Supporting Information

Synthesis of fluorinated spiro-1,3-oxazines and thiazines via Selectfluor-mediated intramolecular cyclization

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Experimental

General Methods. Chromatographic separations were performed using silica gel 60-120. Melting points are uncorrected. NMR spectra were recorded in Bruker Avance-III 500 MHz and 400 MHz FTNMR spectrometer using tetramethylsilane (TMS) as an internal standard in the indicated solvent. HRMS data were recorded in a Waters XEVO G2-Xs QTof apparatus.

General Procedure for the preparation of N-(2-(cyclohex-1-en-1-yl) ethyl) benzothioamide (6a-6o):

Benzaldehyde (1 mmol), elemental sulfur (1.2 mmol), amine (1.1 mmol), and sodium sulfide (1.5 mmol) were dissolved in N,N-dimethyl formamide (5 ml). The reaction was allowed to reflux at 100 °C for 1 hour, and then water (5 mL) was added. The reaction was extracted with ethyl acetate (2 x 20 mL) and washed with water (2 x 10 mL), saturated sodium bicarbonate (15 mL), and brine (5 mL). Drying over Na_2SO_4 and removal of the solvent under reduced pressure produced the crude mixture, which was purified by column chromatography.

General procedure for the synthesis of mono-fluorinated spiro-1,3-thiazines (7a-7o):

To a solution of amide (1 mmol) in dichloromethane (10 mL), Selectfluor (1 mmol) was added portion-wise at 0 °C, and the mixture was stirred at 28 °C for 5 h. after completion, as checked by TLC, water (10 mL) was added, and the solution was extracted with chloroform (2 × 20 mL). The combined organic layer was washed with water (2 × 10 mL) and brine (10 mL). The resultant mixture was then dried over Na_2SO_4, and the solvent was removed under reduced pressure. The crude mixture obtained was purified by column chromatography using ethyl acetate: hexane as the eluent.

General Procedure for the preparation of *N*-2-(1-cyclohexenyl) ethyl benzamides (8a-8s):

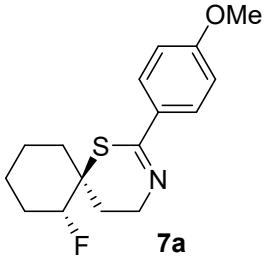
Benzoyl chloride (1 mmol) was dissolved in dry dichloromethane (10 mL). The mixture was cooled in an ice bath, followed by the addition of triethyl amine (2 mmol) and amine (1.2 mmol). The reaction was allowed to stir at room temperature for 2 hours and then water (5 mL) was added. The reaction was extracted with DCM (2 x 10 mL) and washed with saturated sodium bicarbonate (5 mL) and brine (5 mL). Drying over Na_2SO_4 and removal of the solvent under reduced pressure produced the crude mixture, which was purified by column chromatography.

General procedure for the synthesis of mono-fluorinated spiro-1,3-oxazines (9a-9s):

To a cooled solution (-5 °C, generated through ice salt mixture) of amide (1 mmol) in acetonitrile (10 mL), Selectfluor (1 mmol) was added portion-wise. The mixture was then stirred at the same temperature for 1 h. After completion of the reaction, as checked by TLC, acetonitrile was removed under vacuum, and the remaining solution was extracted with ethyl acetate (2 × 20 mL). The combined organic layer was washed with water (2 × 10 mL) and

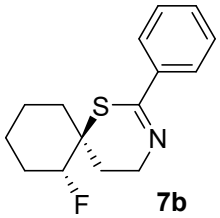
brine (10 mL). The resulting mixture was then dried over Na_2SO_4, and the solvent was removed under reduced pressure. The crude mixture obtained was purified by column chromatography using ethyl acetate: hexane as the eluent.

Spectral Data



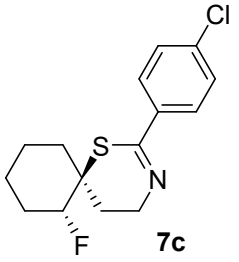
7a

7-fluoro-2-(4-methoxyphenyl)-1-thia-3-azaspiro[5.5]undec-2-ene (**7a**): Colourless semisolid compound; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73 (d, 2H, J = 8.8 Hz), 6.87 (d, 2H, J = 8.8 Hz), 4.71 (dt, 1H, J = 48.4, 3.6 Hz), 3.96-3.92 (m, 2H), 3.81 (s, 3H), 2.02-1.91 (m, 4H), 1.82-1.79 (m, 1H), 1.72-1.66 (m, 1H), 1.63-1.55 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.5 (C), 156.8 (C), 131.9 (C), 128.0 (CH), 113.6 (CH), 92.2 (d, J = 180.1 Hz, CH), 55.5 (CH_3), 48.4 (d, J = 20.5 Hz, C), 45.3 (CH_2), 34.7 (CH_2), 29.2 (CH_2), 26.9 (d, J = 21.3 Hz, CH_2), 20.6 (CH_2), 19.9 (CH_2); HRMS (ESI): m/z 294.1325 ([M+H] $C_{16}H_{20}FNOS$ requires 294.1322)



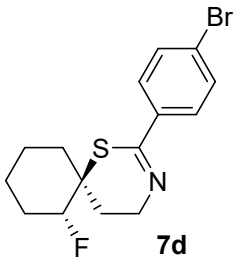
7b

7-fluoro-2-phenyl-1-thia-3-azaspiro[5.5]undec-2-ene (**7b**): Colourless semisolid compound; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79 - 7.76 (m, 2H), 7.40 - 7.34 (m, 3H), 4.71 (dt, 1H, J = 48.8, 2.8 Hz), 3.99 - 3.96 (m, 2H), 2.03 - 1.92 (m, 4H), 1.83 - 1.79 (m, 1H), 1.75 - 1.67(m, 1H), 1.63 - 1.55 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$):δ 157.5 (C), 139.3 (C), 130.5 (CH), 128.4 (CH), 126.6 (CH), 92.2 (d, J = 180.1 Hz, CH), 48.4 (d, J = 20.8 Hz, C), 45.5 (CH_2), 34.7 (CH_2), 29.1 (CH_2), 27.0 (d, J = 21 Hz, CH_2), 20.5 (CH_2), 19.9 (CH_2) HRMS (ESI): m/z 264.1221 ([M+H] $C_{15}H_{18}FNS$ requires 264.1217)



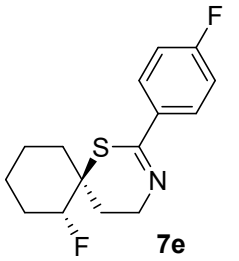
7c

2-(4-chlorophenyl)-7-fluoro-1-thia-3-azaspiro[5.5]undec-2-ene (**7c**): Mp: 65-67 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72 (d, 2H, J = 8.4 Hz), 7.33 (d, 2H, J = 8.4 Hz), 4.70 (dt, 1H, J = 48.8, 4.0 Hz), 3.98 - 3.95 (m, 2H), 2.03 - 1.92 (m, 4H), 1.82 - 1.79 (m, 1H), 1.74 - 1.67(m, 1H), 1.63 - 1.55 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.4 (C), 137.6 (C), 136.6 (C), 128.6 (CH), 127.9 (CH), 92.1 (d, J = 180.7 Hz, CH), 48.6 (d, J = 19.0 Hz, C), 45.4 (CH_2), 34.7 (CH_2), 28.9 (CH_2), 26.9 (d, J = 21.3 Hz, CH_2), 20.5 (CH_2), 19.8 (CH_2) HRMS (ESI): m/z 298.0830 ([M+H] $C_{15}H_{17}ClFNS$ requires 298.0827)



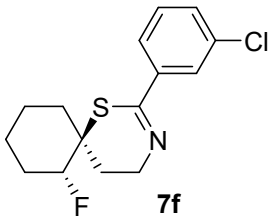
7d

2-(4-bromophenyl)-7-fluoro-1-thia-3-azaspiro[5.5]undec-2-ene (**7d**): Mp: 78-80 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.65 (d, 2H, J = 8.8 Hz), 7.49 (d, 2H, J = 8.8 Hz), 4.69 (dt, 1H, J = 48.8, 2.8 Hz), 3.97-3.94 (m, 2H), 2.04 - 1.51 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.6 (C), 137.7 (C), 131.6 (CH), 128.2 (CH), 125.2 (C), 92.0 (CH, d, J = 180.2 Hz), 48.8 (C, d, J = 20.9 Hz), 45.2 (CH_2), 34.7 (CH_2), 28.8 (CH_2), 26.9 (CH_2, d, J = 21.1 Hz), 20.5 (CH_2), 19.8 (CH_2); HRMS (ESI): m/z 342.0323 ([M+H] $C_{15}H_{17}BrFNS$ requires 342.0323).



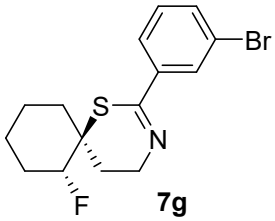
7e

7-fluoro-2-(4-fluorophenyl)-1-thia-3-azaspiro[5.5]undec-2-ene (**7e**): Colourless semisolid compound; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79 - 7.75 (m, 2H), 7.06 - 7.02 (m, 2H), 4.70 (dt, 1H, J = 48.8, 2.8 Hz), 3.97 - 3.94 (m, 2H), 2.03 - 1.97 (m, 2H), 1.95 - 1.90 (m, 2H), 1.83 - 1.79 (m, 1H), 1.74-1.67 (m, 2H) 1.63-1.60 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.6 (C), 163.1 (C), 156.3 (C), 128.6 (CH, d, J = 10 Hz), 115.3 (CH, d, J = 21 Hz), 92.1 (CH, d, J = 181.7 Hz), 48.6 (C, d, J = 20.8 Hz), 45.4 (CH_2), 34.7 (CH_2), 29.0 (CH_2), 26.9 (CH_2, d, J = 22 Hz), 20.5 (CH_2), 19.9 (CH_2) HRMS (ESI): m/z 282.1126 ([M+H] $C_{15}H_{17}F_2NS$ requires 282.1123).



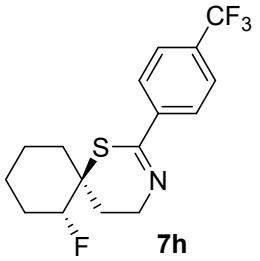
7f

2-(3-chlorophenyl)-7-fluoro-1-thia-3-azaspiro[5.5]undec-2-ene (**7f**): Colourless semisolid compound; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78 (t, 1H, J = 2.0 Hz), 7.65 (dt, 1H, J = 8.0, 2.0 Hz), 7.39 - 7.36 (m, 1H), 7.29 (t, 1H, J = 8.0 Hz), 4.69 (dt, 1H, J = 48.4, 3.0 Hz), 4.00 - 3.96 (m, 2H), 2.04 - 1.54 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.2 (C), 140.9 (C), 134.5 (C), 130.5 (CH), 129.6 (CH), 126.8 (CH), 124.7 (CH), 92.1 (CH, d, J = 180.4 Hz), 48.7 (C, d, J = 21.0 Hz), 45.5 (CH_2), 34.7 (CH_2), 28.9 (CH_2, d, J = 3.8 Hz), 26.9 (CH_2, d, J = 21.2 Hz), 20.5 (CH_2), 19.8 (CH_2) HRMS (ESI): m/z 298.0830 ([M+H] $C_{15}H_{17}ClFNS$ requires 298.0827)



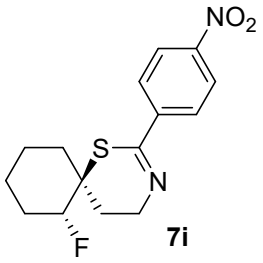
7g

2-(3-bromophenyl)-7-fluoro-1-thia-3-azaspiro[5.5]undec-2-ene (**7g**): Colourless semisolid compound; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 (t, 1H, J = 1.6 Hz), 7.70 (dt, 1H, J = 8.0, 1.6 Hz) 7.54 - 7.51 (m, 1H), 7.23 (t, 1H, J = 8.0 Hz), 4.69 (dt, 1H, J = 48.8, 3.6 Hz), 3.99 - 3.96 (m, 2H), 2.04 - 1.90 (m, 4H), 1.82 - 1.78 (m, 1H), 1.74 - 1.67 (m, 1H), 1.63-1.60 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.9 (C), 141.1 (C), 133.4 (CH), 129.9 (CH), 129.6 (CH), 125.2 (CH), 122.6 (C), 92.1 (CH, d, J = 180.2 Hz), 48.7 (C, d, J = 23.1 Hz), 45.5 (CH_2), 34.7 (CH_2), 28.9 (CH_2, d, J = 3.8 Hz), 26.9 (CH_2, d, J = 21.2 Hz), 20.5 (CH_2), 19.8 (CH_2) HRMS (ESI): m/z 342.0322 ([M+H] $C_{15}H_{17}BrFNS$ requires 342.0322)



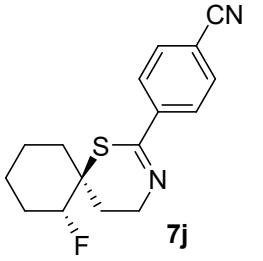
7h

7-fluoro-2-(4-(trifluoromethyl)phenyl)-1-thia-3-azaspiro[5.5]undec-2-ene (**7h**): Mp: 72-74 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, 2H, J = 8.0 Hz), 7.63 (d, 2H, J = 8.0 Hz), 4.72 (dt, 1H, J = 48.8, 2.8 Hz), 4.04 - 3.96 (m, 2H), 2.08 - 1.52 (m, 10H);^{13}C NMR (125 MHz, $CDCl_3$): δ 156.2 (C), 142.3 (C), 132.2 (CF_3, q, J = 32.4 Hz), 128.7 (C), 127.0 (CH), 125.4 (CH, q, J = 4.0 Hz), 92.1 (CH, d, J = 180.6 Hz), 48.7 (C, d, J = 21.0 Hz), 45.6 (CH_2), 34.7 (CH_2), 28.8 (CH_2, d, J = 3.8 Hz), 26.9 (CH, d, J = 21.1 Hz), 20.5 (CH_2), 19.8 (CH_2); HRMS (ESI): m/z 332.1093 ([M+H] $C_{16}H_{17}F_4NS$ requires 332.1091)



