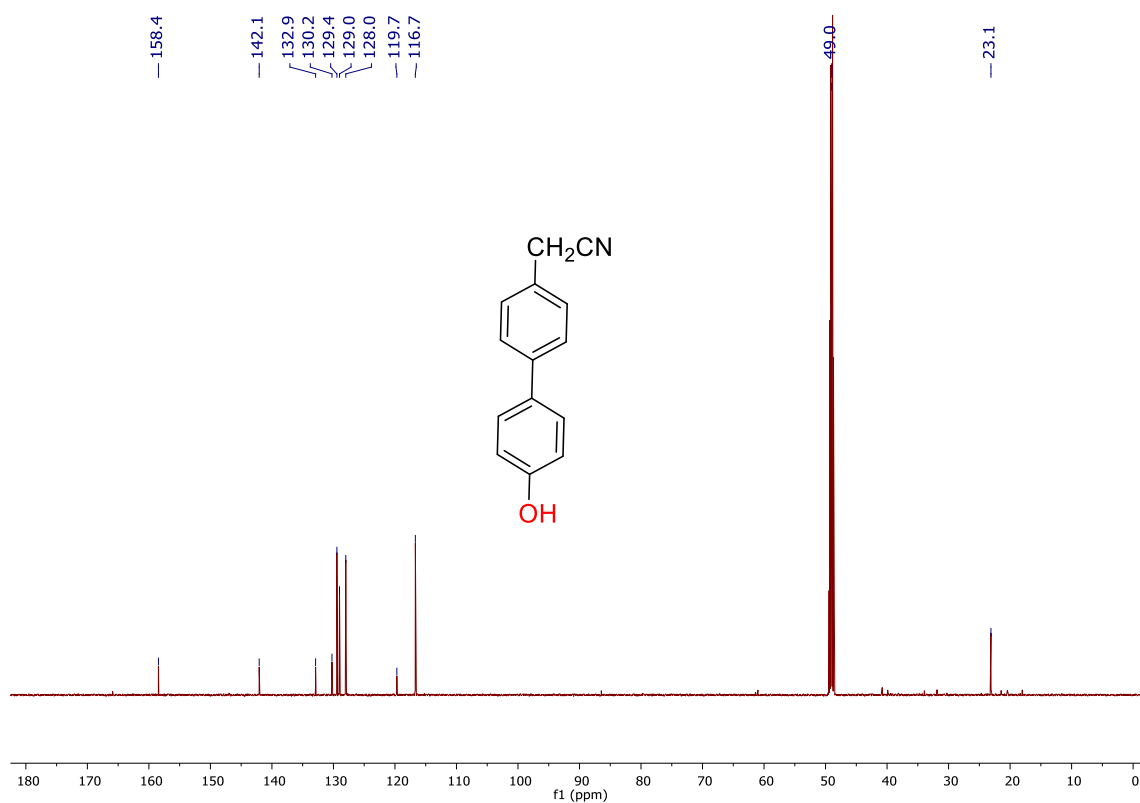
^{13}C NMR spectrum of **3f**



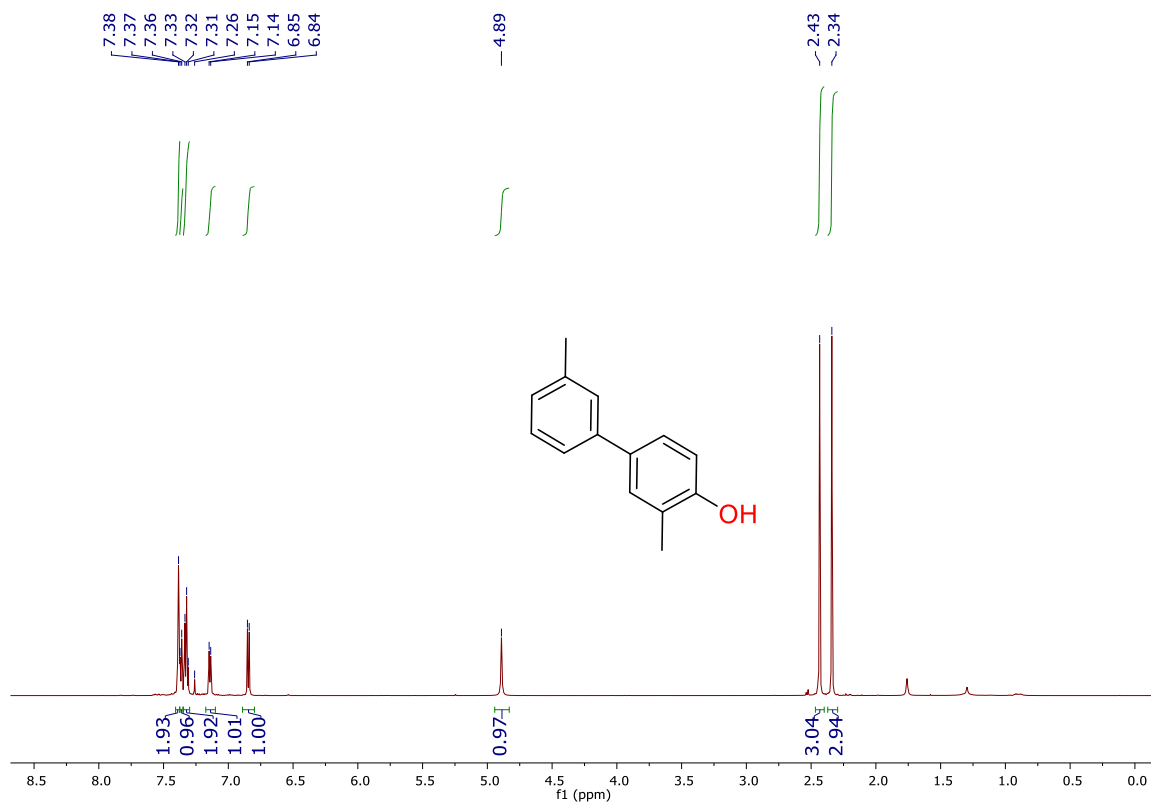
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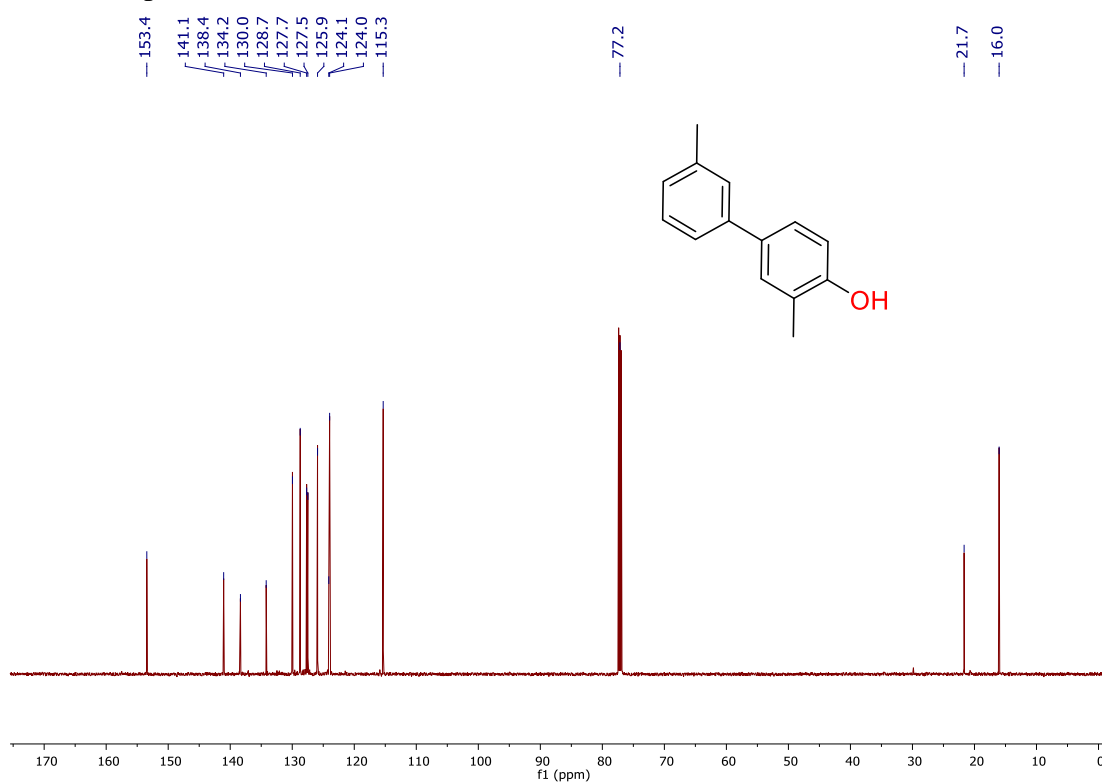
¹³C NMR spectrum of **3g**