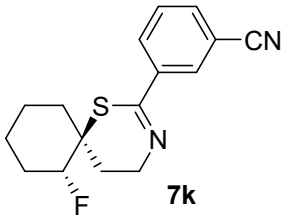
7i

7-fluoro-2-(4-nitrophenyl)-1-thia-3-azaspiro[5.5]undec-2-ene (**7i**): Mp: 90-92 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.23 (d, 2H, J = 9.0 Hz), 7.95 (d, 2H, J = 9.0 Hz), 4.71 (dt, 1H, J = 48.0, 3.0 Hz), 4.05 - 4.03 (m, 2H), 2.01 -1.92 (m, 3H), 1.85 - 1.81 (m, 1H), 1.78 - 1.72 (m, 1H), 1.66 - 1.58 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 155.4 (C), 148.9 (C), 144.4 (C), 127.4 (CH), 123.4 (CH), 91.8 (CH, d, J = 178.5 Hz), 48.8 (C, d, J = 22.1 Hz), 45.6 (CH_2), 34.5 (CH_2), 29.6 (CH_2), 26.8 (CH_2, d, J = 21.1 Hz), 20.3 (CH_2), 19.7 (CH_2) HRMS (ESI): *m*/*z* 309.1071 ([M+H] $C_{15}H_{17}FN_2O_2S$ requires 309.1068)



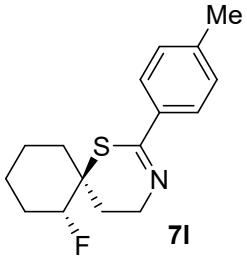
7j

4-(7-fluoro-1-thia-3-azaspiro[5.5]undec-2-en-2-yl)benzonitrile (**7j**): Mp: 105-107 °C ; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89 (d, J = 2H, 8.4 Hz), 7.66 (d, 2H, J = 8.4 Hz,), 4.68 (dt, 1H, J = 48.8, 4.0 Hz), 4.02-3.99 (m, 2H), 2.06-1.93 (m, 4H), 1.83-1.78 (m, 1H), 1.74-1.68 (m, 1H), 1.63-1.57 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 155.8 (C), 142.7 (C), 132.0 (CH), 127.0 (CH), 118.3 (C), 113.7 (C), 91.8 (CH, d, J = 180.9 Hz), 48.7 (C, d, J = 20.8 Hz), 45.5 (CH_2), 34.5 (CH_2), 28.5(CH_2), 26.7 (CH_2, d, J = 21.1 Hz), 20.3 (CH_2), 19.7 (CH_2); HRMS (ESI): *m*/*z* 289.1172 ([M+H] $C_{16}H_{17}FN_2S$ requires 289.1169).



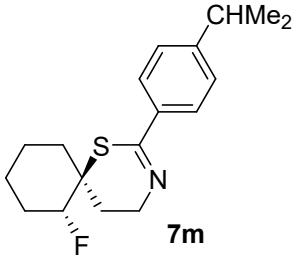
7k

3-(7-fluoro-1-thia-3-azaspiro[5.5]undec-2-en-2-yl)benzonitrile (**7k**): Colourless semisolid compound; ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.11 (t, 1H, J = 1.5 Hz), 8.03 (dt, 1H, J = 8.0, 1.5 Hz), 7.70 (dt, 1H, J = 8.0, 1.5 Hz), 7.50 (t, 1H, J = 8.0 Hz), 4.71 (dt, 1H, J = 49.5, 2.5 Hz), 4.02-4.00 (m, 2H), 2.07-1.94 (m, 4H), 1.84-1.81 (m, 1H), 1.77-1.71 (m, 1H), 1.64-1.57 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 155.2 (C), 140.0 (C), 133.5 (CH), 130.6 (CH), 130.2 (CH), 129.1 (CH), 118.3 (C), 112.5 (C), 91.8 (CH, d, J = 181.2 Hz), 48.7 (C, d, J = 20.9 Hz), 45.4 (CH_2), 34.5 (CH_2), 28.5 (CH_2, d, J = 20.9 Hz), 26.7 (CH_2, d, J = 21.1 Hz), 20.3 (CH_2), 19.7 (CH_2); HRMS (ESI): *m*/*z* 289.1172 ([M+H] $C_{16}H_{17}FN_2S$ requires 289.1169).



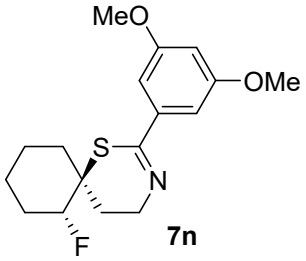
7l

7-fluoro-2-(p-tolyl)-1-thia-3-azaspiro[5.5]undec-2-ene (**7l**): Colourless semisolid compound; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.61 (d, 2H, J = 6.5 Hz), 7.11 (d, 2H, J = 6.5 Hz), 4.65 (dt, 1H, J = 49.0, 2.5 Hz), 3.93-3.87 (m, 2H), 2.30 (s, 3H), 2.00-1.45 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 157.3 (C), 140.7 (C), 136.5 (C), 129.1 (CH), 126.4 (CH), 92.2 (CH, d, J = 180.2 Hz), 48.4 (C, d, J = 21.0 Hz), 45.3 (CH_2), 34.7 (CH_2), 29.1 (CH_2, d, J = 3.8 Hz), 26.9 (CH_2, d, J = 21.1 Hz), 21.4 (CH_3), 20.5 (CH_2), 19.9 (CH_2); HRMS (ESI): *m/z* 278.1375 ([M+H] $C_{16}H_{20}FNS$ requires 278.1373)



7m

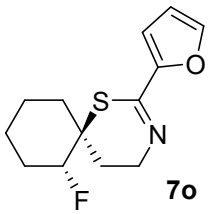
7-fluoro-2-(4-isopropylphenyl)-1-thia-3-azaspiro[5.5]undec-2-ene (**7m**): Colourless semisolid compound; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.60 (d, 2H, J = 8.5 Hz), 7.17 (d, 2H, J = 8.5 Hz), 4.67 (dt, 1H, J = 48.5, 3.5 Hz), 3.92-3.90 (m, 2H), 2.86 (septet, 1H, J = 7.0 Hz), 1.97-1.88 (m, 4H), 1.79-1.75 (m, 1H), 1.69-1.63 (m, 1H), 1.58-1.56 (m, 4 H), 1.19 (d, J = 7 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 157.0 (C), 154.4 (C), 136.7 (C), 126.3 (CH), 126.3 (CH), 92.0 (CH, d, J = 180.0 Hz), 48.1 (C, d, J = 20.8 Hz), 45.2 (CH_2), 34.5 (CH_2), 33.9 (CH_2), 29.0 (CH), 26.7 (CH_2, d, J = 21.5 Hz), 23.7 (CH_3), 20.4 (CH_2), 19.7 (CH_2); HRMS (ESI): *m/z* 306.1688 ([M+H] $C_{18}H_{24}FNS$ requires 306.1686)



7n

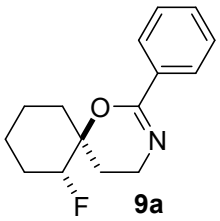
2-(3,5-dimethoxyphenyl)-7-fluoro-1-thia-3-azaspiro[5.5]undec-2-ene (**7n**): Colourless semisolid compound; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.93 (d, 2H, J = 2.4 Hz), 6.50 (t, 1H, J = 2.4 Hz), 4.70 (dt, 1H, J = 48.8, 3.6 Hz), 3.97-3.94 (m, 2H), 3.80 (s, 6H), 2.03-1.92 (m, 4H), 1.82-1.78 (m, 1H), 1.73-1.66 (m, 1H), 1.61-1.53 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.7 (C), 157.4 (C), 141.3 (C), 104.5 (CH), 103.0 (CH), 92.1 (CH, d, J = 180.1 Hz), 55.6 (CH_3), 48.5 (C, d, J = 20.6 Hz), 45.4 (CH_2), 34.7 (CH_2), 29.1 (CH_2, d, J = 3.8 Hz), 26.9 (CH_2,

d, J = 21.3 Hz), 20.5 (CH_2), 19.8 (CH_2); HRMS (ESI): m/z 324.1430 ([M+H] $C_{17}H_{22}FNO_2S$ requires 324.1428).



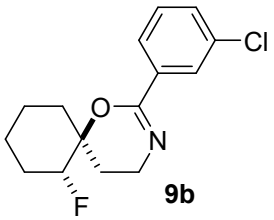
7o

7-fluoro-2-(furan-2-yl)-1-thia-3-azaspiro[5.5]undec-2-ene (**7o**): Reddish brown semisolid compound; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 (d, 1H, J = 1.2 Hz), 6.82 (dd, 1H, J = 3.6, 1.2 Hz), 6.42 (dd, 1H, J = 3.6, 2.0 Hz), 4.68 (d, 1H, J = 48.4, 2.4 Hz), 3.98-3.93 (m, 2H), 2.05-1.96 (m, 2H), 1.93-1.88 (m, 2H), 1.82-1.68 (m, 2H), 1.61-1.56 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.2 (C), 148.0 (C), 144.2 (CH), 111.4 (CH), 110.8 (CH), 91.9 (CH, d, J = 180.3 Hz), 48.1 (C, d, J = 21.1 Hz), 44.9 (CH_2), 34.6 (CH_2), 29.4 (CH_2), 26.9 (CH_2, d, J = 21.6 Hz), 20.5 (CH_2), 19.8 (CH_2); HRMS (ESI): m/z 254.1012 ([M+H] $C_{13}H_{16}FNOS$ requires 254.1009).



9a

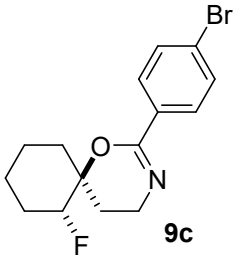
7-Fluoro-2-phenyl-1-oxa-3-azaspiro[5.5]undec-2-ene (**9a**): Mp: 125-127 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.94 (d, 2H, J = 8.5 Hz), 7.46-7.35 (m, 3H), 4.59 (dt, 1H, J = 48.5, 4.5 Hz), 3.74-3.55 (m, 2H), 2.07-1.50 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.4 (C), 133.9 (C), 130.3 (CH), 128.0 (CH), 126.8 (CH), 90.8 (CH, d, J = 175.4 Hz), 74.9 (C, d, J = 23.7 Hz), 39.6 (CH_2), 31.9 (CH_2), 27.1 (CH_2, d, J = 20.0 Hz), 26.9 (CH_2), 20.3 (CH_2), 20.0 (CH_2); HRMS (ESI): m/z 248.1454 ([M+H] $C_{15}H_{19}FNO$ requires 248.1451).



9b

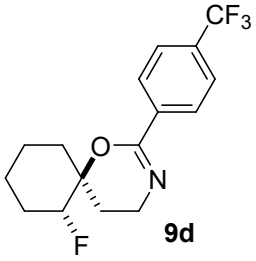
2-(3-Chlorophenyl)-7-fluoro-1-oxa-3-azaspiro[5.5]undec-2-ene (**9b**): Mp: 85-87 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.87 (s, 1H), 7.78 (d, 1H, J = 8.0 Hz), 7.38 (d, 1H, J = 8.0 Hz), 7.29 (t, 1H, J = 8.0 Hz), 4.55 (d, 1H, J = 48.0 Hz), 3.70-3.52 (m, 2H), 2.00-1.50 (m, 10H); ^{13}C NMR

(125 MHz, $CDCl_3$): δ 153.7 (C), 136.0 (C), 134.4 (C), 130.6 (CH), 129.5 (CH), 127.3 (CH), 125.2 (CH), 91.2 (CH, d, J = 176.2 Hz), 75.8 (C, d, J = 23.7 Hz), 40.0 (CH_2), 32.3 (CH_2), 27.5 (CH_2, d, J = 20.0 Hz), 26.8 (CH_2), 20.7 (CH_2), 20.4 (CH_2); HRMS (ESI): m/z 282.1059 ([M+H] $C_{15}H_{18}ClFNO$ requires 282.1061).



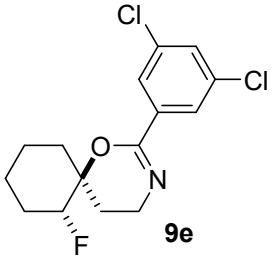
9c

2-(4-Bromophenyl)-7-fluoro-1-oxa-3-azaspiro[5.5]undec-2-ene (**9c**): Mp: 136-138 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.77 (d, 2H, J = 8.5 Hz), 7.51 (d, 2H, J = 8.5 Hz), 4.56 (dt, 1H, J = 48.5, 4.0 Hz), 3.70-3.54 (m, 2H), 2.05-1.52 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.8 (C), 132.8 (C), 131.2 (CH), 128.4 (CH), 124.9 (C), 90.9 (CH, d, J = 176.2 Hz), 75.4 (C, d, J = 23.7 Hz), 39.6 (CH_2), 32.0 (CH_2), 27.2 (CH_2, d, J = 20.0 Hz), 26.6 (CH_2), 20.4 (CH_2), 20.0 (CH_2); HRMS (ESI) m/z 326.0558 ([M+H] $C_{15}H_{18}BrFNO$ requires 326.0556).