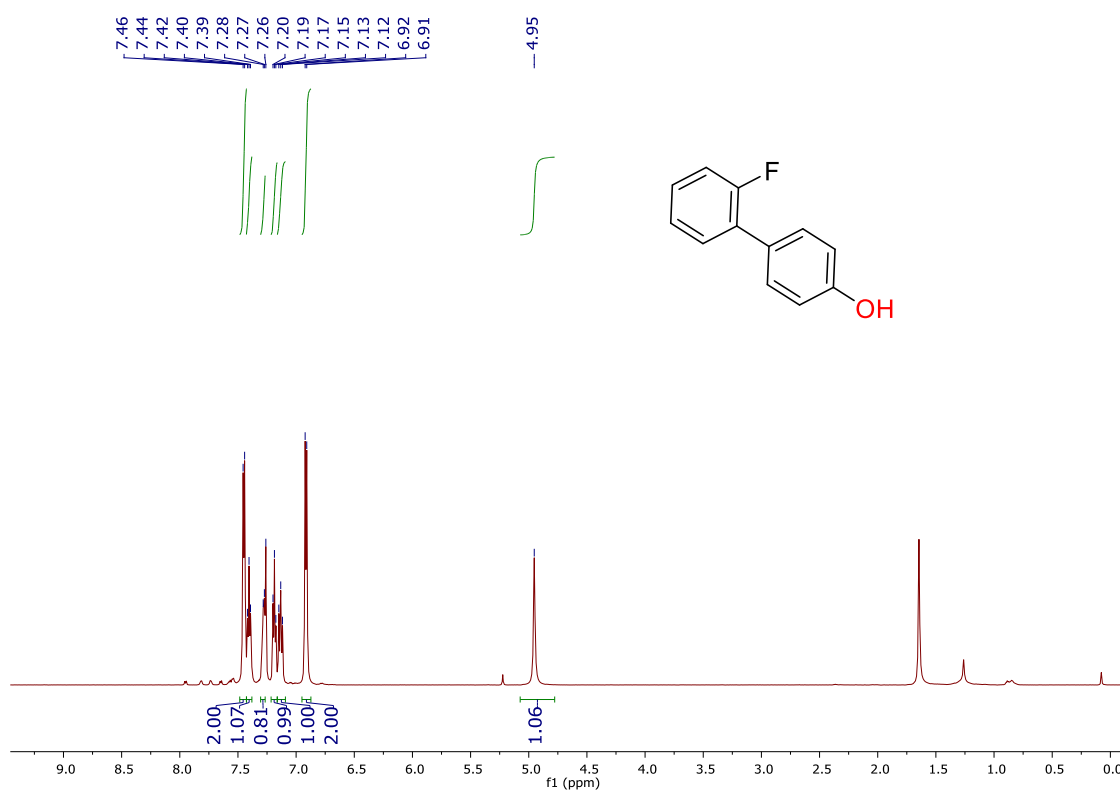
¹H NMR spectrum of **3h**



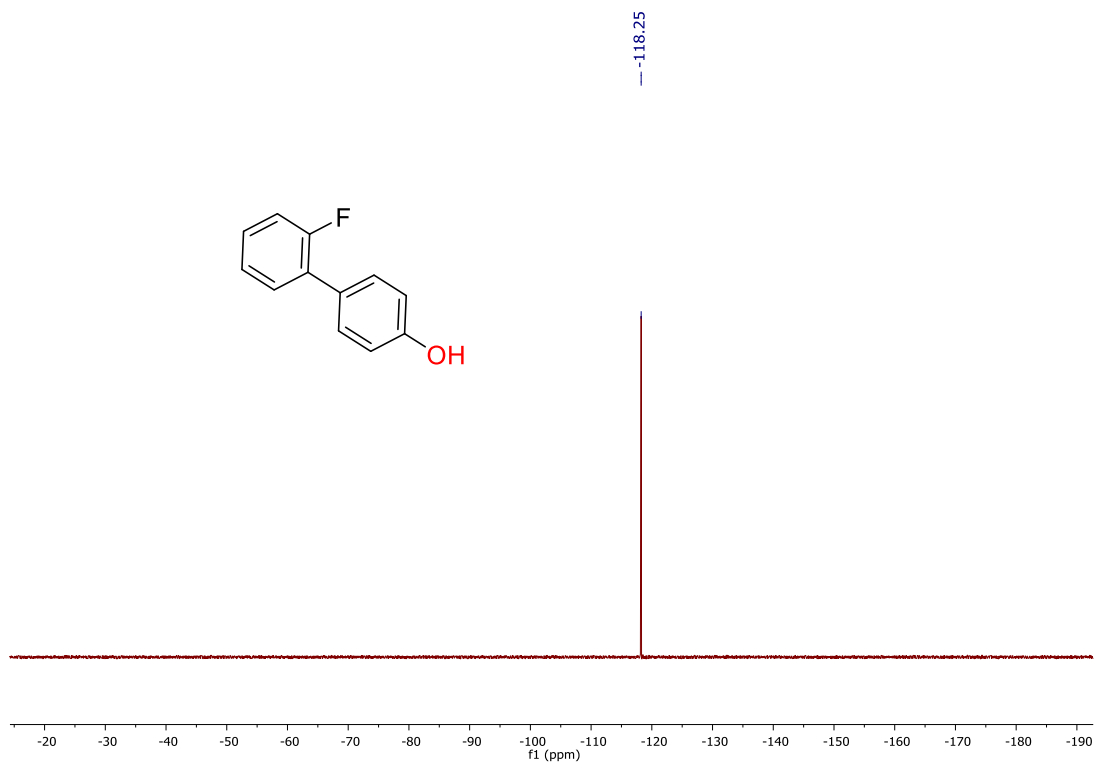
¹³C NMR spectrum of 3h



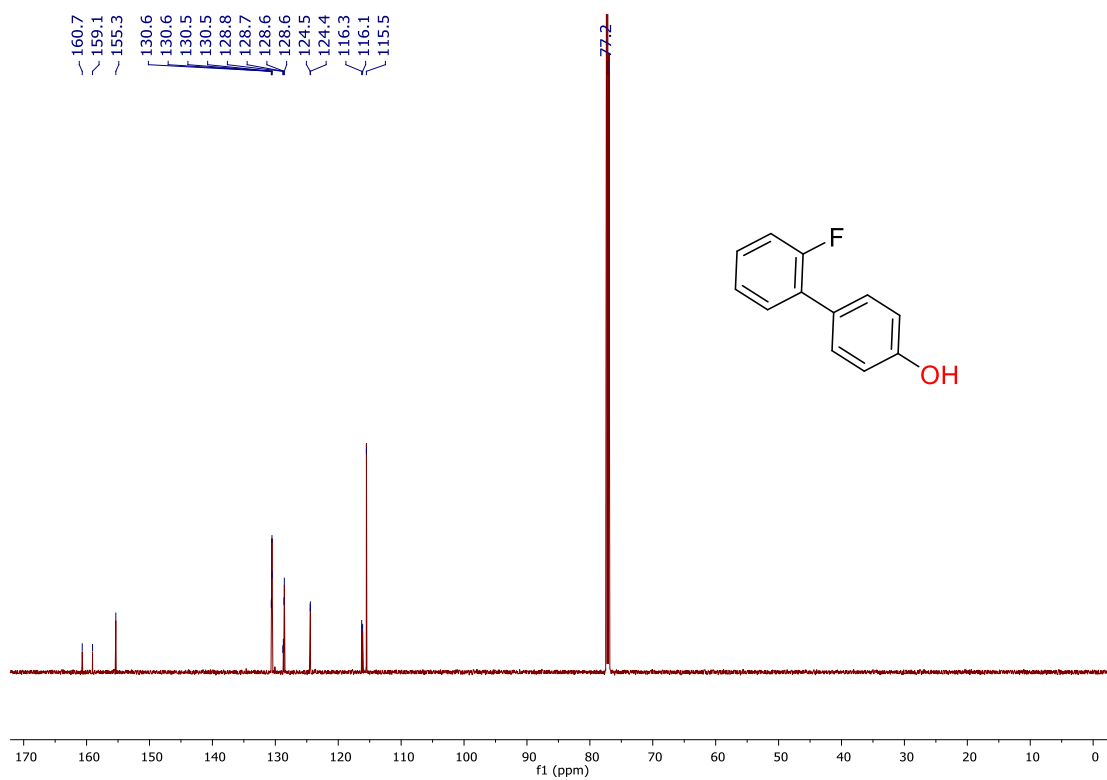
¹H NMR spectrum of 3i



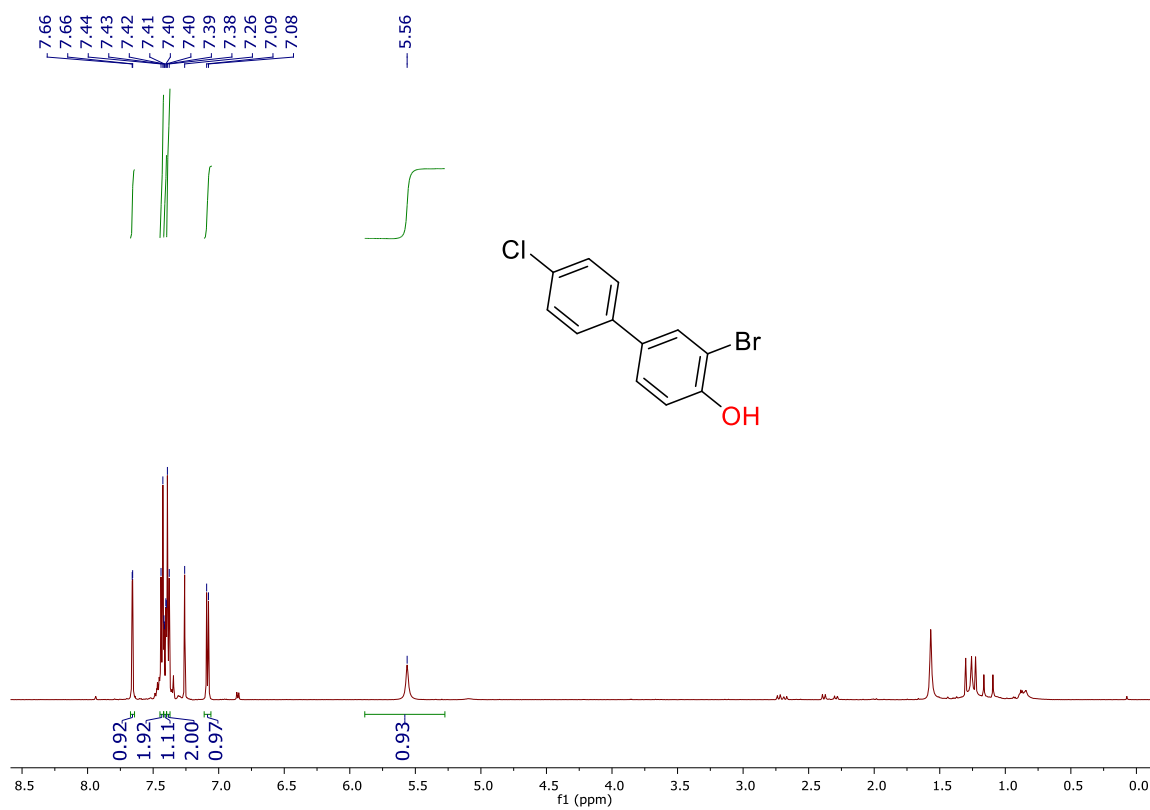
^{19}F NMR spectrum of **3i**



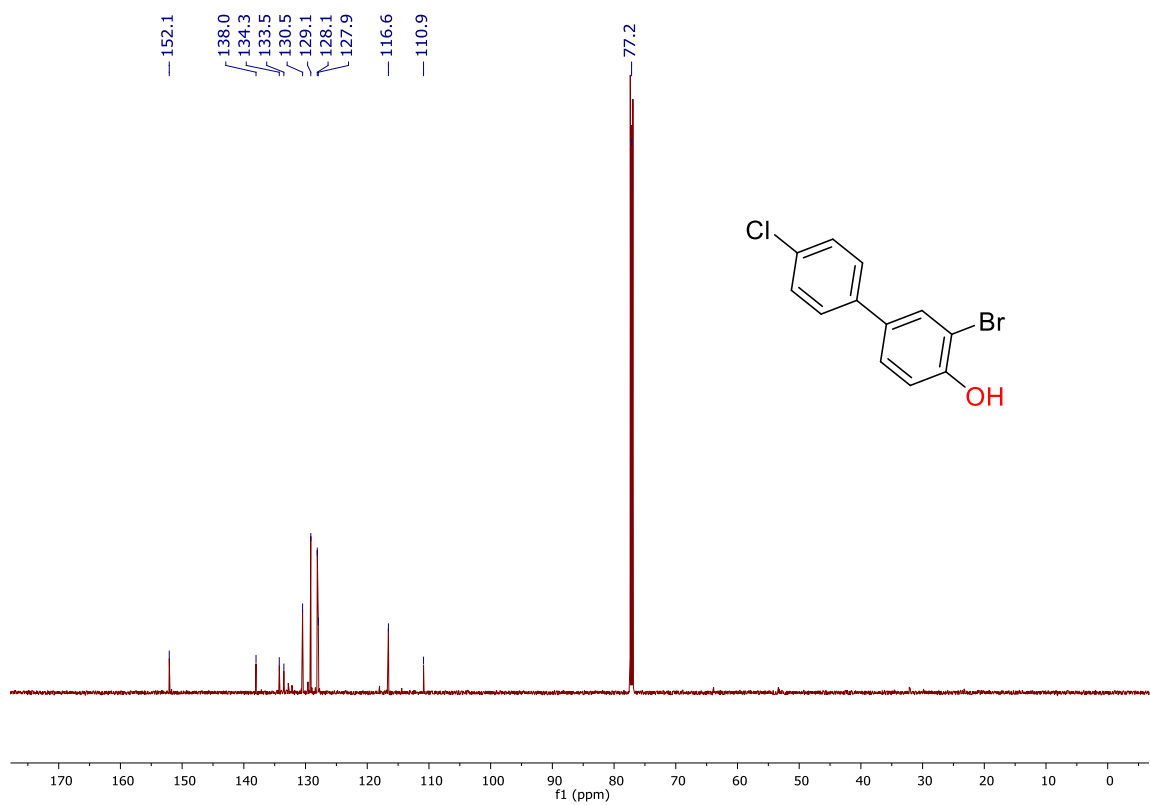
^{13}C NMR spectrum of **3i**



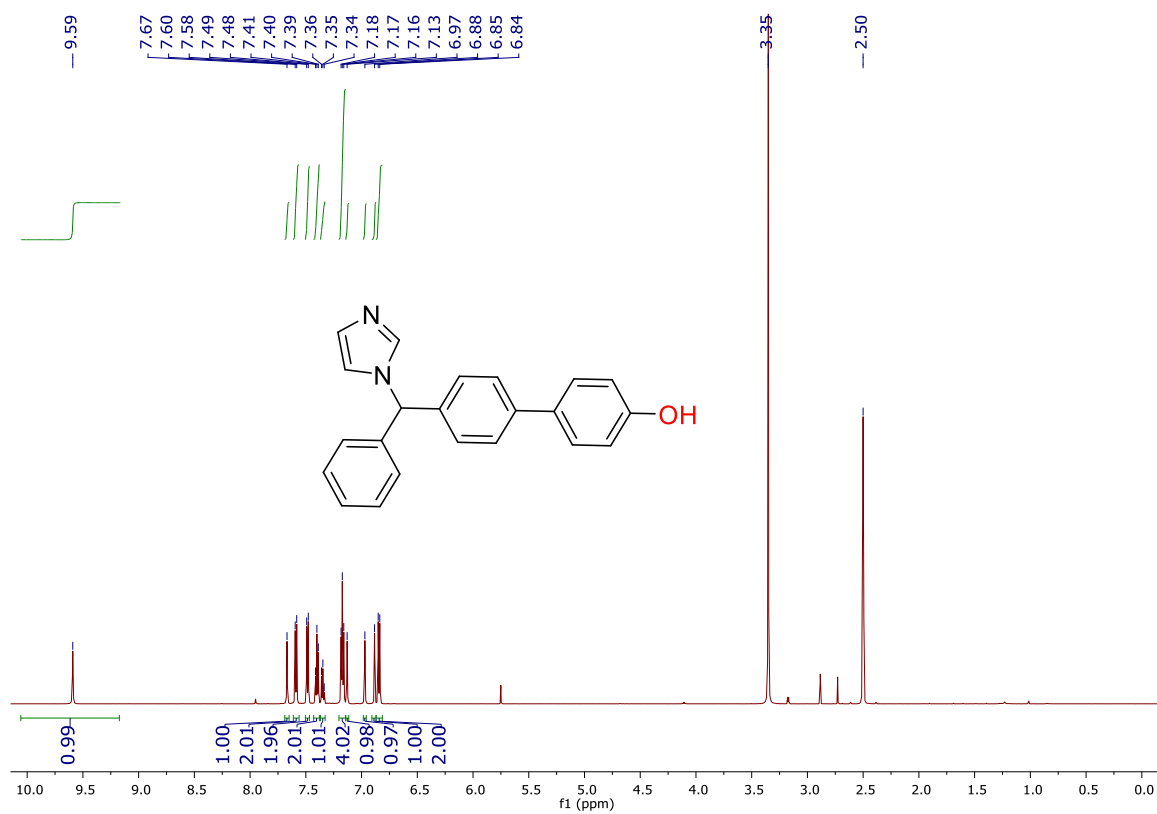
¹H NMR spectrum of 3j



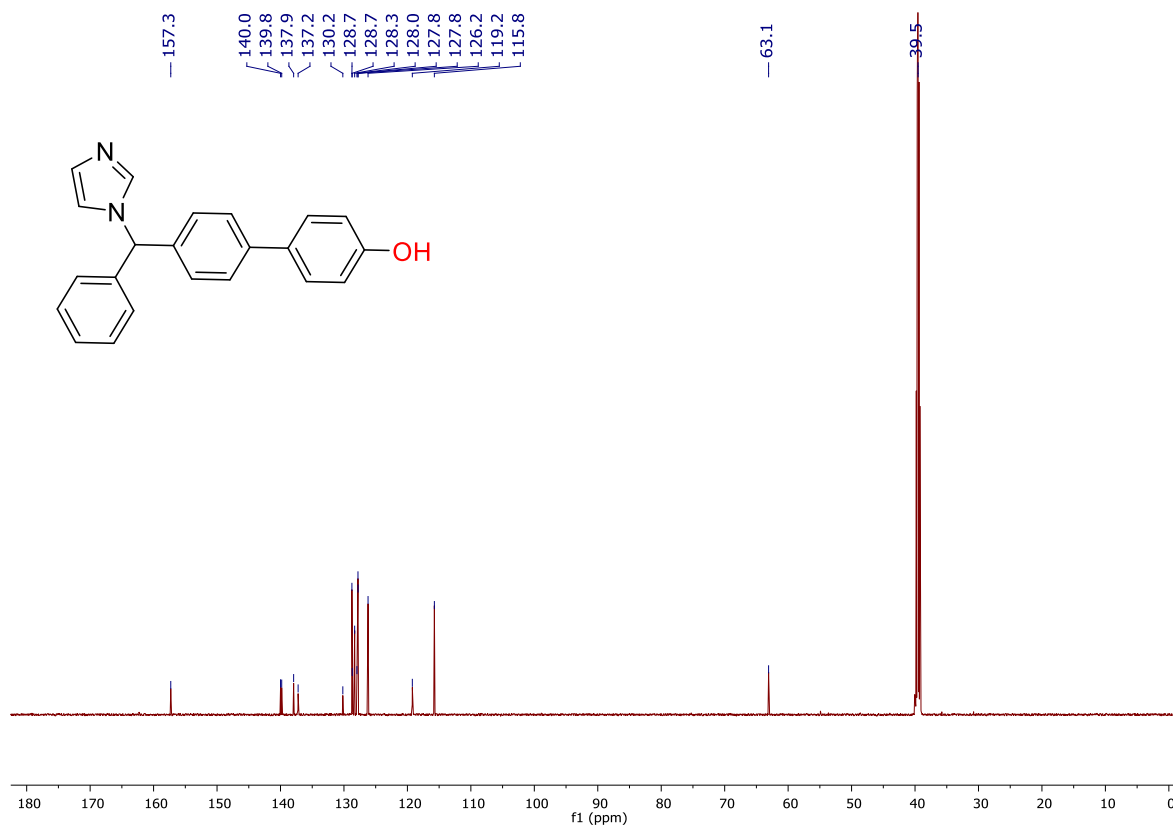
¹³C NMR spectrum of 3j



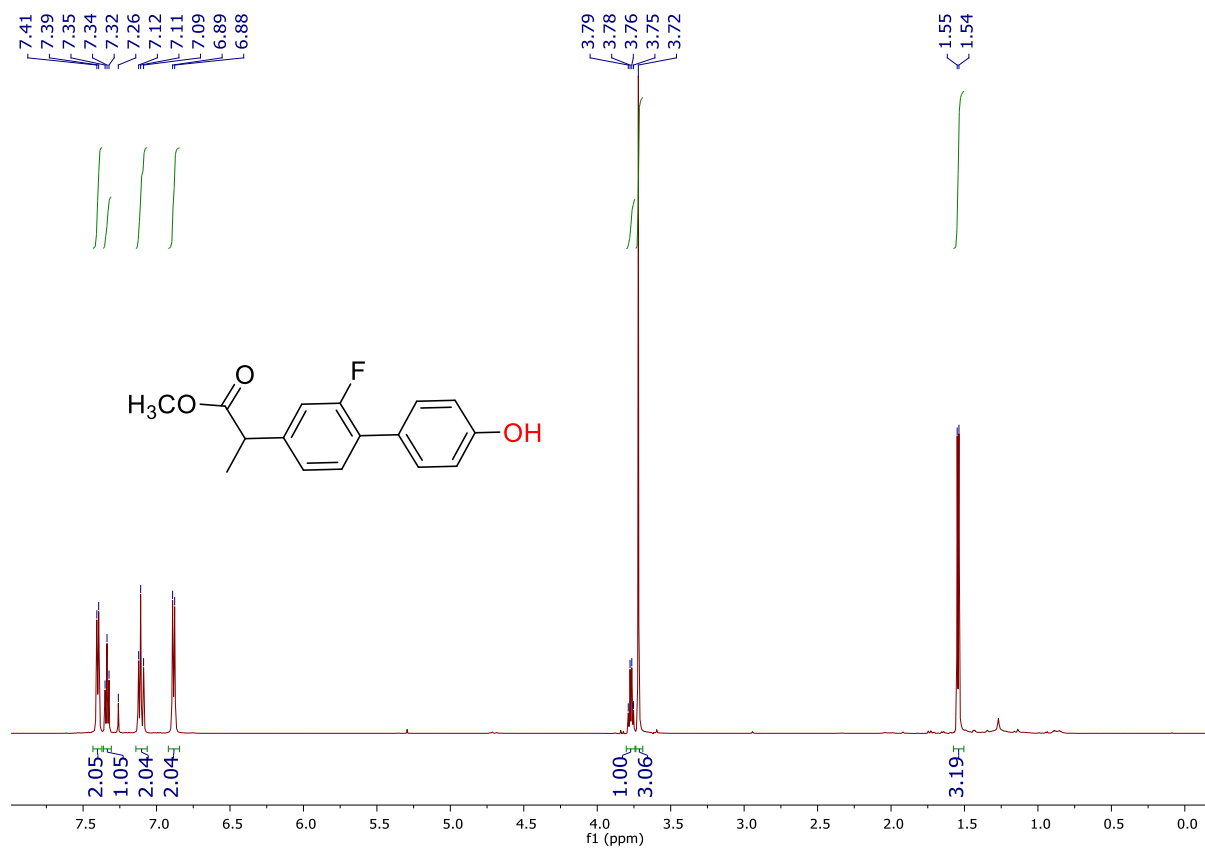
¹H NMR spectrum of 3k



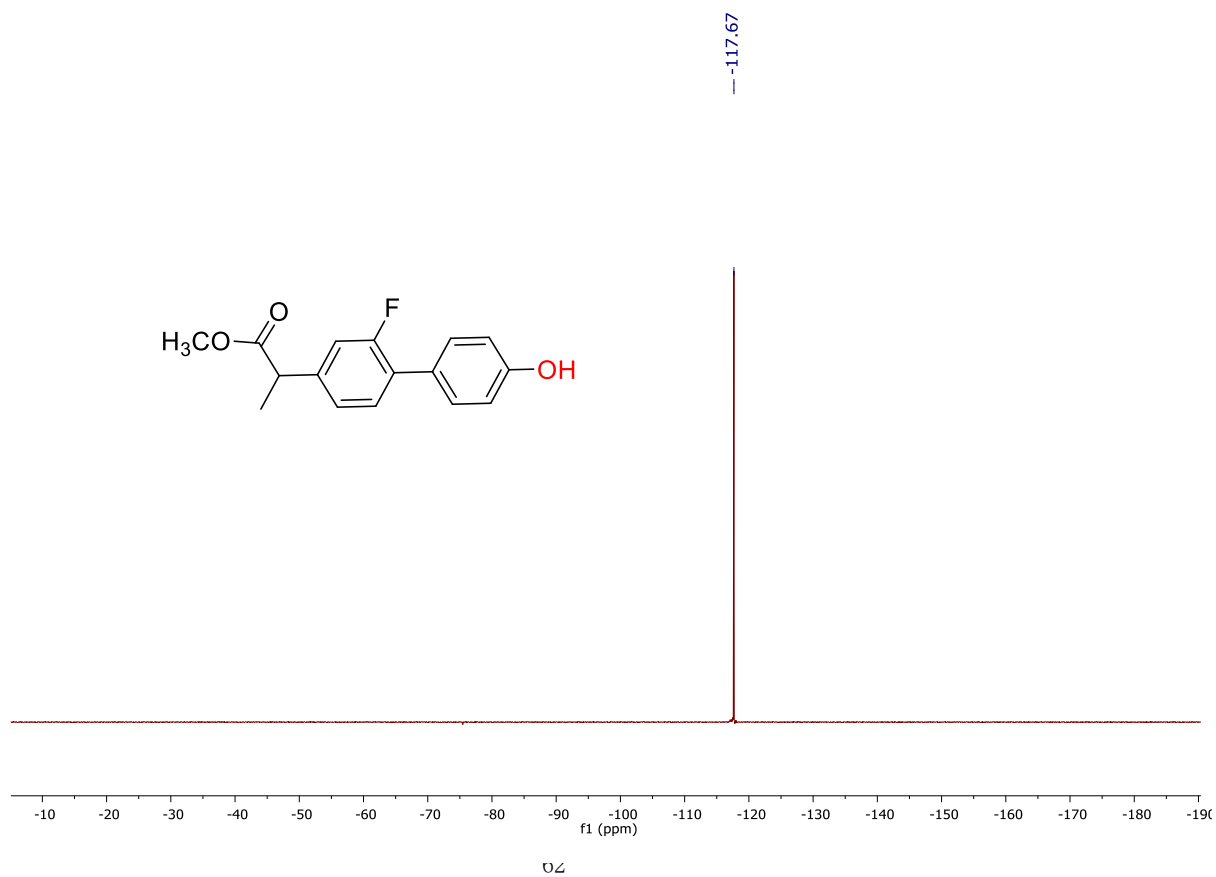