9d

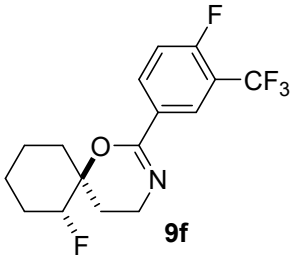
7-Fluoro-2-(4-(trifluoromethyl)phenyl)-1-oxa-3-azaspiro[5.5]undec-2-ene (**9d**): Mp: 150-152 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.00 (d, 2H, J = 8.5 Hz), 7.61 (d, 2H, J = 8.5 Hz), 4.55 (dt, 1H, J = 49.0, 4.0 Hz), 3.70-3.55 (m, 2H), 2.10-1.50 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.7 (C), 137.5 (C), 132.3 (CF_3, q, J = 32.5 Hz), 127.4 (CH), 125.3 (CH), 123.1 (C), 91.2 (CH, d, J = 176.2 Hz), 75.9 (C, d, J = 23.7 Hz), 40.0 (CH_2), 32.3 (CH_2), 27.5 (CH_2, d, J = 20.0 Hz), 26.8 (CH_2), 20.7 (CH_2), 20.4 (CH_2); HRMS (ESI): m/z 316.1324 ([M+H] $C_{16}H_{18}F_4NO$ requires 316.1325).



9e

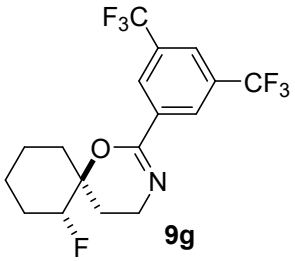
2-(3,5-Dichlorophenyl)-7-fluoro-1-oxa-3-azaspiro[5.5]undec-2-ene (**9e**): Mp: 88-90 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.74 (s, 2H), 7.37 (s, 1H), 4.52 (dt, 1H, J = 48.0, 4.0 Hz), 3.68-3.51 (m, 2H), 2.03-1.45 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.6 (C), 137.1 (C), 135.0 (C), 130.4 (CH), 125.6 (CH), 91.3 (CH, d, J = 176.2 Hz), 76.3 (C, d, J = 23.7 Hz), 40.0

(CH_2), 32.3 (CH_2), 27.5 (CH_2, d, J = 18.7 Hz), 26.4 (CH_2), 20.7 (CH_2), 20.5 (CH_2); HRMS (ESI): m/z 316.0669 ([M+H] $C_{15}H_{17}Cl_2FNO$ requires 316.0671).



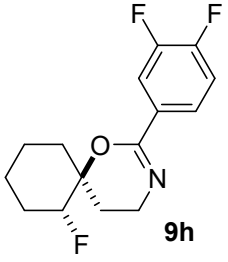
9f

7-Fluoro-2-(4-fluoro-3-(trifluoromethyl)phenyl)-1-oxa-3-azaspiro[5.5]undec-2-ene (**9f**): Mp: 155-157 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.16 (dd, 1H, J = 7.0, 1.5 Hz), 8.09-8.05 (m, 1H), 7.17 (t, 1H, J = 9.0 Hz), 4.55 (dt, 1H, J = 48.0, 4.0 Hz), 3.69-3.51 (m, 2H), 2.05-1.50 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 161.2 (C, d, J = 256.5 Hz), 152.9 (C), 132.6 (CH, d, J = 9.0 Hz), 130.6 (C), 126.3 (CH), 121.5 (C), 118.4 (CF_3, q, J = 20.0 Hz),116.8 (CH, d, J = 21.0 Hz), 91.4 (CH, d, J = 176.2 Hz), 76.3 (C, d, J = 22.5 Hz), 39.9 (CH_2), 32.4 (CH_2), 27.5 (CH_2, d, J = 18.7 Hz), 26.4 (CH_2), 20.8 (CH_2), 20.5 (CH_2); HRMS (ESI): m/z 334.1226 ([M+H] $C_{16}H_{17}F_5NO$ requires 334.1230).



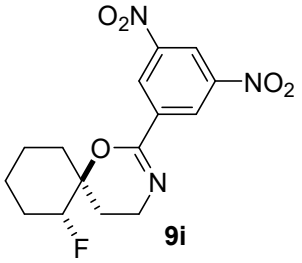
9g

2-(3,5-bis(Trifluoromethyl)phenyl)-7-fluoro-1-oxa-3-azaspiro[5.5]undec-2-ene (**9g**): Mp: 148-150 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.34 (s, 2H), 7.90 (s, 1H), 4.58 (dt, 1H, J = 49.0, 4.5 Hz), 3.70-3.55 (m, 2H), 2.10-1.45 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.5 (C), 136.3 (C), 131.7(CF_3, q, J = 33.7 Hz), 127.3 (CH), 124.5 (C), 124.0 (CH), 91.6 (CH, d, J = 177.5 Hz), 76.9 (C, d, J = 23.7 Hz), 40.1 (CH_2), 32.6 (CH_2), 27.7 (CH_2, d, J = 18.7 Hz), 26.1 (CH_2), 20.9 (CH_2), 20.7 (CH_2); HRMS (ESI): m/z 384.1195 ([M+H] $C_{17}H_{17}F_7NO$ requires 384.1198).



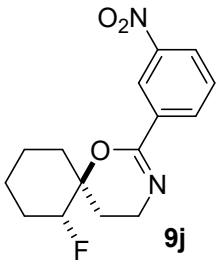
9h

2-(3,4-Difluorophenyl)-7-fluoro-1-oxa-3-azaspiro[5.5]undec-2-ene (**9h**): Mp: 145-147 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.72-7.62 (m, 2H), 7.16-7.05 (m, 1H), 4.53 (dt, 1H, J = 48.0, 4.0 Hz), 3.66-3.51 (m, 2H), 2.01-1.50 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.1 (C, d, J = 13.7 Hz), 150.2 (C, d, J = 246.2 Hz), 150.1 (C, d, J = 246.2 Hz), 131.2 (C, d, J = 10.0 Hz), 123.5 (CH, d, J = 11.2 Hz), 117.1 (CH, d, J = 17.5 Hz), 116.4 (CH, d, J = 18.7 Hz), 91.2 (CH, d, J = 176.2 Hz), 76.0 (C, d, J = 23.7 Hz), 39.9 (CH_2), 32.3 (CH_2), 27.5 (CH_2, d, J = 20.0 Hz), 26.7 (CH_2), 20.7 (CH_2), 20.4 (CH_2); HRMS (ESI): m/z 284.1261 ([M+H] $C_{15}H_{17}F_3NO$ requires 284.1262).



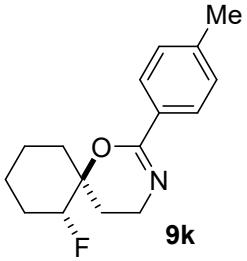
9i

2-(3,5-Dinitrophenyl)-7-fluoro-1-oxa-3-azaspiro[5.5]undec-2-ene (**9i**): Mp: 140-142 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.06 (s, 1H), 9.03 (s, 2H), 4.62 (dq, 1H, J = 48.0, 3.0 Hz), 3.78-3.57 (m, 2H), 2.10-1.45 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.2 (C), 148.6 (C), 138.0 (C), 127.1 (CH), 120.2 (CH), 91.8 (CH, d, J = 177.5 Hz), 77.8 (C, d, J = 23.7 Hz), 40.3 (CH_2), 32.7 (CH_2), 27.8 (CH_2, d, J = 18.7 Hz), 25.7 (CH_2), 21.0 (CH_2), 20.9 (CH_2); HRMS (ESI): m/z 338.1155 ([M+H] $C_{15}H_{17}FN_3O_5$ requires 338.1152).



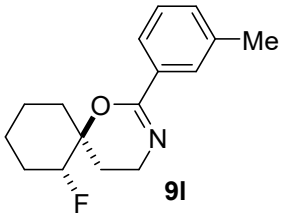
9j

7-Fluoro-2-(3-nitrophenyl)-1-oxa-3-azaspiro[5.5]undec-2-ene (**9j**): Mp: 101-103 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.75 (s, 1H), 8.35-8.25 (m, 2H), 7.59 (t, 1H, J = 8.0 Hz), 4.62 (dt, 1H, J = 48.0, 4.5 Hz), 3.76-3.62 (m, 2H), 2.10-1.55 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.9 (C), 148.4 (C), 136.0 (C), 133.0 (CH), 129.3 (CH), 125.2 (CH), 122.2 (CH), 91.5 (CH, d, J = 176.2 Hz), 76.5 (C, d, J = 22.5 Hz), 40.1 (CH_2), 32.5 (CH_2), 27.6 (CH_2, d, J = 20.0 Hz), 26.5 (CH_2), 20.8 (CH_2), 20.6 (CH_2); HRMS (ESI): m/z 293.1301 ([M+H] $C_{15}H_{18}FN_2O_3$ requires 293.1301).



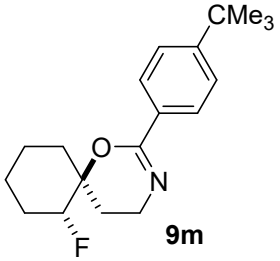
9k

7-Fluoro-2-(p-tolyl)-1-oxa-3-azaspiro[5.5]undec-2-ene (**9k**): Mp: 130-132 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.82 (d, 2H, J = 8.5 Hz), 7.20 (d, 2H, J = 8.5 Hz), 4.58 (dt, 1H, J = 48.0, 3.5 Hz), 3.70-3.56 (m, 2H), 2.40 (s, 3H), 2.05-1.52 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.8 (C), 140.8 (C), 131.4 (C), 129.0 (CH), 127.0 (CH), 91.1 (CH, d, J = 175.0 Hz), 75.1 (C, d, J = 24.2 Hz), 39.9 (CH_2), 32.2 (CH_2), 27.4 (CH_2, d, J = 20.0 Hz), 27.3 (CH_2), 21.6 (CH_3), 20.6 (CH_2), 20.3 (CH_2); HRMS (ESI): m/z 262.1608 ([M+H] $C_{16}H_{21}FNO$ requires 262.1607).



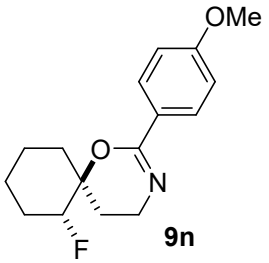
9l

7-Fluoro-2-(m-tolyl)-1-oxa-3-azaspiro[5.5]undec-2-ene (**9l**): Mp: 125-127 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.74 (s, 1H), 7.69 (d, 1H, J = 7.0 Hz), 7.30-7.21 (m, 2H), 4.55 (dt, 1H, J = 49.0, 4.0 Hz), 3.70-3.52 (m, 2H), 2.38 (s, 3H), 2.00-1.50 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 155.2 (C), 138.0 (C), 133.9 (C), 131.5 (CH), 128.2 (CH), 127.7 (CH), 124.2 (CH), 91.2 (CH, d, J = 176.2 Hz), 75.5 (C, d, J = 23.7 Hz), 39.9 (CH_2), 32.3 (CH_2), 27.5 (CH_2, d, J = 20.0 Hz), 27.1 (CH_2), 21.6 (CH_3), 20.7 (CH_2), 20.3 (CH_2); HRMS (ESI) m/z 262.1604 ([M+H] $C_{16}H_{21}FNO$ requires 262.1607).



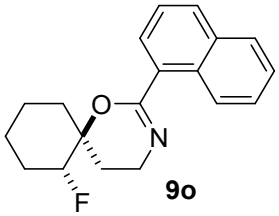
9m

2-(4-(tert-Butyl)phenyl)-7-fluoro-1-oxa-3-azaspiro[5.5]undec-2-ene (**9m**): Mp: 122-124 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.77 (d, 2H, J = 7.0 Hz), 7.33 (d, 2H, J = 7.0 Hz), 4.49 (dt, 1H, J = 48.0, 3.5 Hz), 3.64-3.47 (m, 2H), 1.95-1.45 (m, 10H), 1.26 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.5 (C), 153.7 (C), 131.0 (C), 126.5 (CH), 124.9 (CH), 90.8 (CH, d, J = 176.2 Hz), 74.8 (C, d, J = 23.7 Hz), 39.5 (CH_2), 34.7 (C), 31.9 (CH_2), 31.1 (CH_3) 27.2 (CH_2, d, J = 20.0 Hz), 27.0 (CH_2), 20.3 (CH_2), 19.9 (CH_2); HRMS (ESI): m/z 304.2079 ([M+H] $C_{19}H_{27}FNO$ requires 304.2077).