¹³C NMR spectrum of 3k



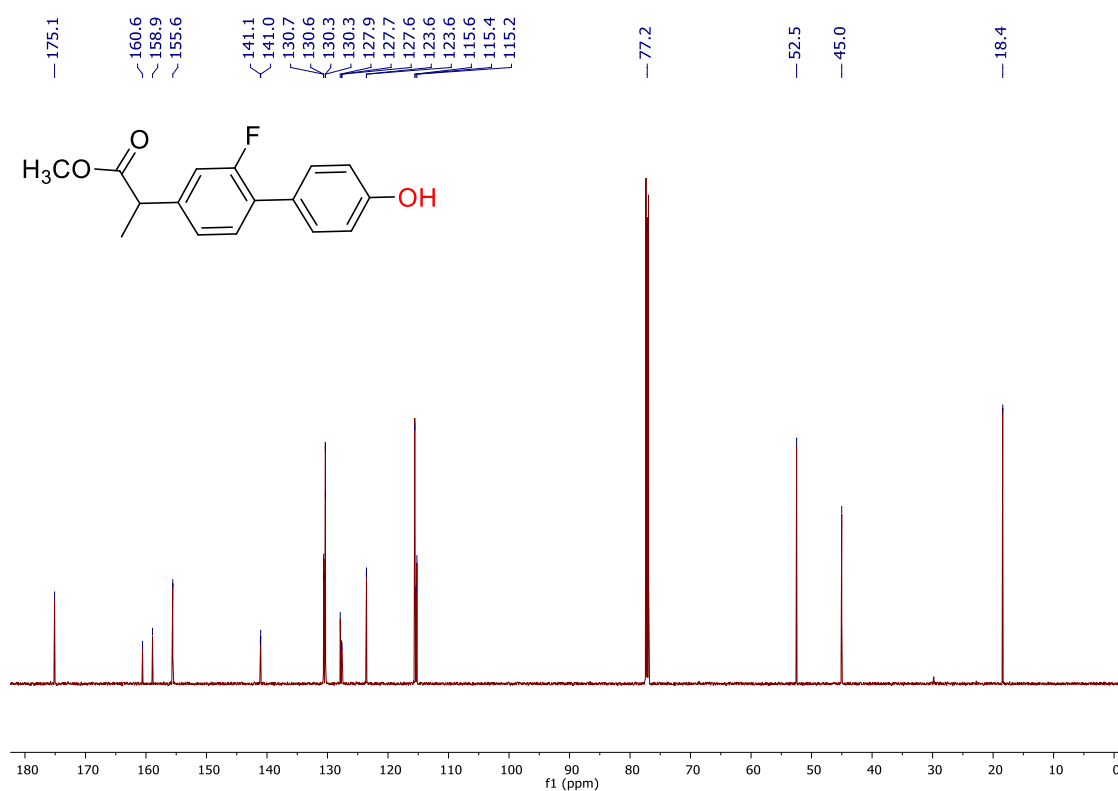
¹H NMR spectrum of 31



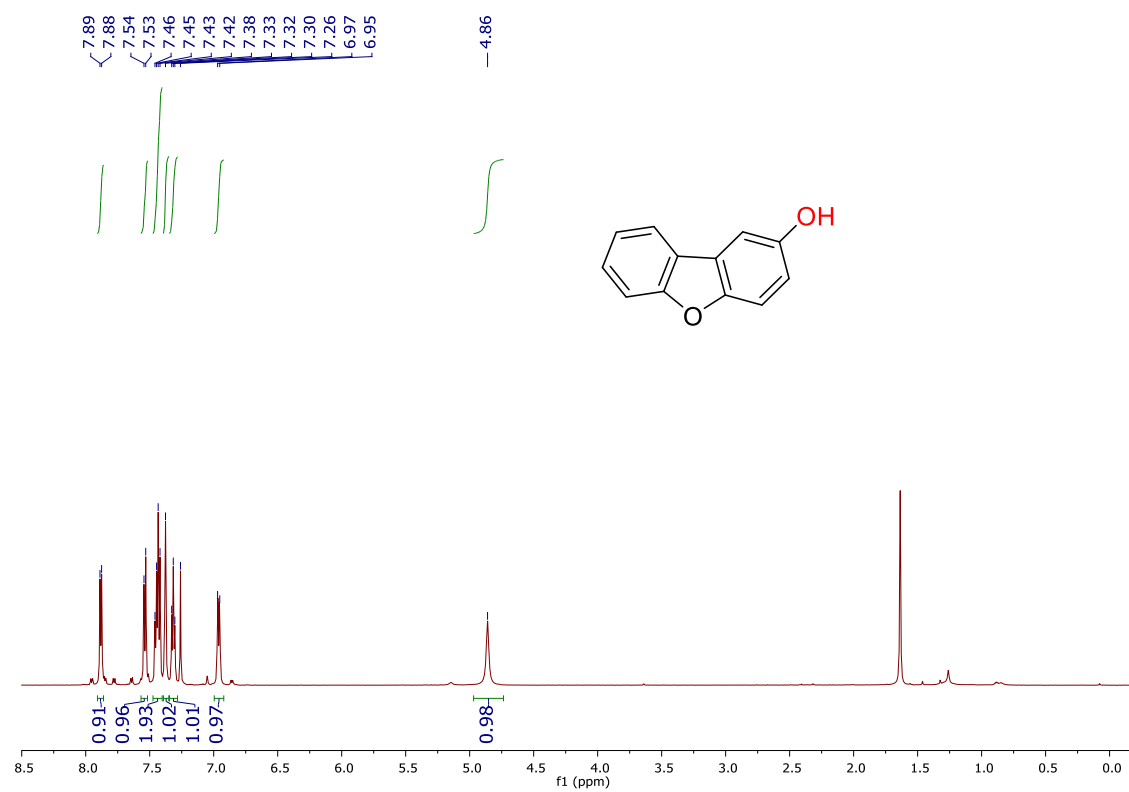
¹⁹F NMR spectrum of 31



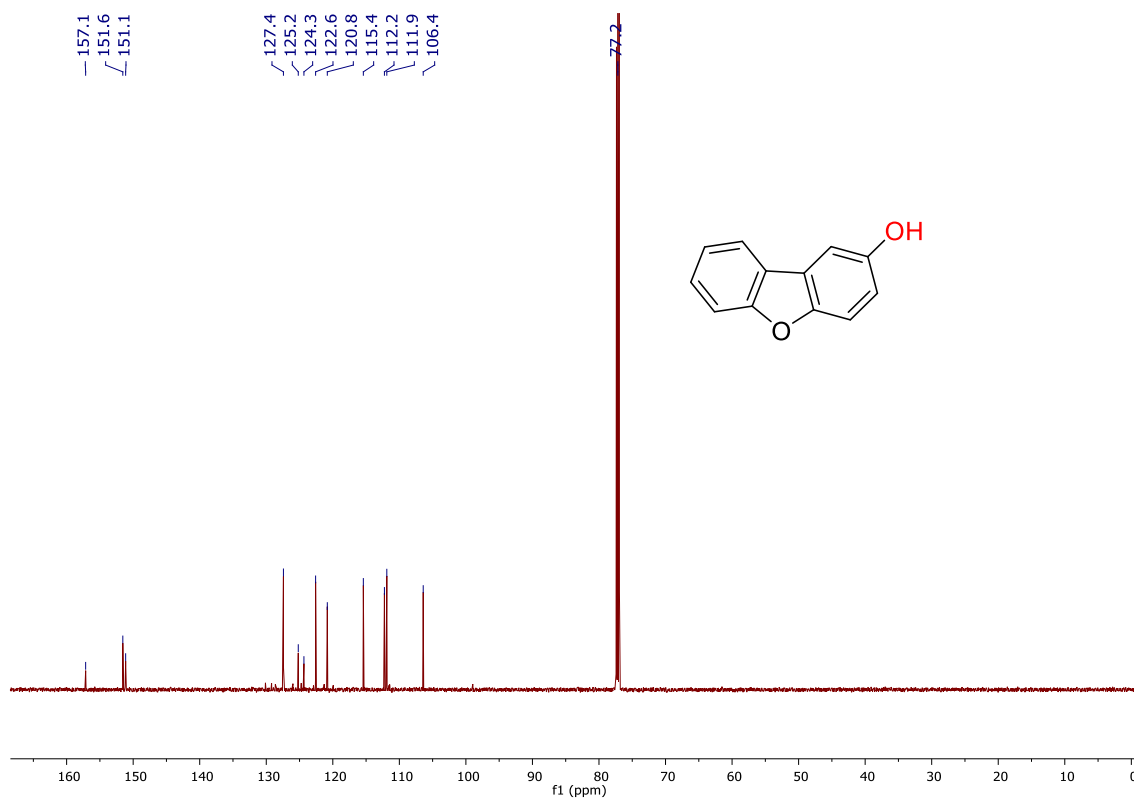
¹³C NMR spectrum of 31



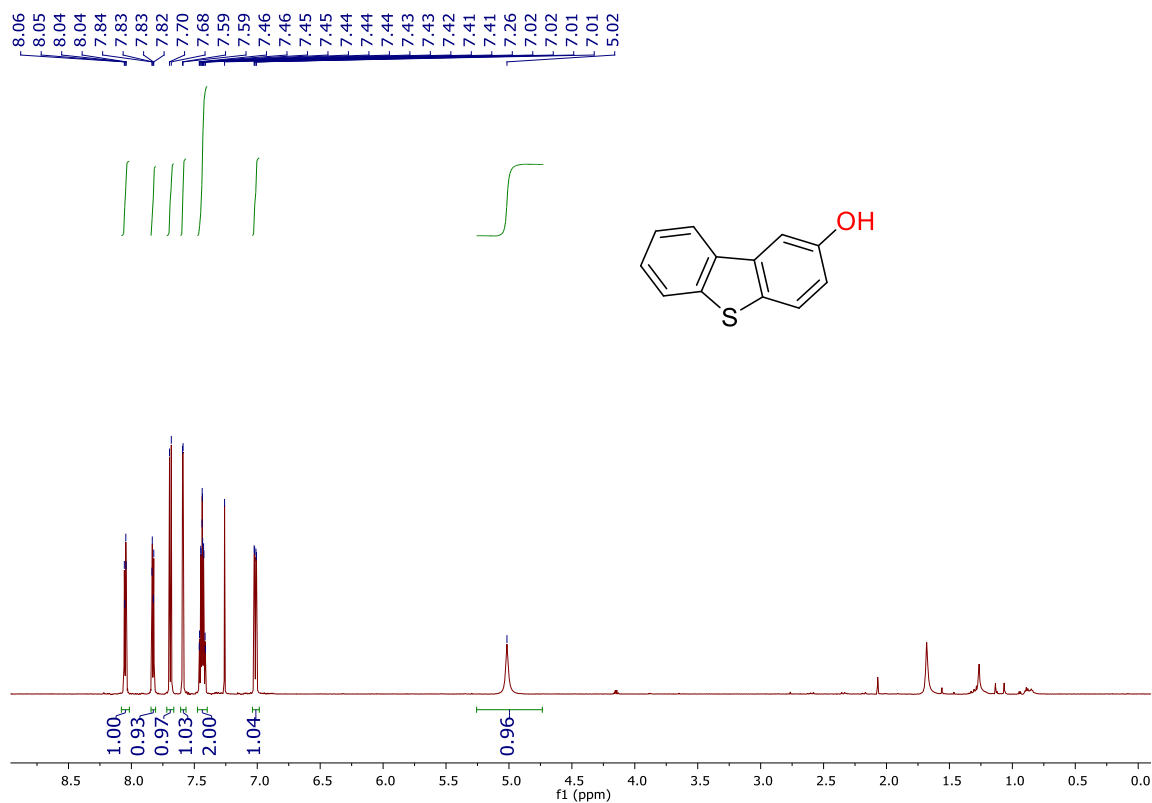
¹H NMR spectrum of 3m



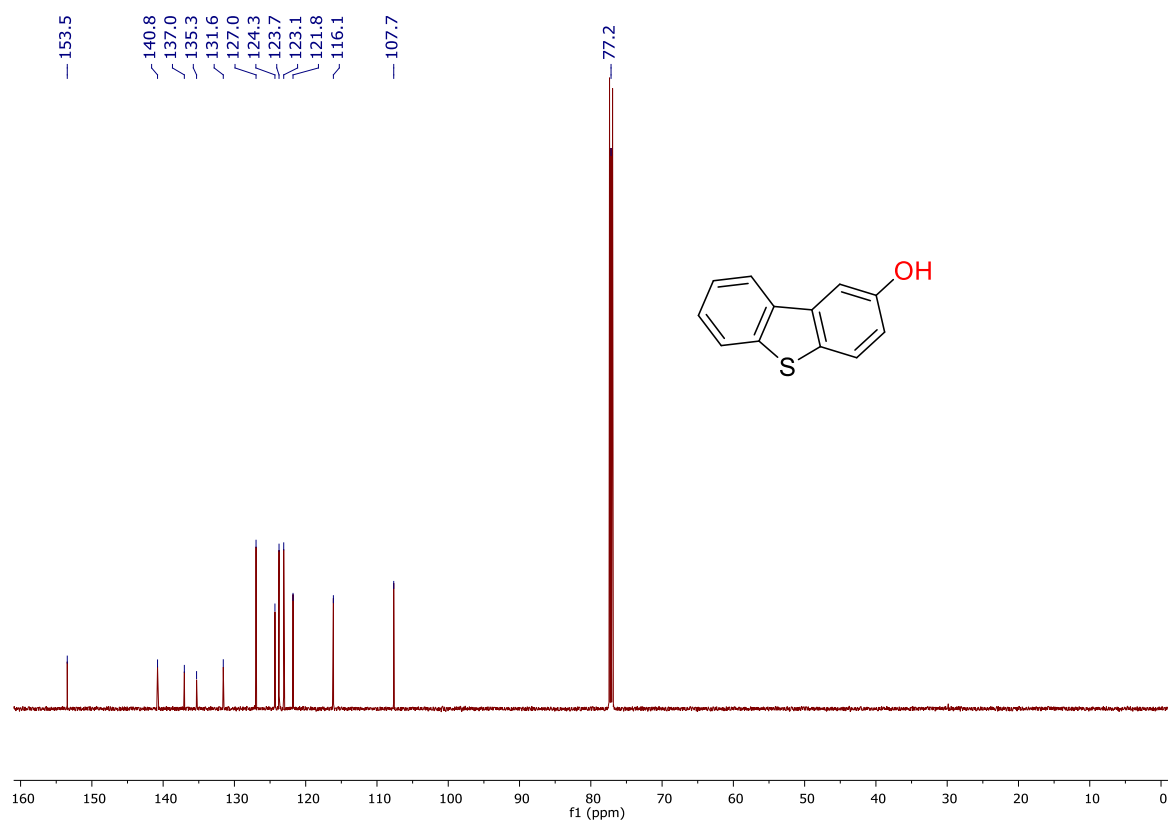
^{13}C NMR spectrum of **3m**



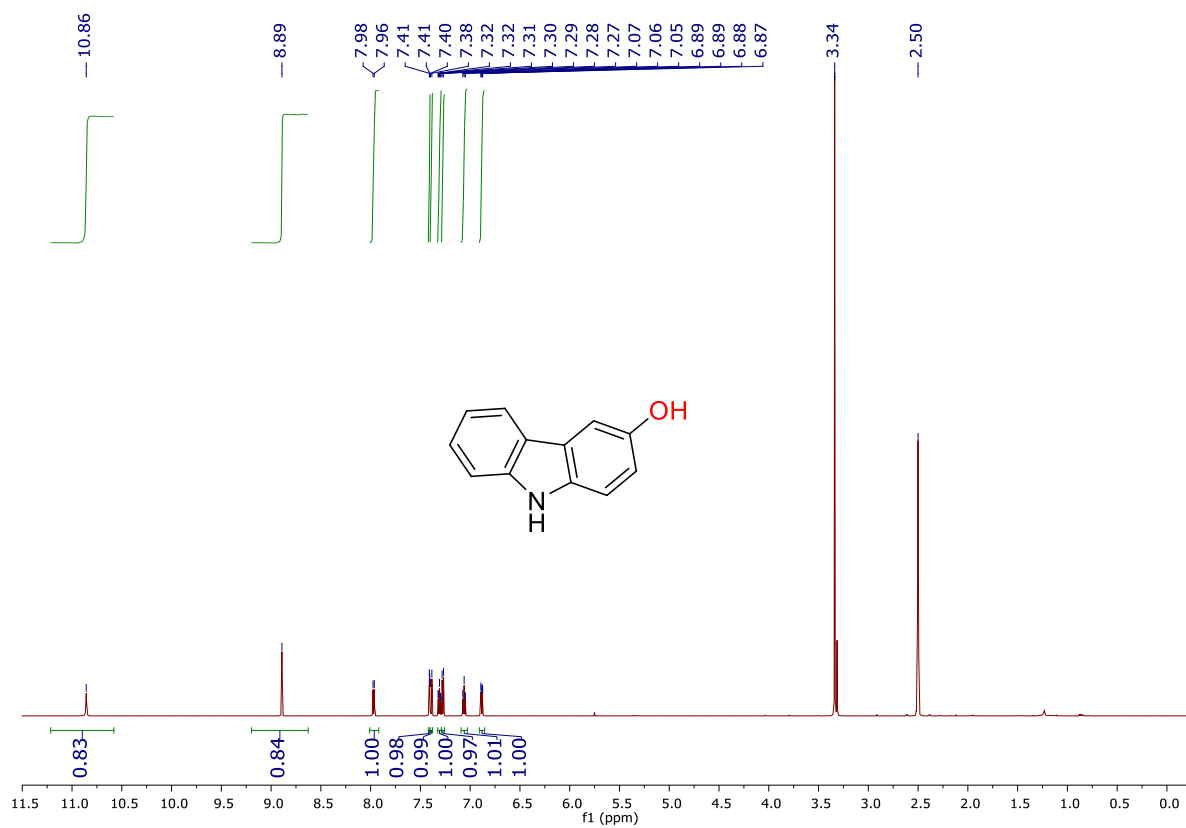
^1H NMR spectrum of **3n**



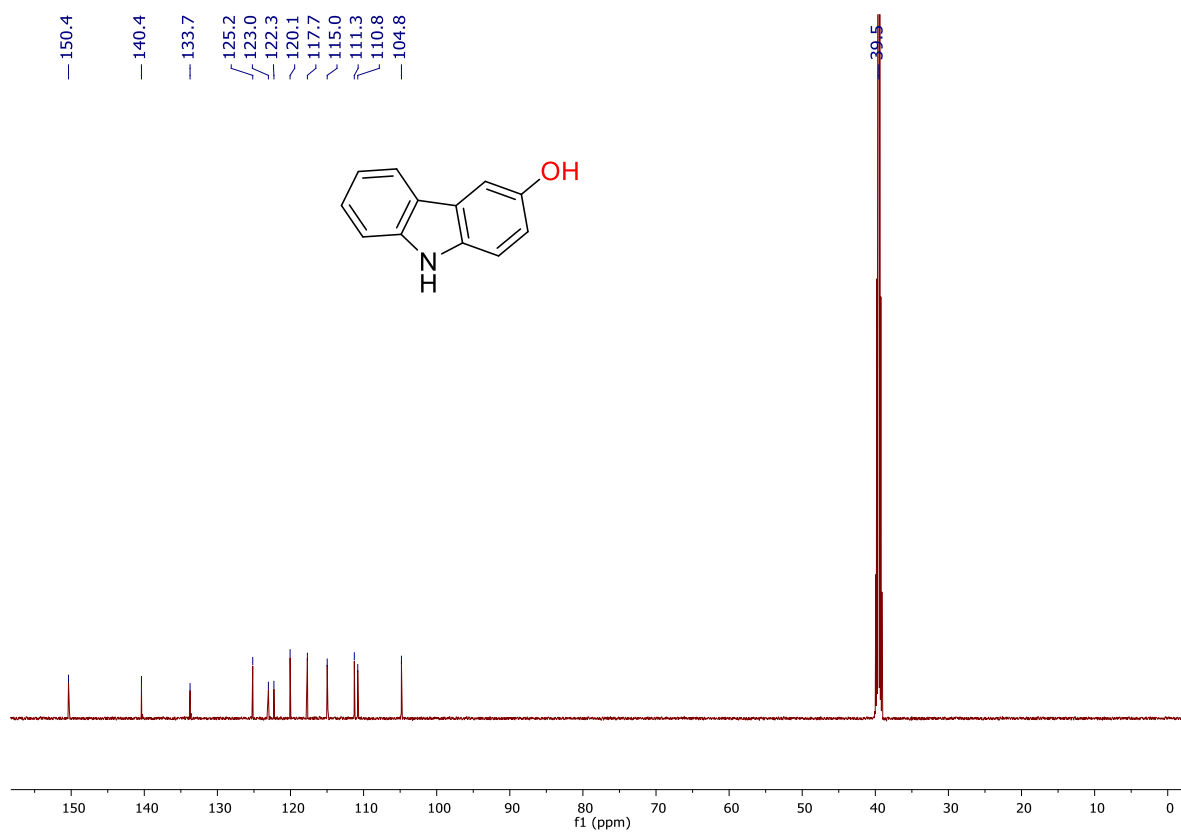
^{13}C NMR spectrum of **3n**



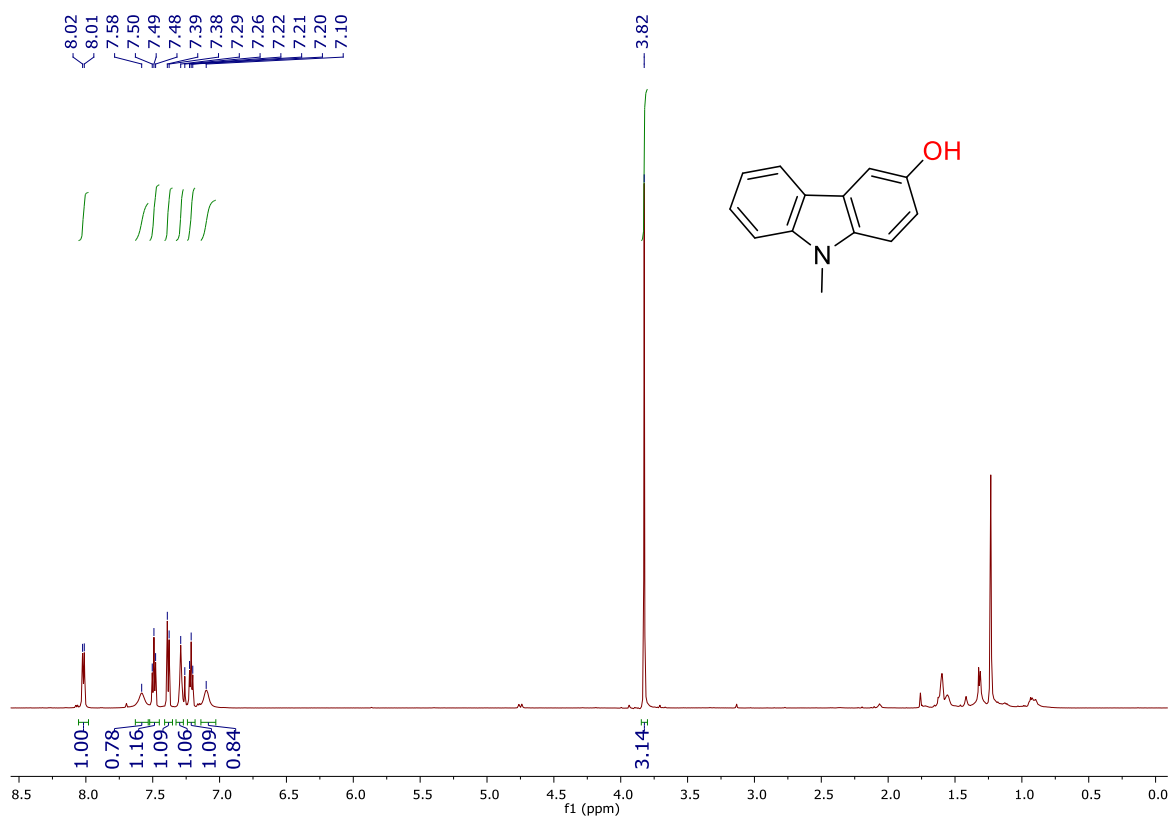