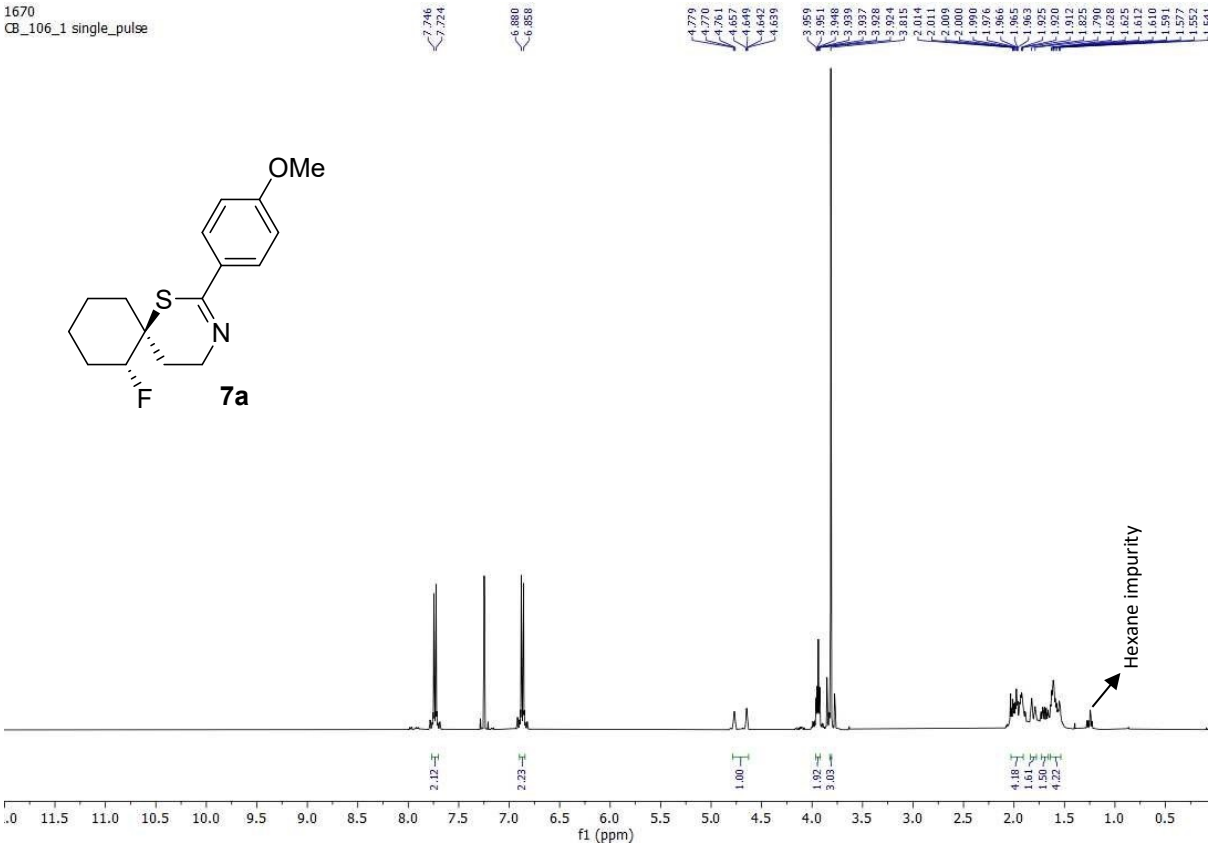
9n

7-Fluoro-2-(4-methoxyphenyl)-1-oxa-3-azaspiro[5.5]undec-2-ene (**9n**): Mp: 108-110 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.84 (d, 2H, J = 9.0 Hz), 6.86 (d, 2H, J = 9.0 Hz), 4.55 (dt, 1H, J = 48.0, 4.0Hz), 3.81 (s, 3H), 3.65-3.51 (m, 2H), 2.00-1.50 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 161.4 (C), 154.4 (C), 128.4 (CH), 126.3 (C), 113.3 (CH), 90.8 (CH, d, J = 173.7 Hz), 74.9 (C, d, J = 24.1 Hz), 55.2 (OCH_3), 39.4 (CH_2), 31.9 (CH_2), 27.1 (CH_2, d, J = 20.0 Hz), 26.9 (CH_2), 20.3 (CH_2), 20.0 (CH_2); HRMS (ESI): m/z 278.1554 ([M+H] $C_{16}H_{21}FNO_2$ requires 278.1556).



9o

7-Fluoro-2-(naphthalen-2-yl)-1-oxa-3-azaspiro[5.5]undec-2-ene (**9o**): Mp: 88-90 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.37 (s, 1H), 8.00 (d, 1H, J = 8.5 Hz), 7.89 (d, 1H, J = 8.5 Hz), 7.85-7.80 (m, 2H),7.52-7.46 (m, 2H), 4.62 (dt, 1H, J = 48.0, 4.0 Hz), 3.75-3.59 (m, 2H), 2.10-1.50 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 155.0 (C), 134.6 (C), 133.0 (C), 131.5 (C), 129.1 (CH), 128.0 (CH), 127.9 (CH), 127.2 (CH), 127.1 (CH), 126.4 (CH), 124.3 (CH), 91.3 (CH, d, J = 175.4 Hz), 75.6 (C, d, J = 23.9 Hz), 40.1 (CH_2), 32.4 (CH_2), 27.5 (CH_2, d, J = 20.0 Hz), 27.1 (CH_2), 20.8 (CH_2), 20.4 (CH_2); HRMS (ESI): m/z 298.1608 ([M+H] $C_{19}H_{21}FNO$ requires 298.1607).


1670
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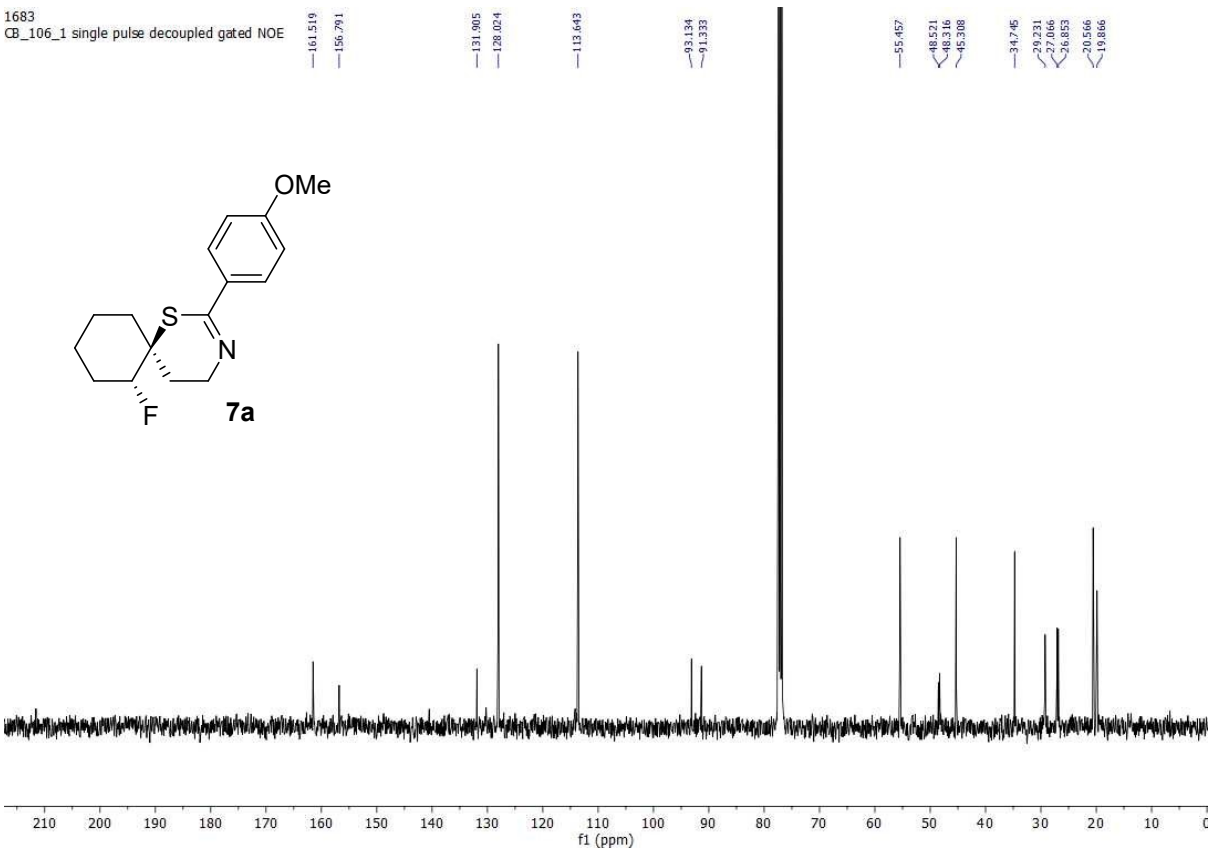
OMe
S
N
F
7a
Hexane impurity
f1 (ppm)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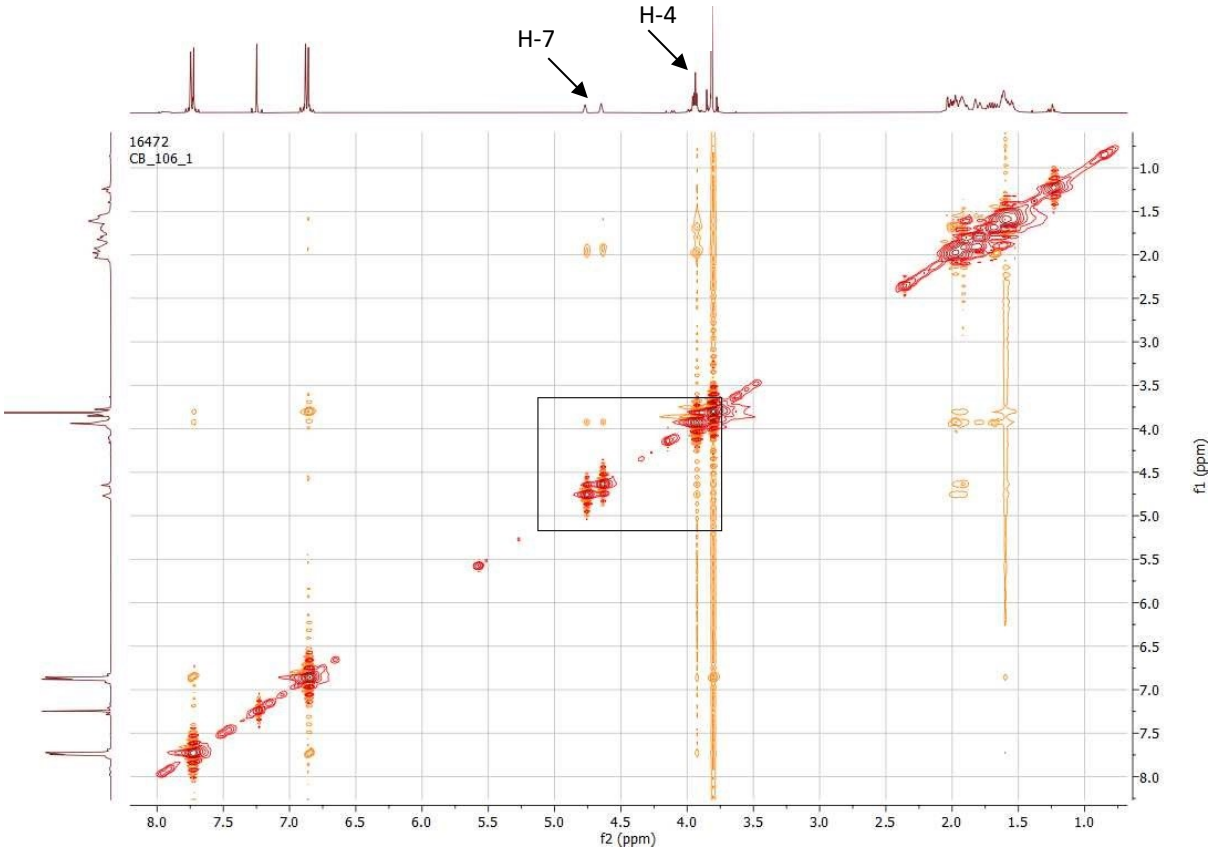
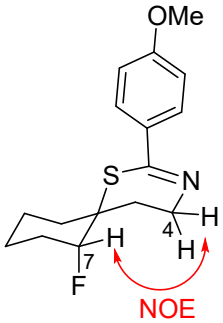
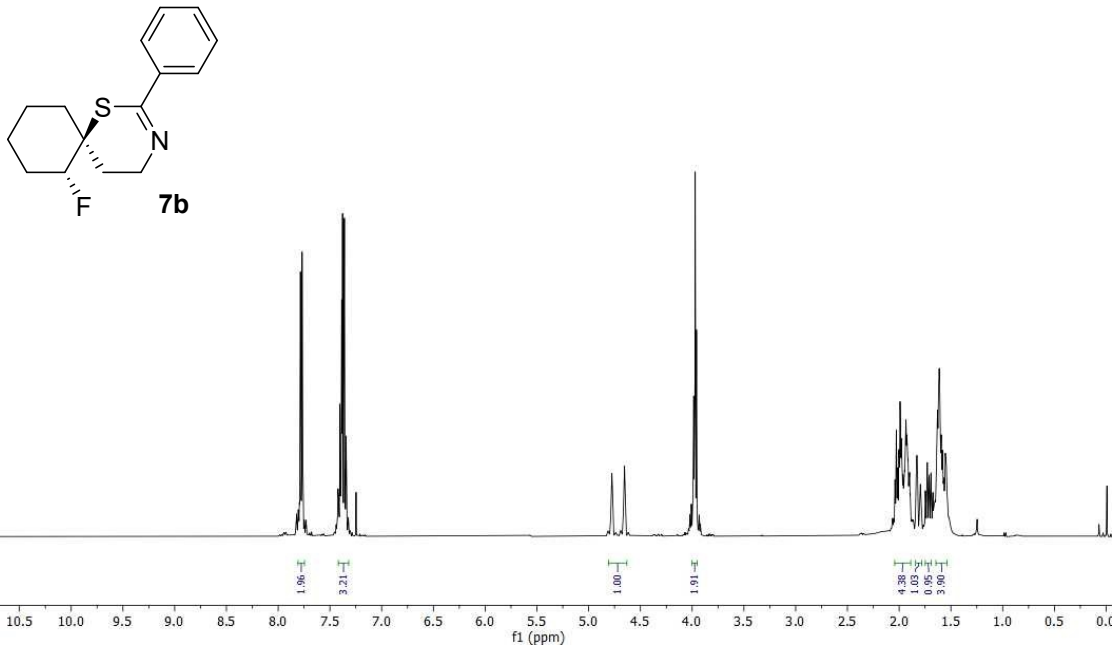
CB_106_1 single pulse decoupled gated NOE
OMe
S
N
F
7a
f1 (ppm)