^1H NMR spectrum of **3o**



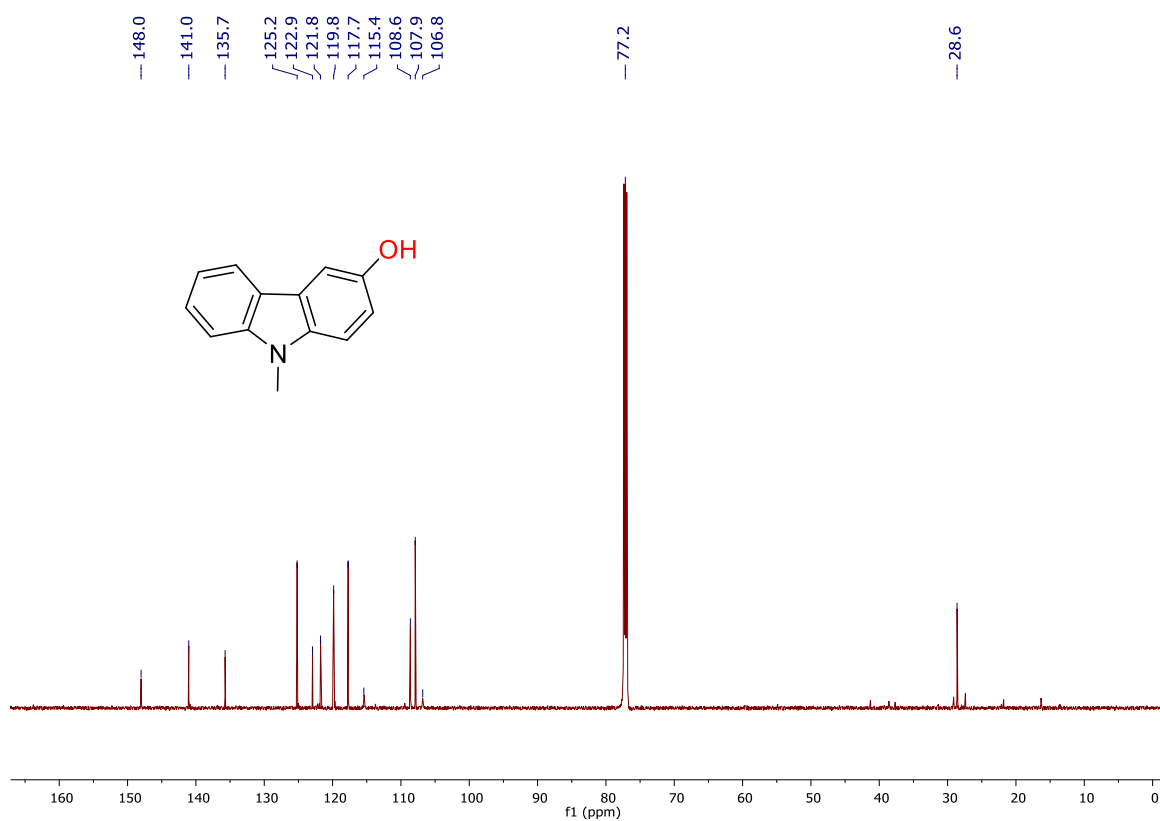
¹³C NMR spectrum of 3o



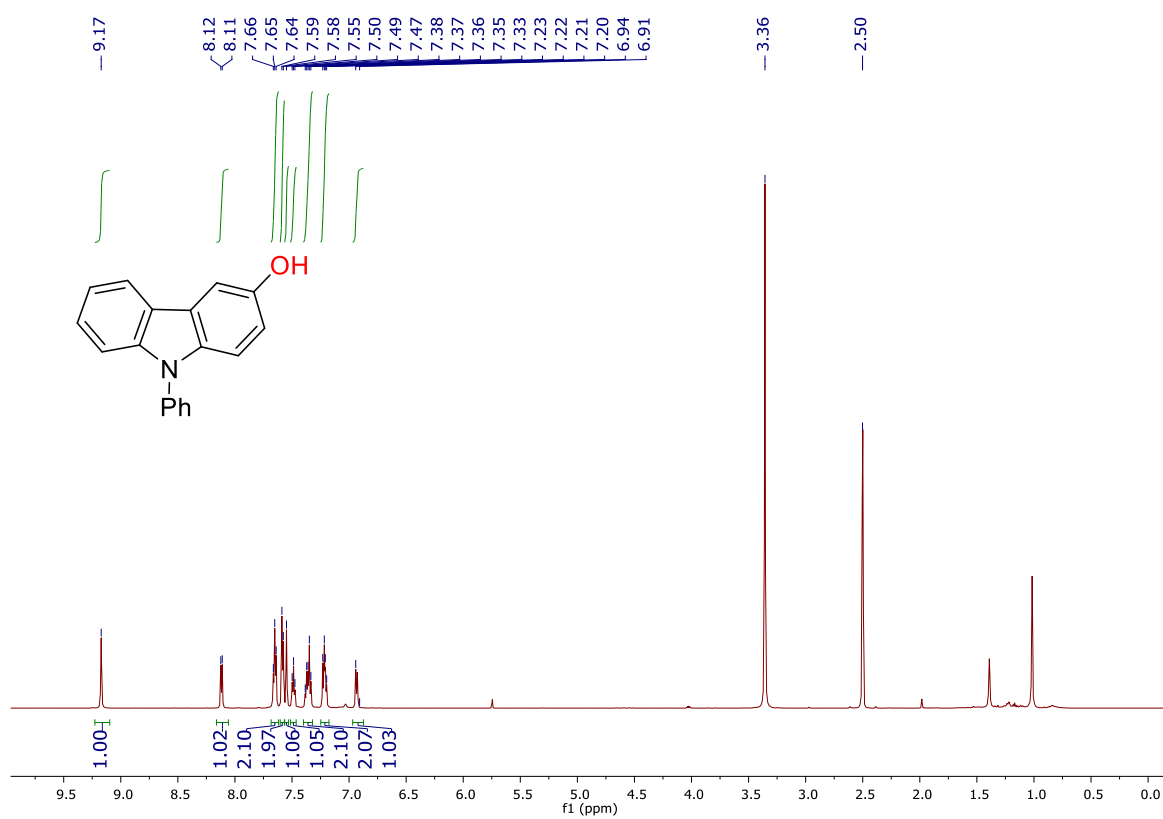
¹H NMR spectrum of 3p



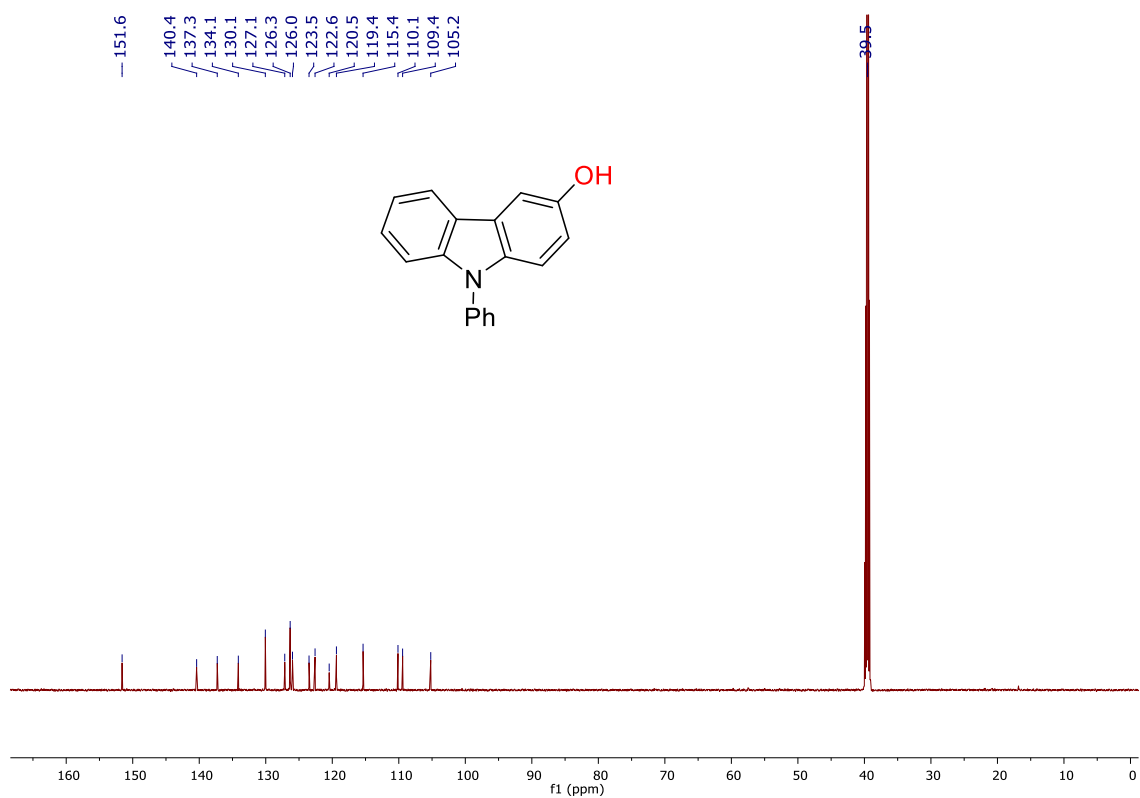
¹³C NMR spectrum of **3p**



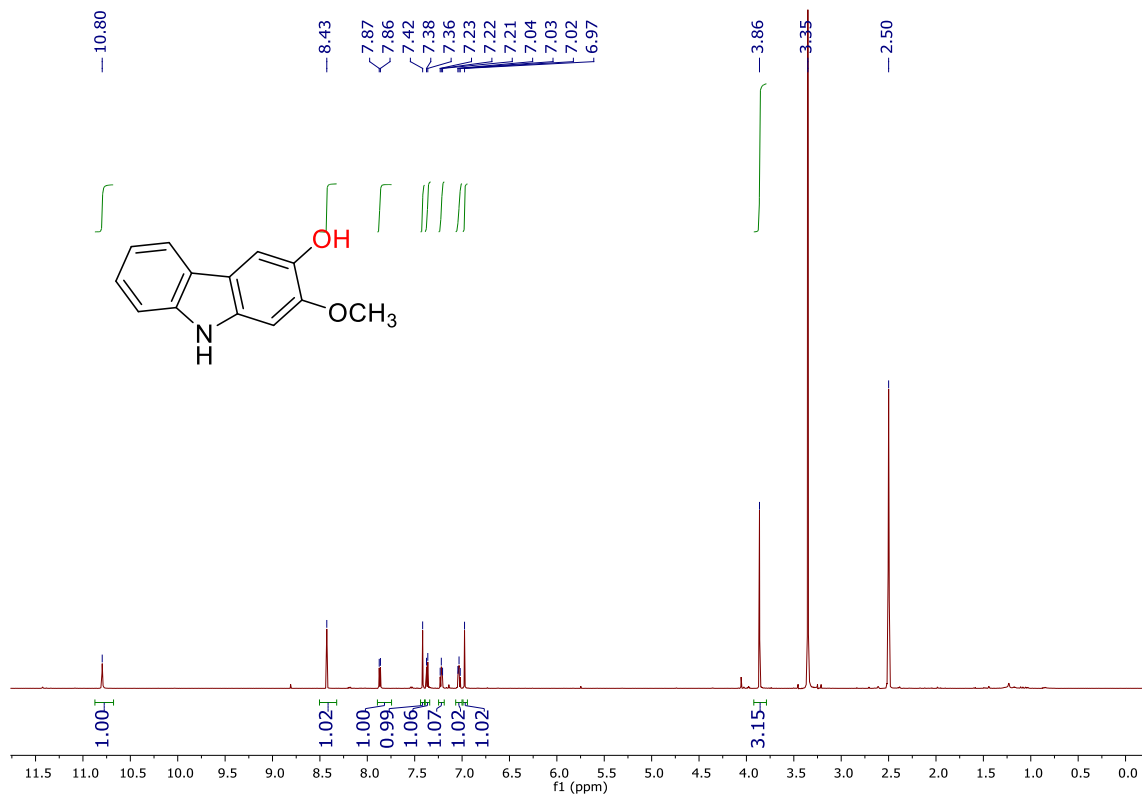