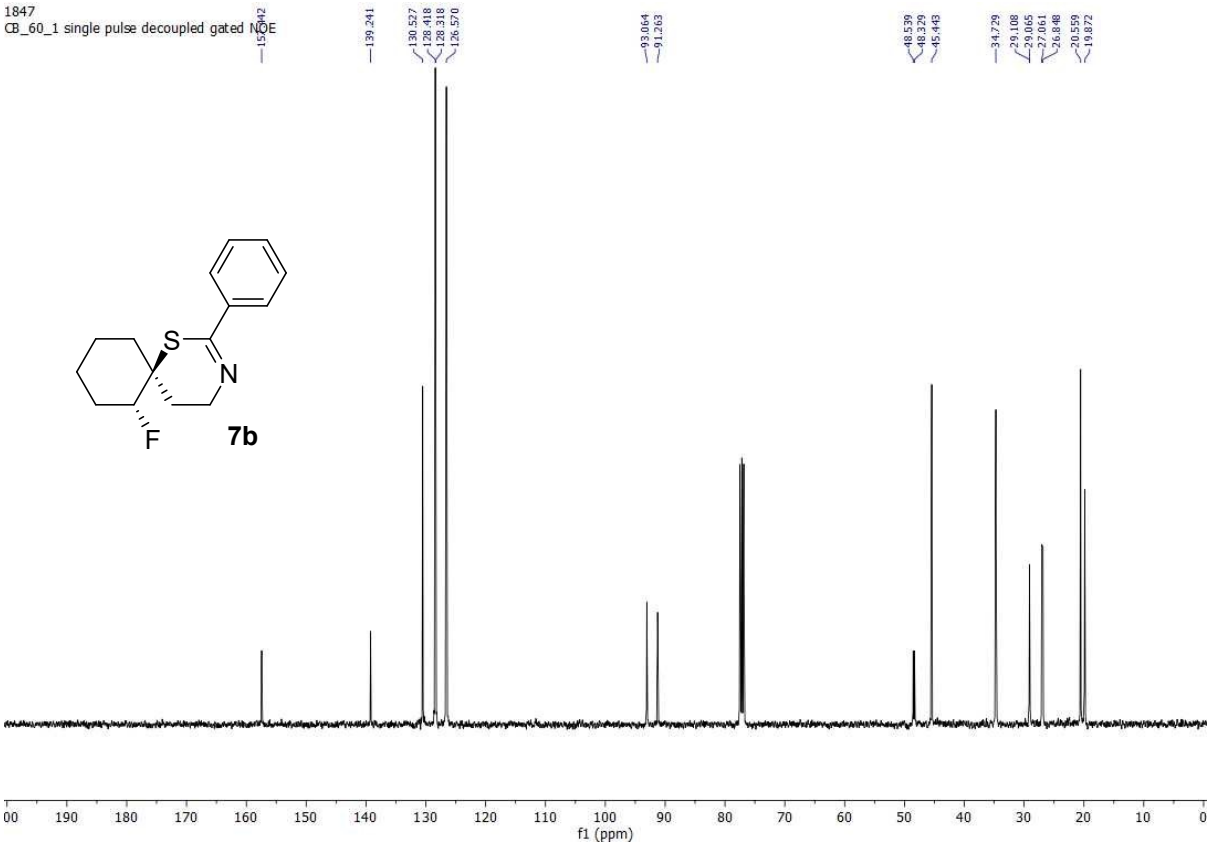

H-7
H-4
16472
CB_106_1
f2 (ppm)
f1 (ppm)

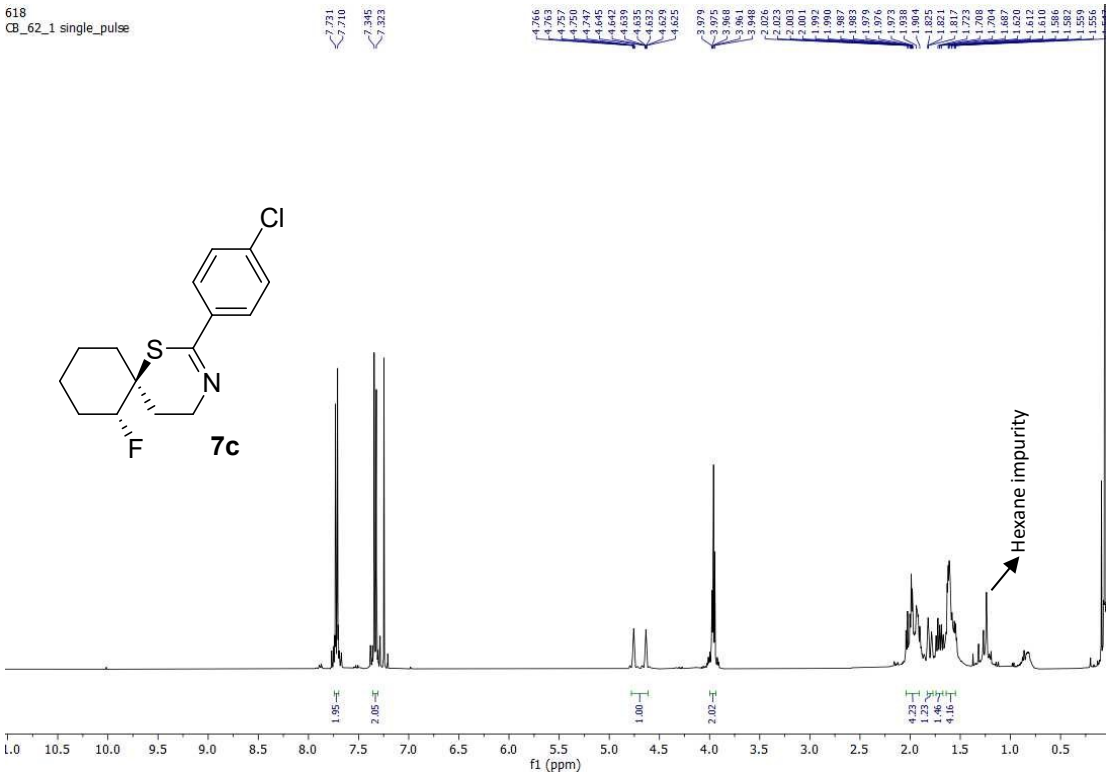

OMe
S
N
H
4
H
H
7
F
NOE

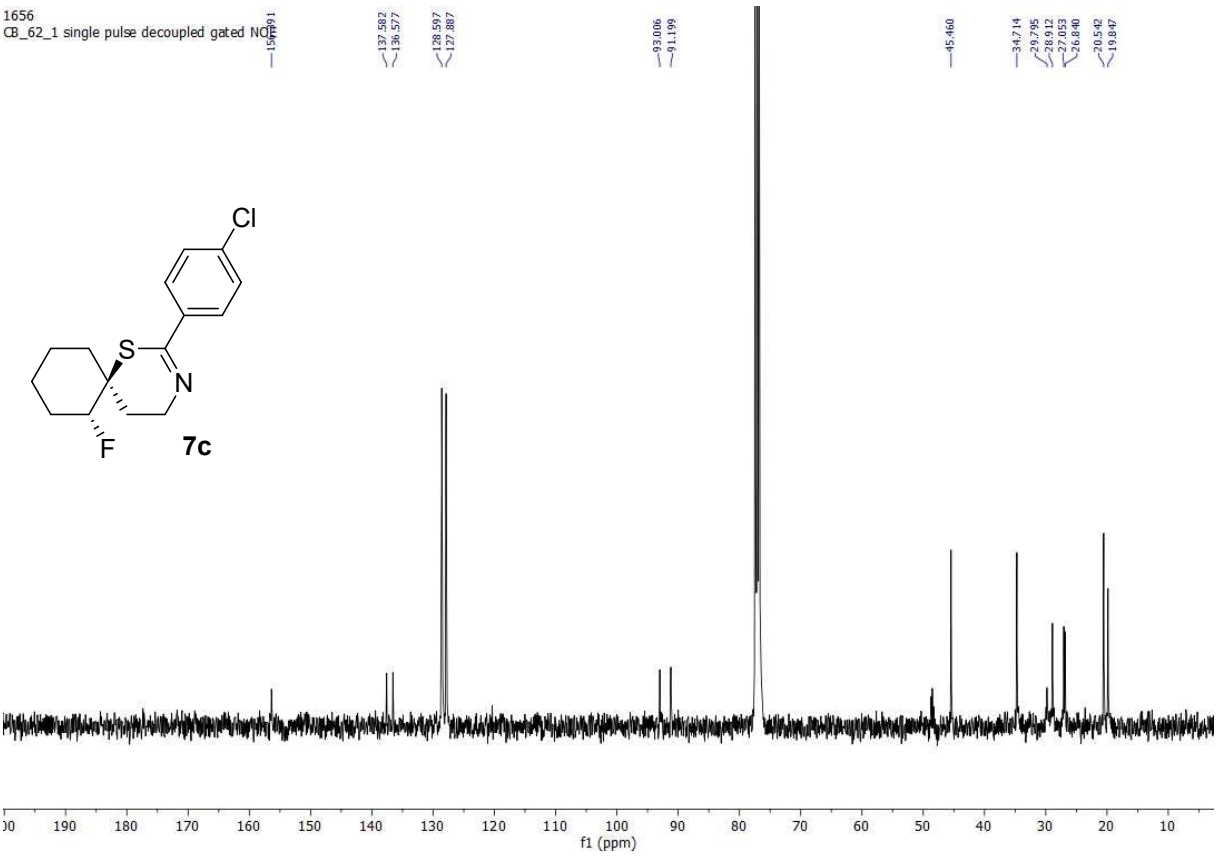
1846
CB_60_1 single_pulse

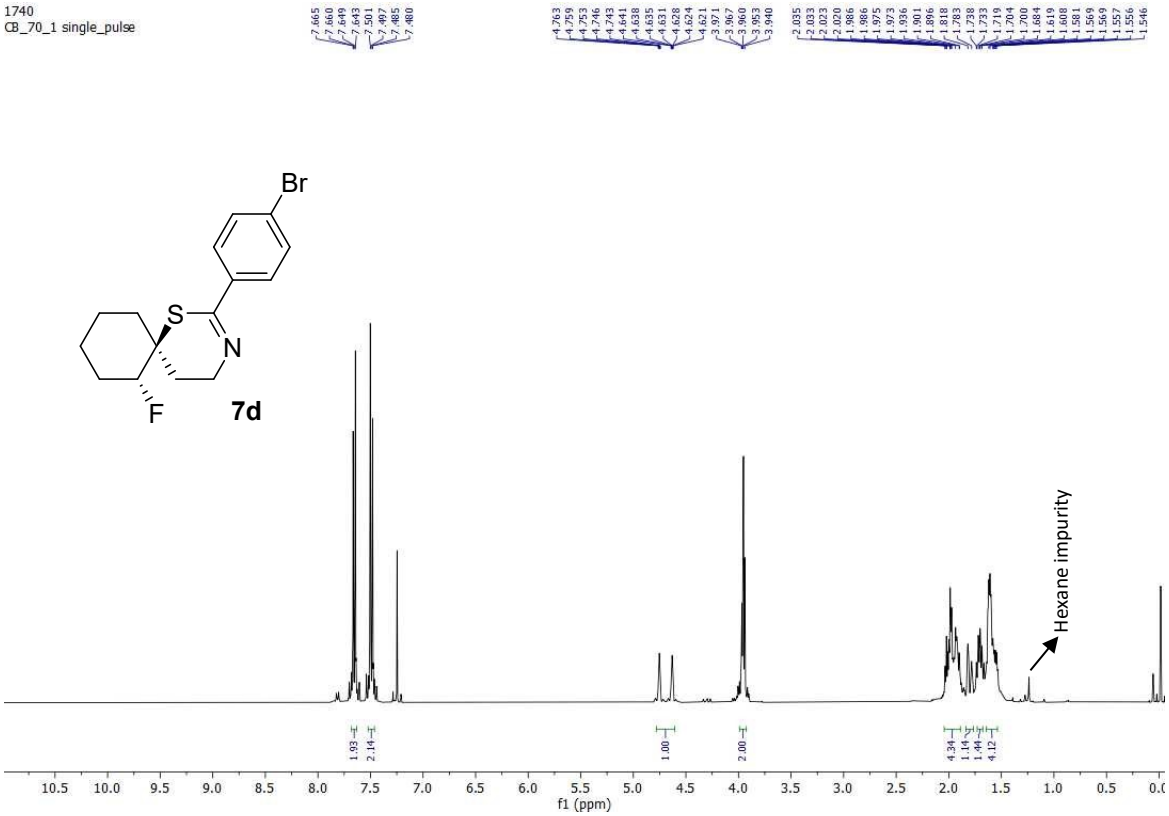


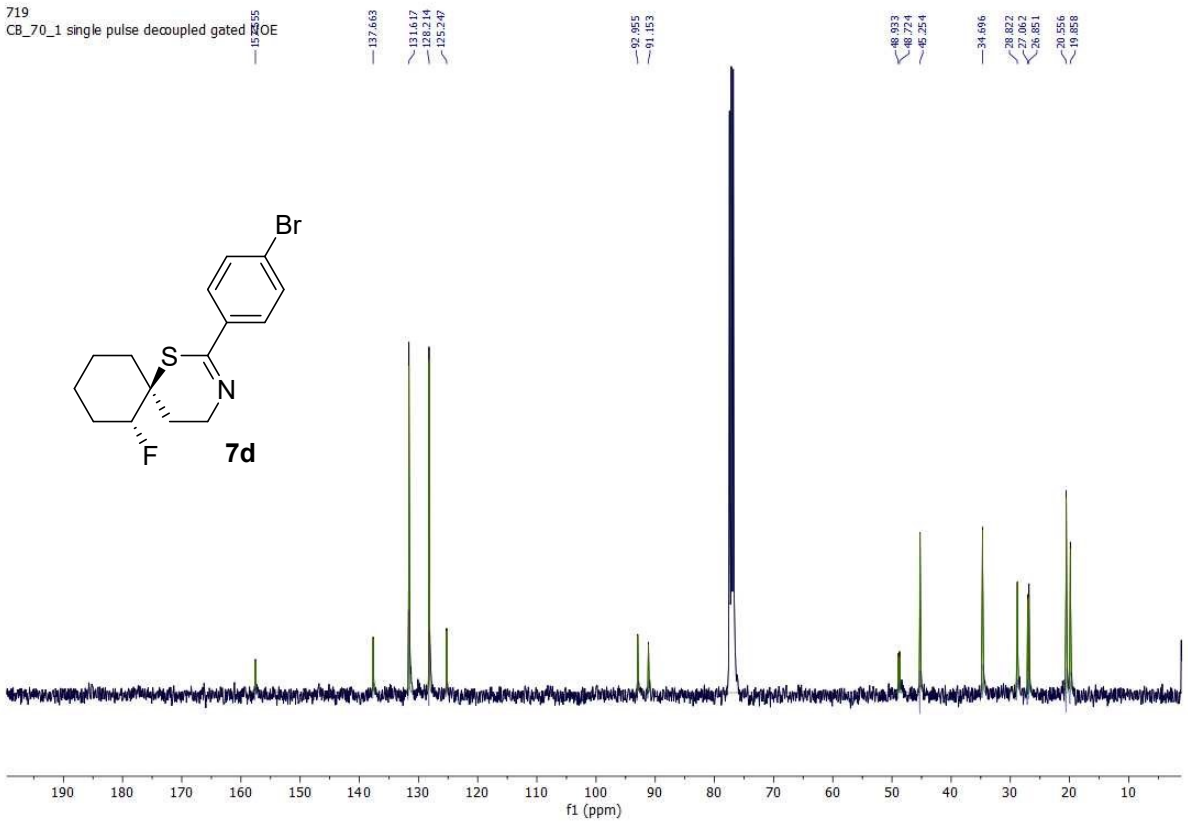
1847
CB_60_1 single pulse decoupled gated NOE

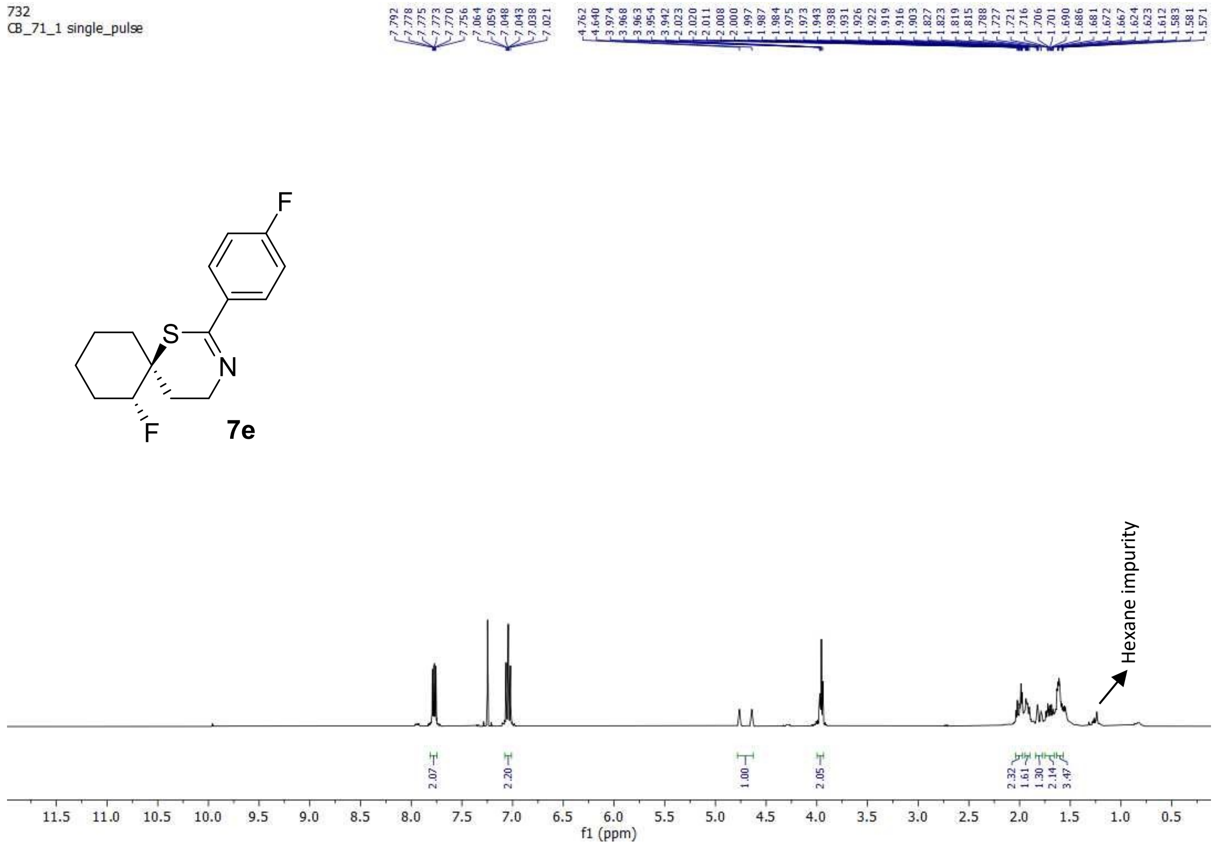



618
CB_62_1 single_pulse
Cl
S
N
F
7c
Hexane impurity
f1 (ppm)

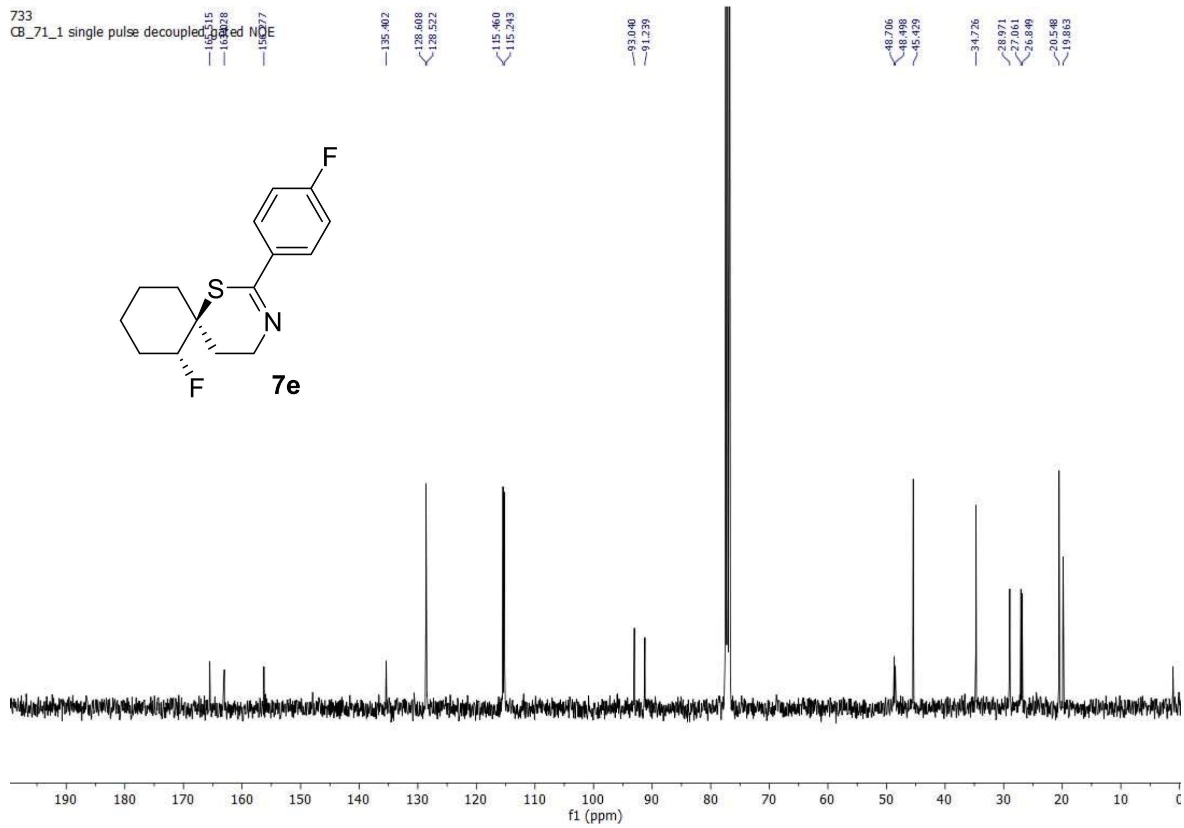

1656
CB_62_1 single pulse decoupled gated NOE
Cl
S
N
F
7c
f1 (ppm)


1740
CB_70_1 single_pulse
Br
S
N
F
7d
Hexane impurity
f1 (ppm)


719
CB_70_1 single pulse decoupled gated NOE
Br
S
N
F
7d
f1 (ppm)

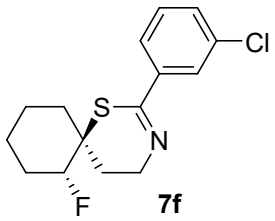


732
CB_71_1 single_pulse
7e
Hexane impurity
2.07
2.20
1.00
2.05
2.32
1.61
1.30
2.14
3.47
f1 (ppm)

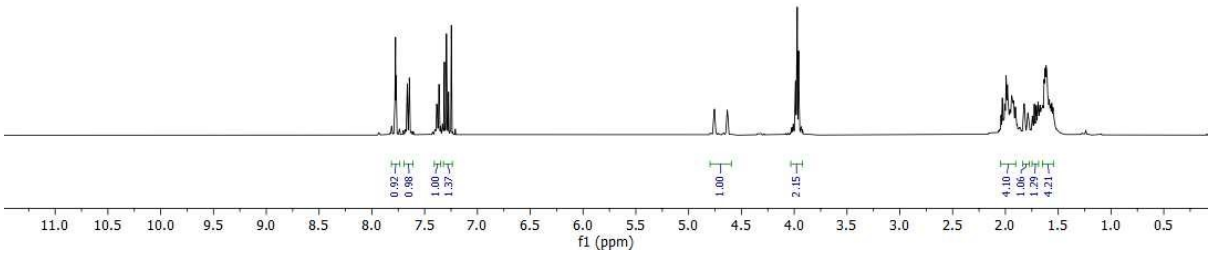


733
CB_71_1 single pulse decoupled gated NOE
135.402
128.608
128.522
115.460
115.243
93.040
91.239
48.706
48.498
45.429
34.726
28.971
27.061
26.849
20.548
19.863
7e
f1 (ppm)

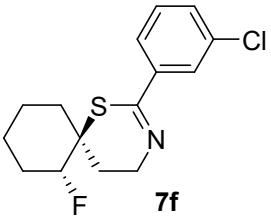
1741
CB_72_1 single_pulse



7f

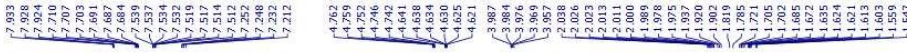


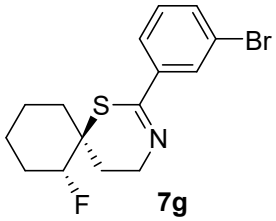
1764
CB_72_1 single pulse decoupled gated NOE