¹H NMR spectrum of **3q**



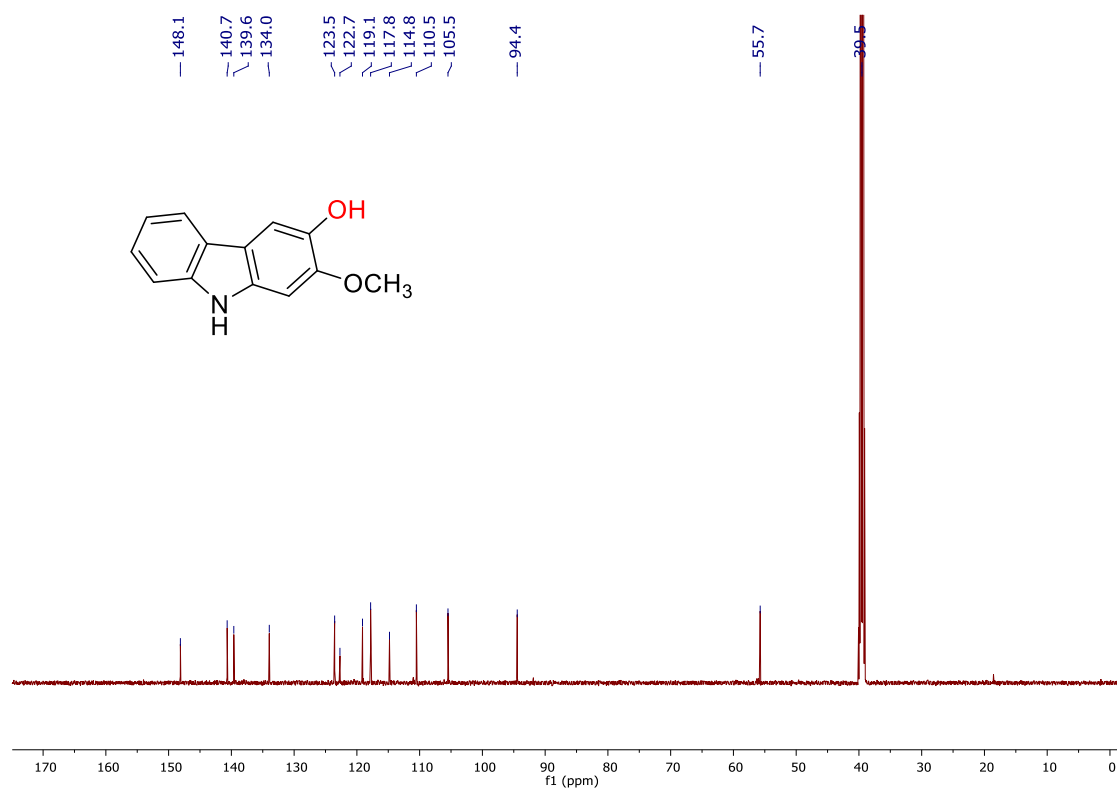
¹³C NMR spectrum of 3q



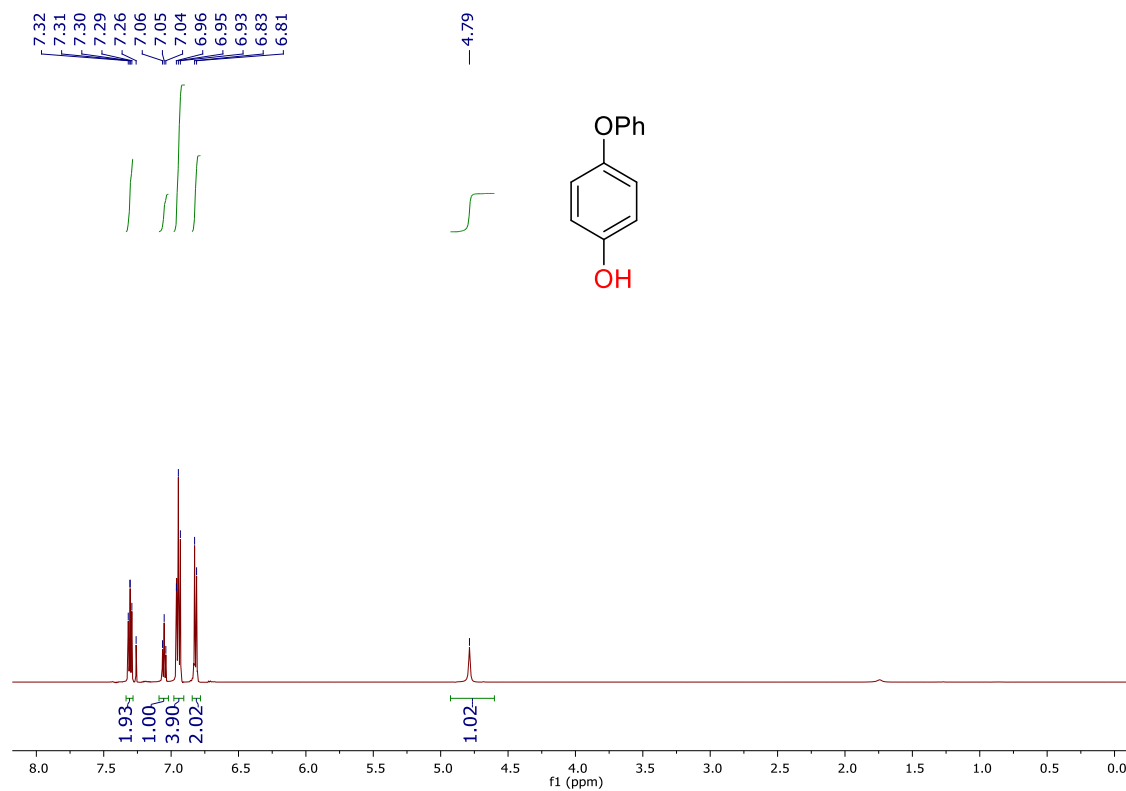
¹H NMR spectrum of 3r



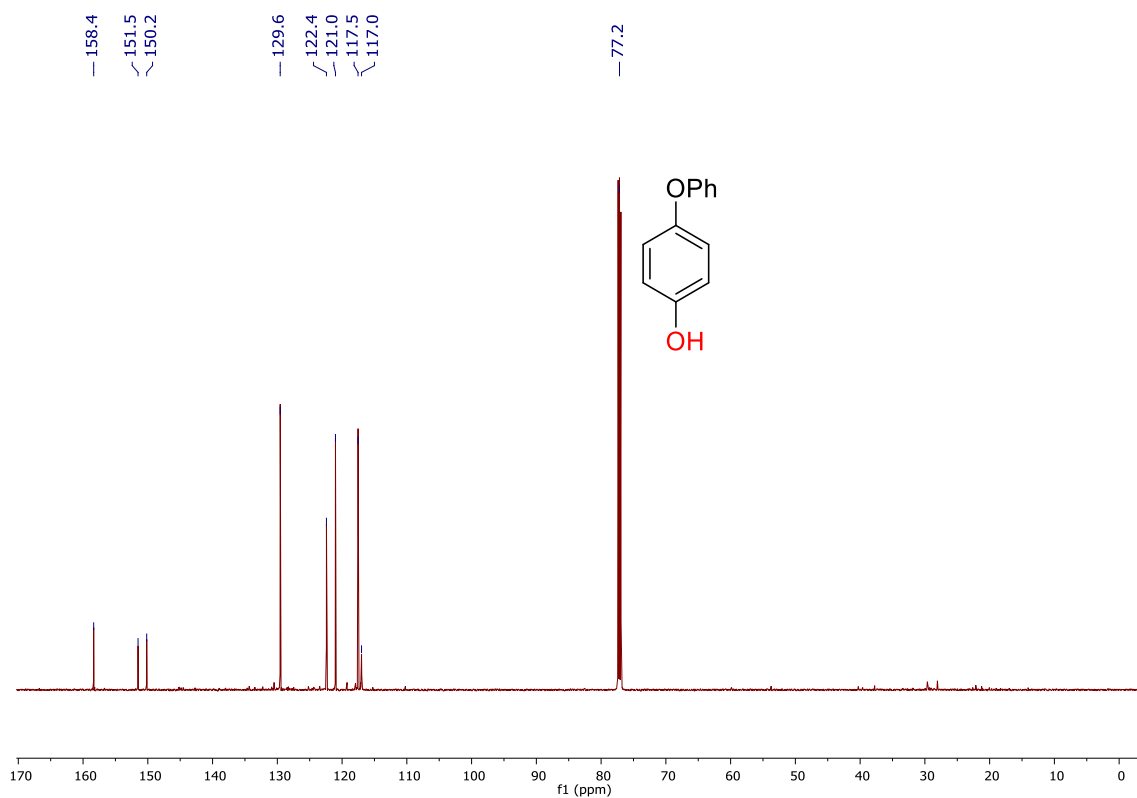
¹³C NMR spectrum of 3r



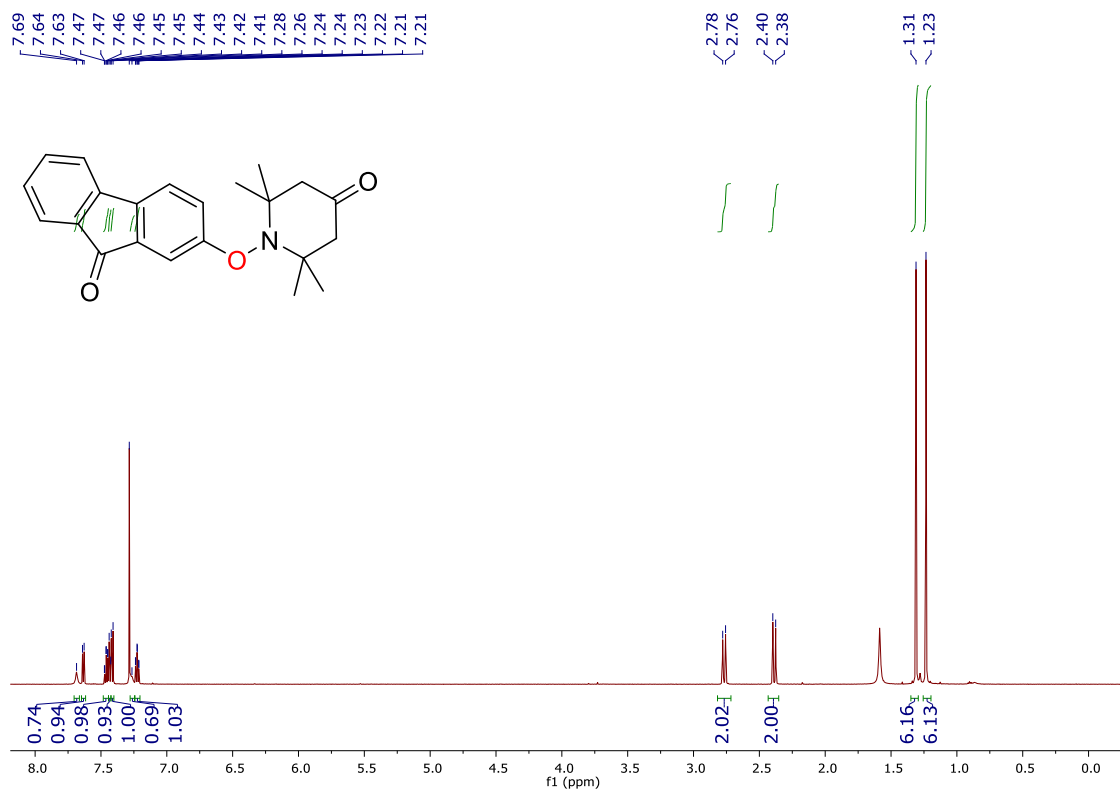
¹H NMR spectrum of 3s