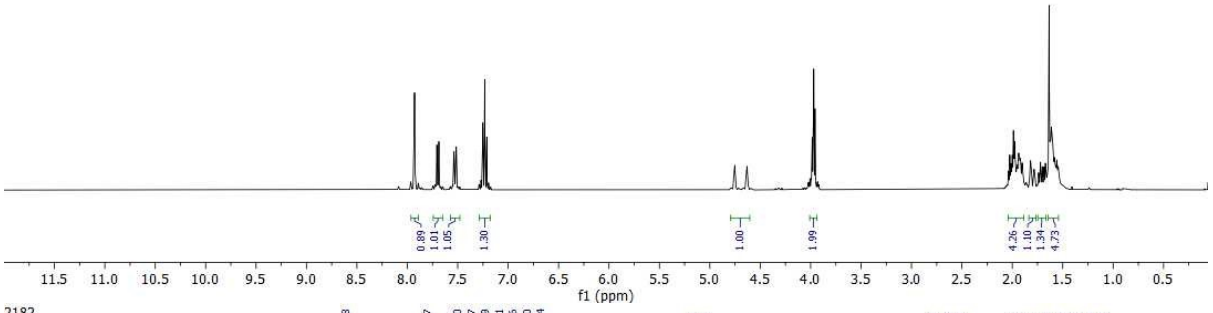
7f

2028
CB_101_1 single_pulse

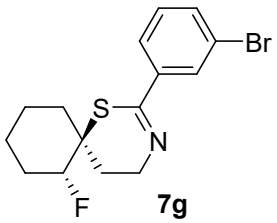




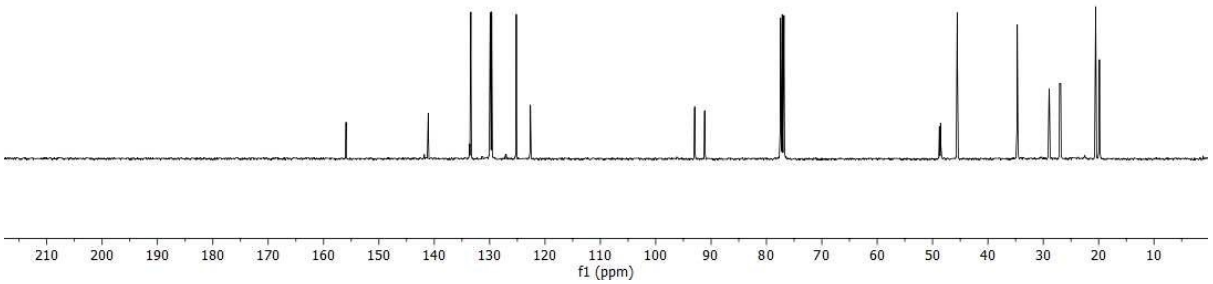
7g

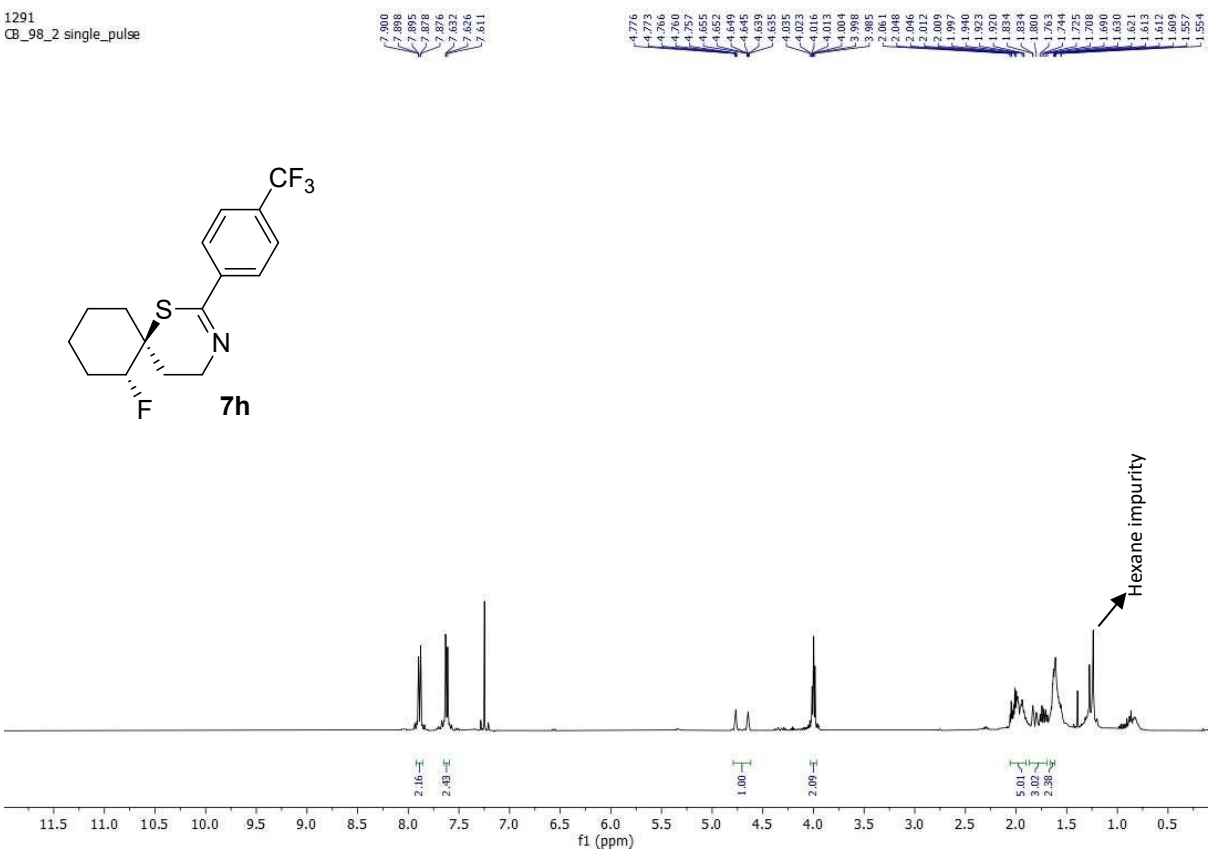


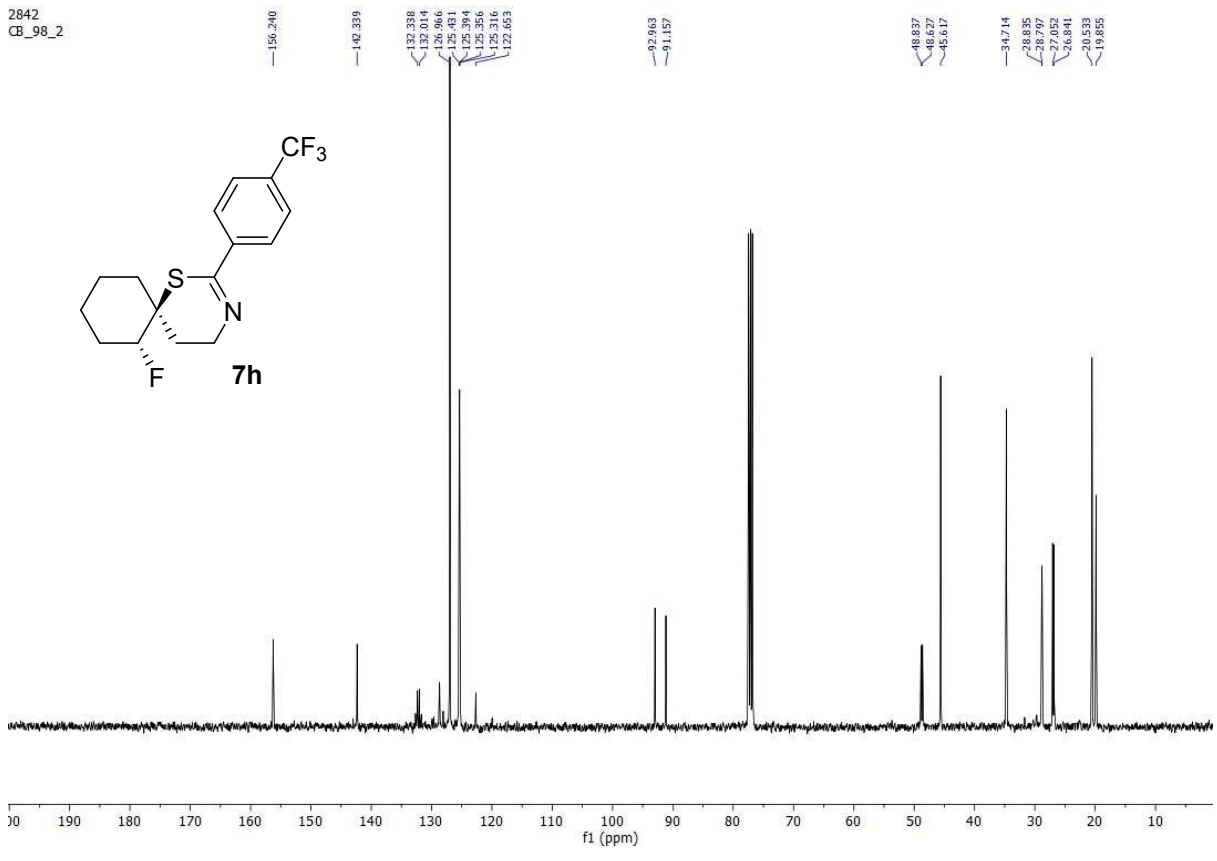
2182
CB_101_1 single pulse decoupled gated NOE

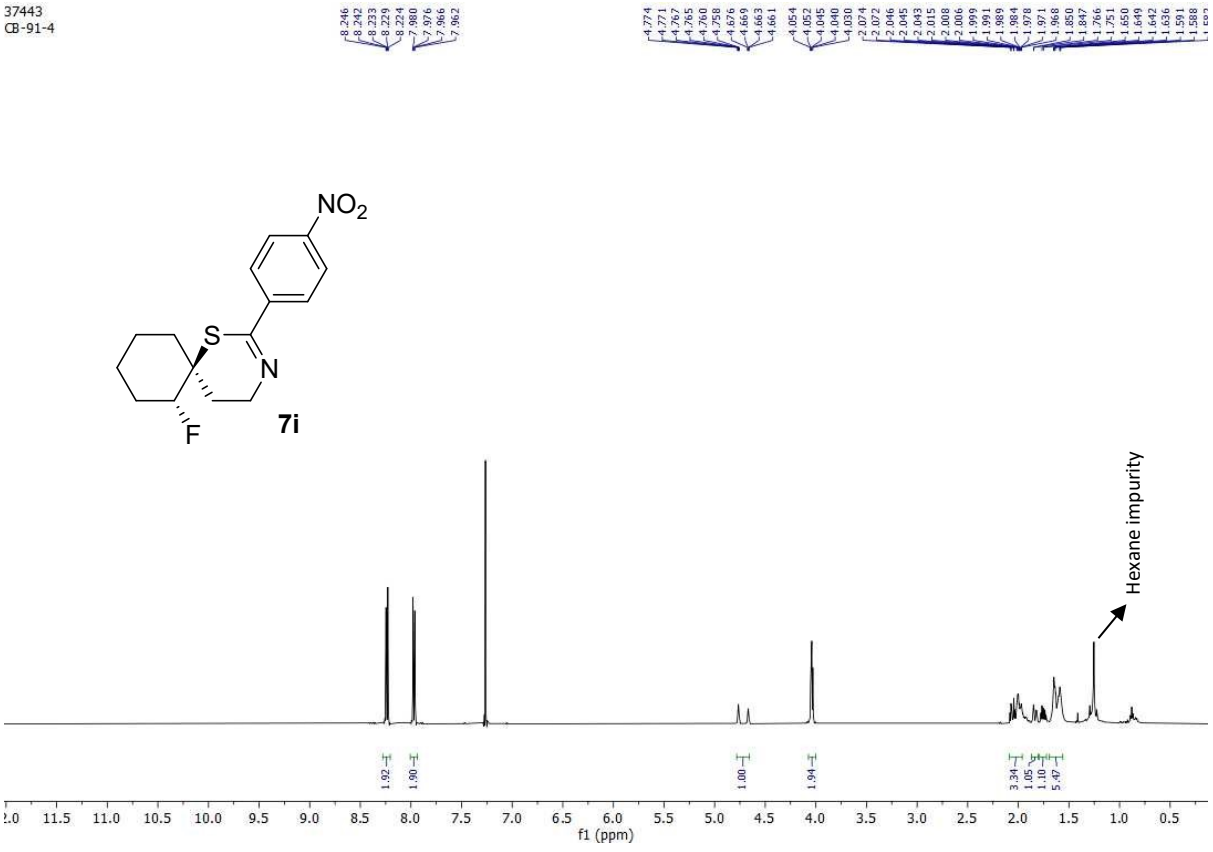


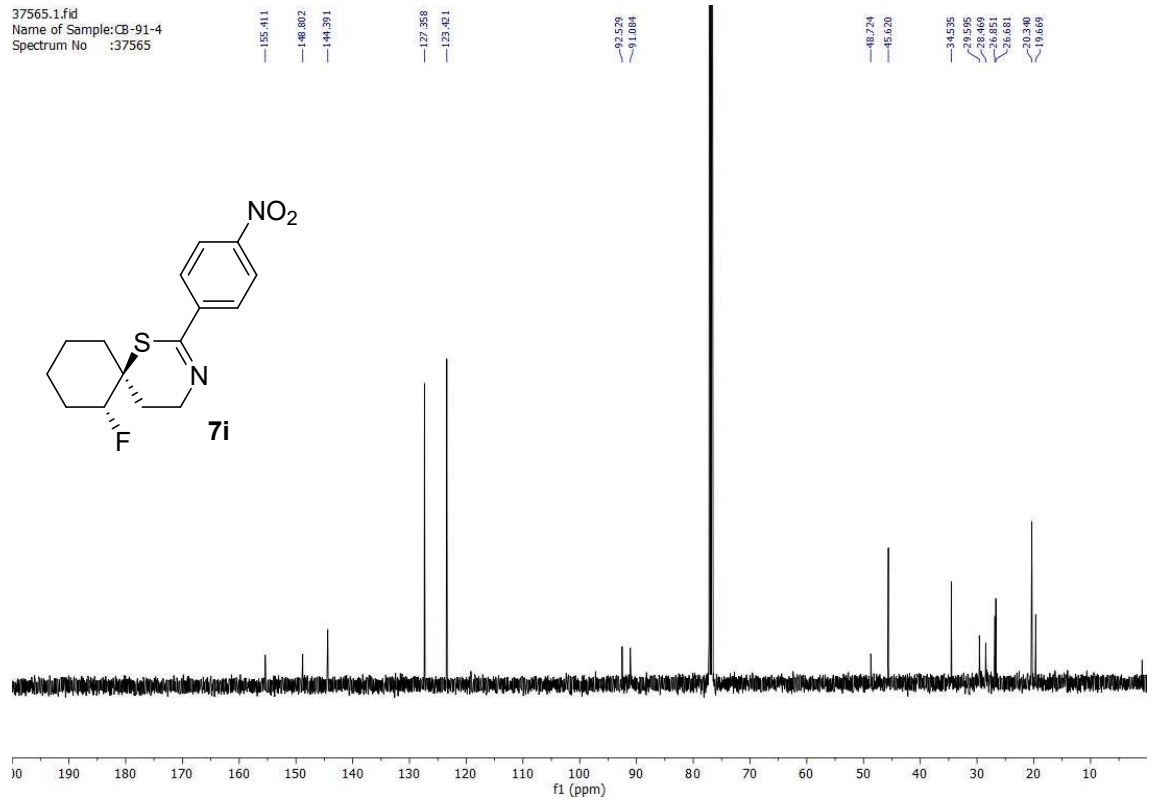
7g



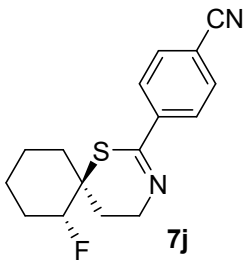

1291
CB_98_2 single_pulse
7.900
7.898
7.895
7.878
7.876
7.632
7.626
7.611
4.776
4.773
4.766
4.760
4.757
4.655
4.652
4.649
4.645
4.639
4.635
4.035
4.023
4.016
4.013
4.004
3.998
3.985
2.061
2.048
2.046
2.012
2.009
1.997
1.940
1.923
1.920
1.834
1.834
1.800
1.763
1.744
1.725
1.708
1.690
1.630
1.621
1.613
1.612
1.609
1.557
1.554
CF3
S
N
F
7h
Hexane impurity
2.16
2.43
1.00
2.09
5.01
3.02
2.38
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


2842
CB_98_2
156.240
142.339
132.338
132.014
126.966
125.431
125.394
125.356
125.316
122.653
92.963
91.157
48.837
48.627
45.617
34.714
28.835
28.797
27.052
26.841
20.533
19.855
CF3
S
N
F
7h
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

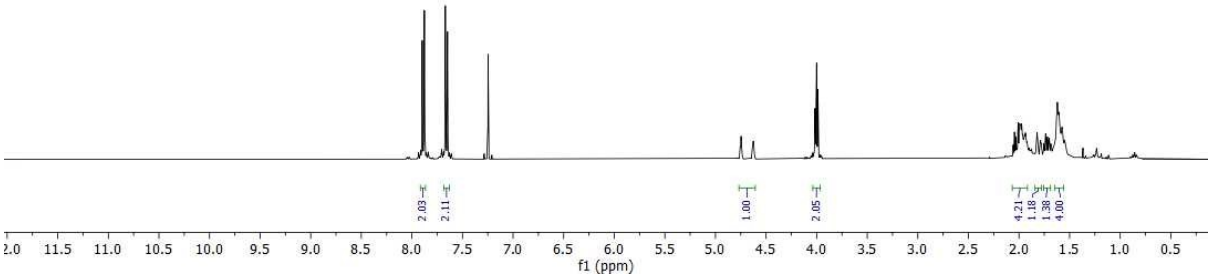

37443
CB-91-4
NO2
S
N
F
7i
Hexane impurity
f1 (ppm)


37565.1.fid
Name of Sample:CB-91-4
Spectrum No :37565
NO2
S
N
F
7i
f1 (ppm)

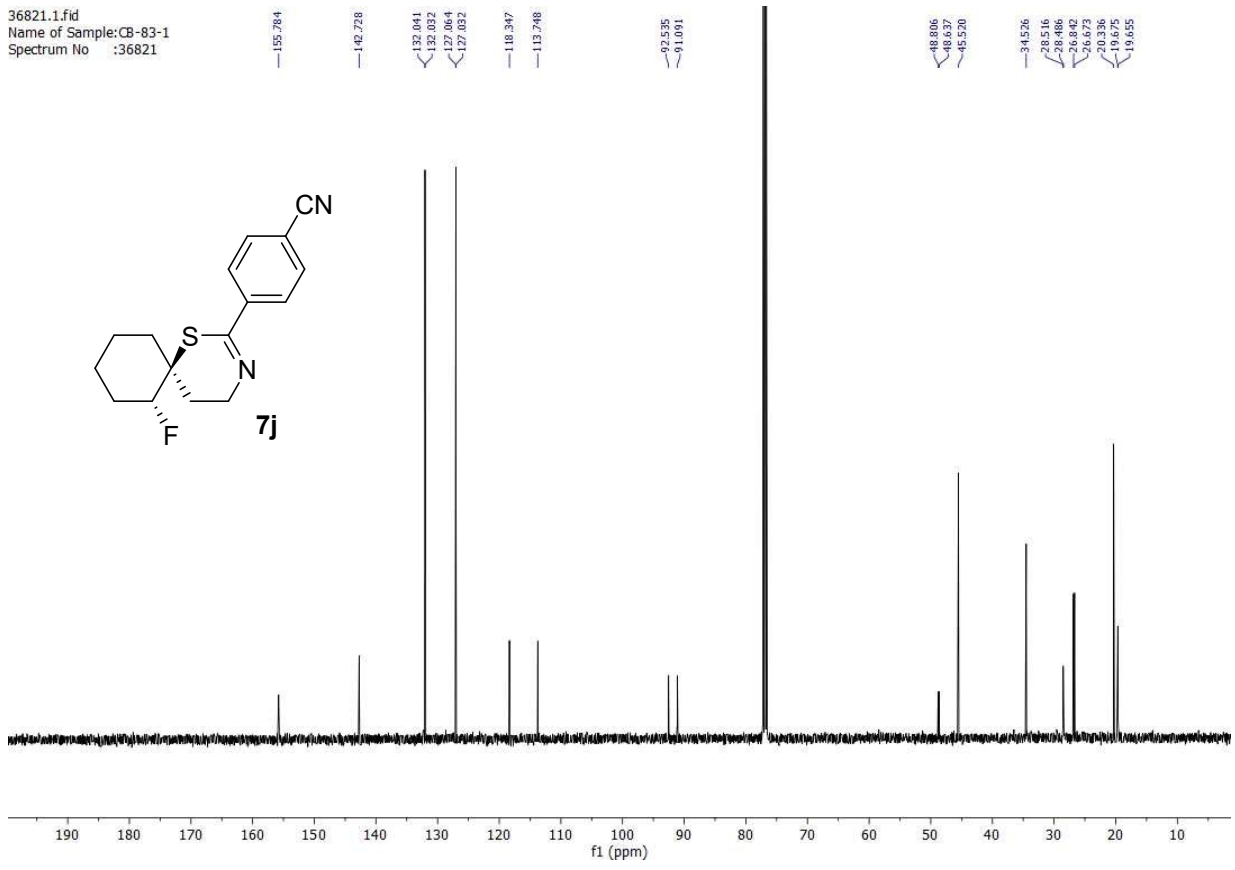
955
CB_83_1 single_pulse



7j

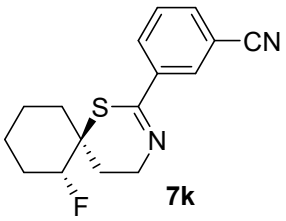


36821.1.fid
Name of Sample:CB-83-1
Spectrum No :36821

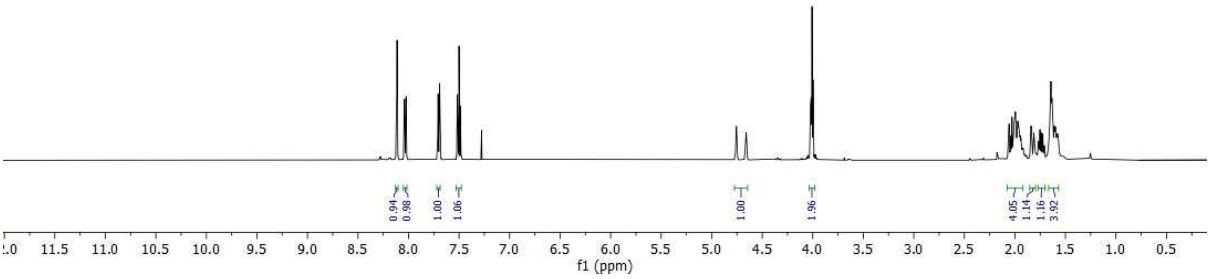


7j

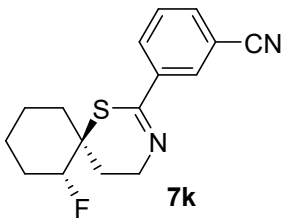
39343
CB-102-1



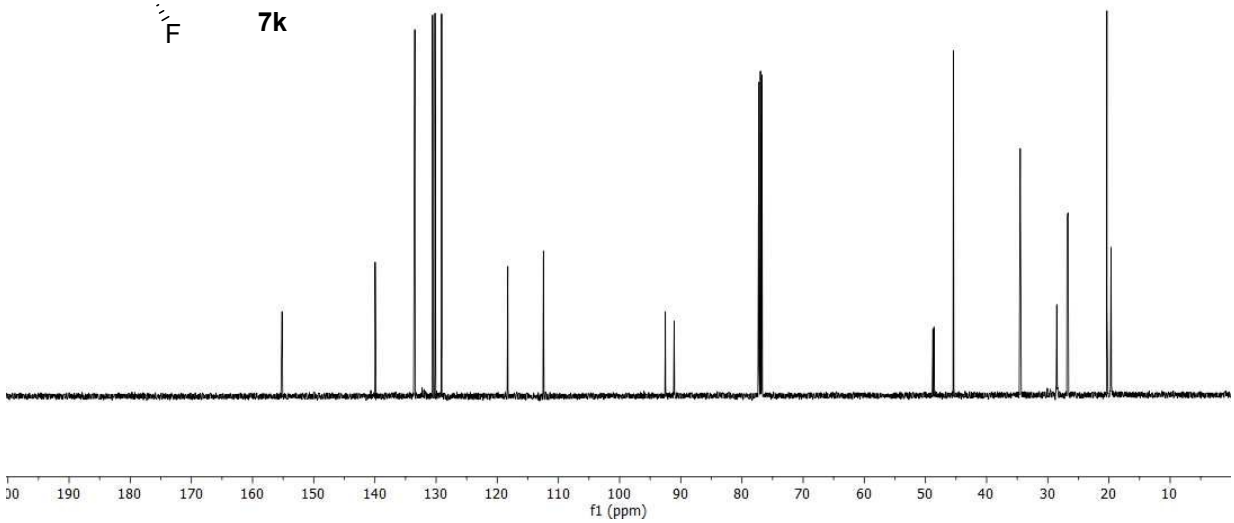
7k



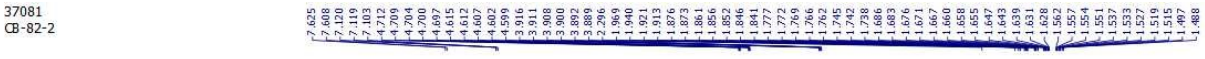
39352.1.fid
39352
CB-102-1

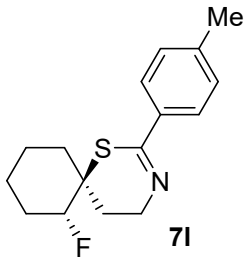


7k

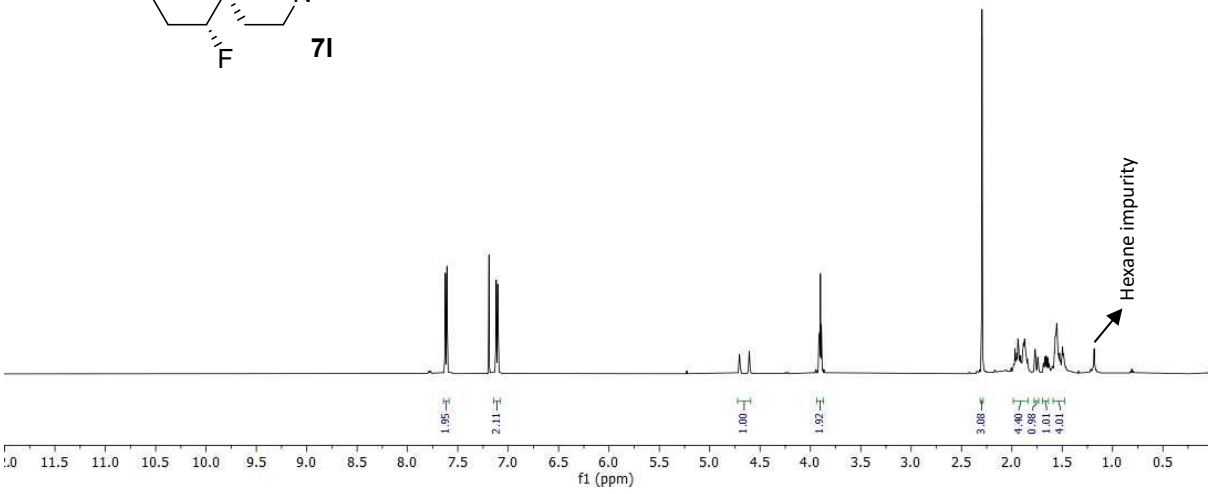


37081
CB-82-2

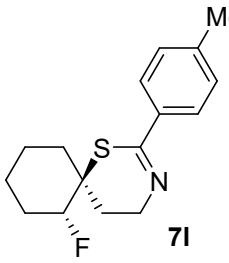