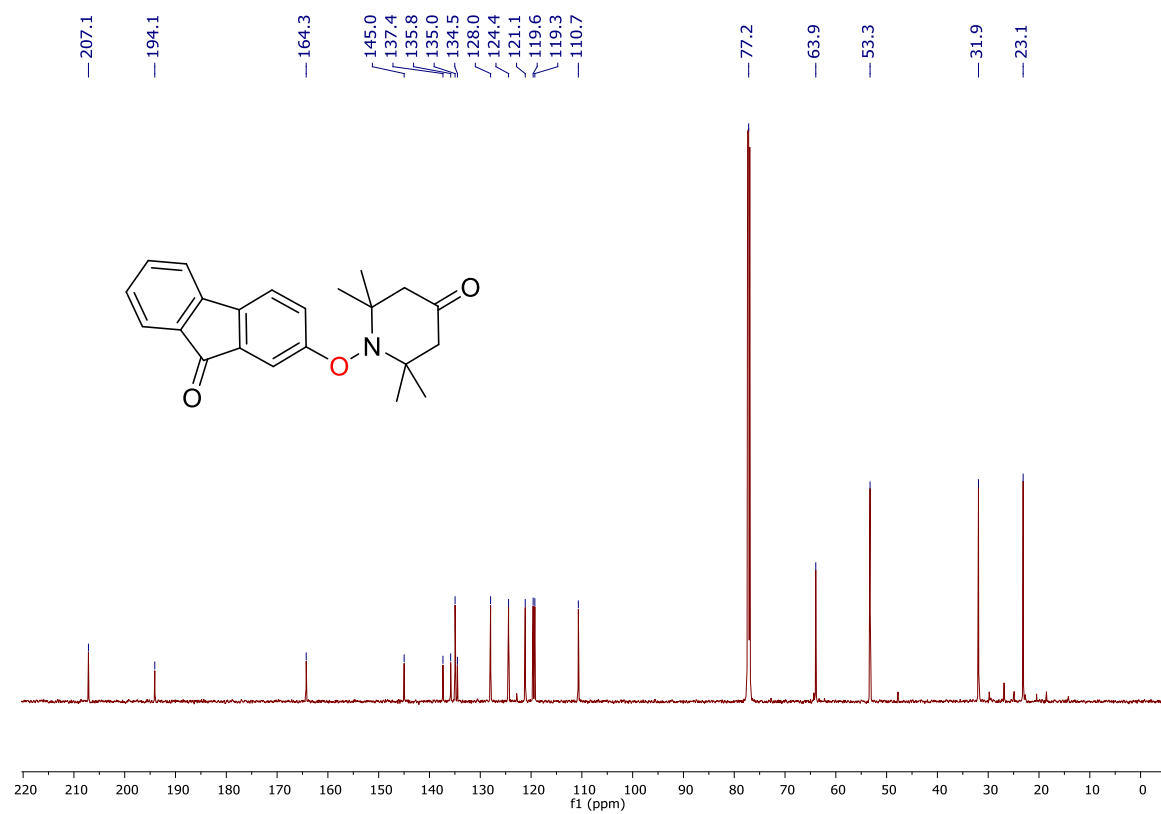
¹³C NMR spectrum of **3s**



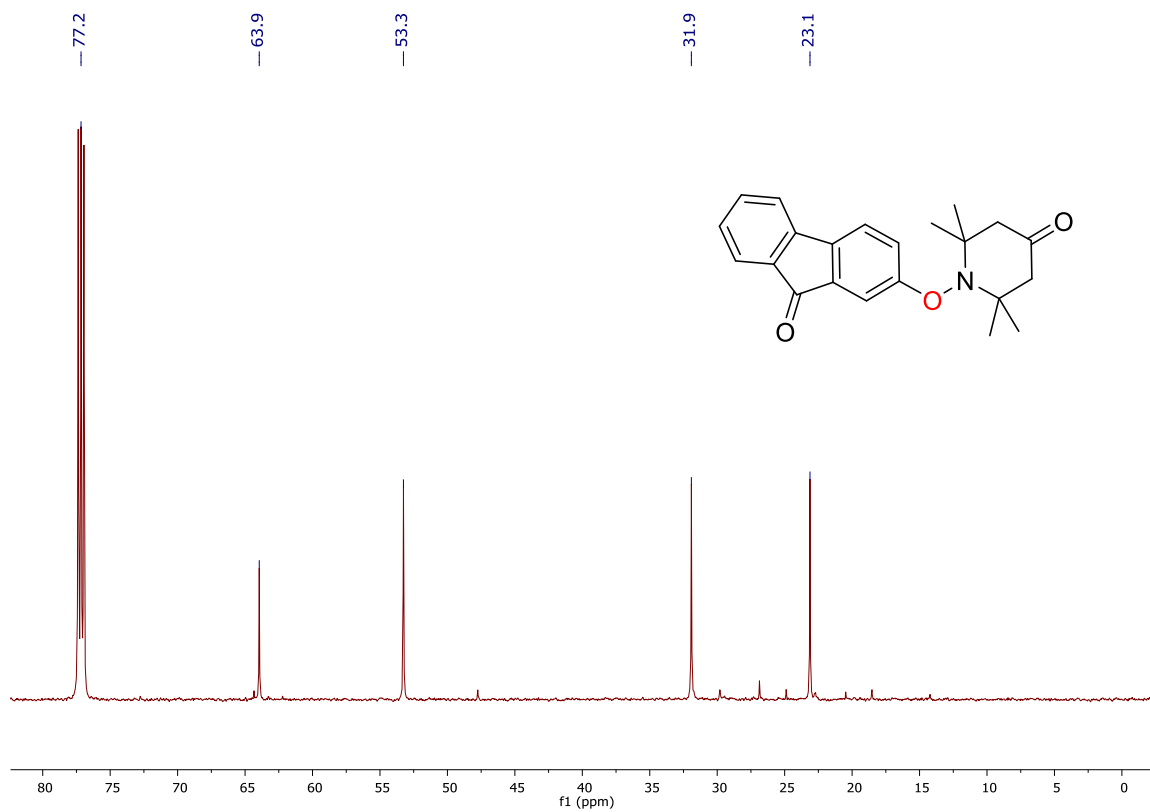
¹H NMR spectrum of **3t**



^{13}C NMR spectrum of **3t**



Zoom in section on the ^{13}C NMR spectrum of **3t**:



Zoom in section on the ^{13}C NMR spectrum of **3t**:

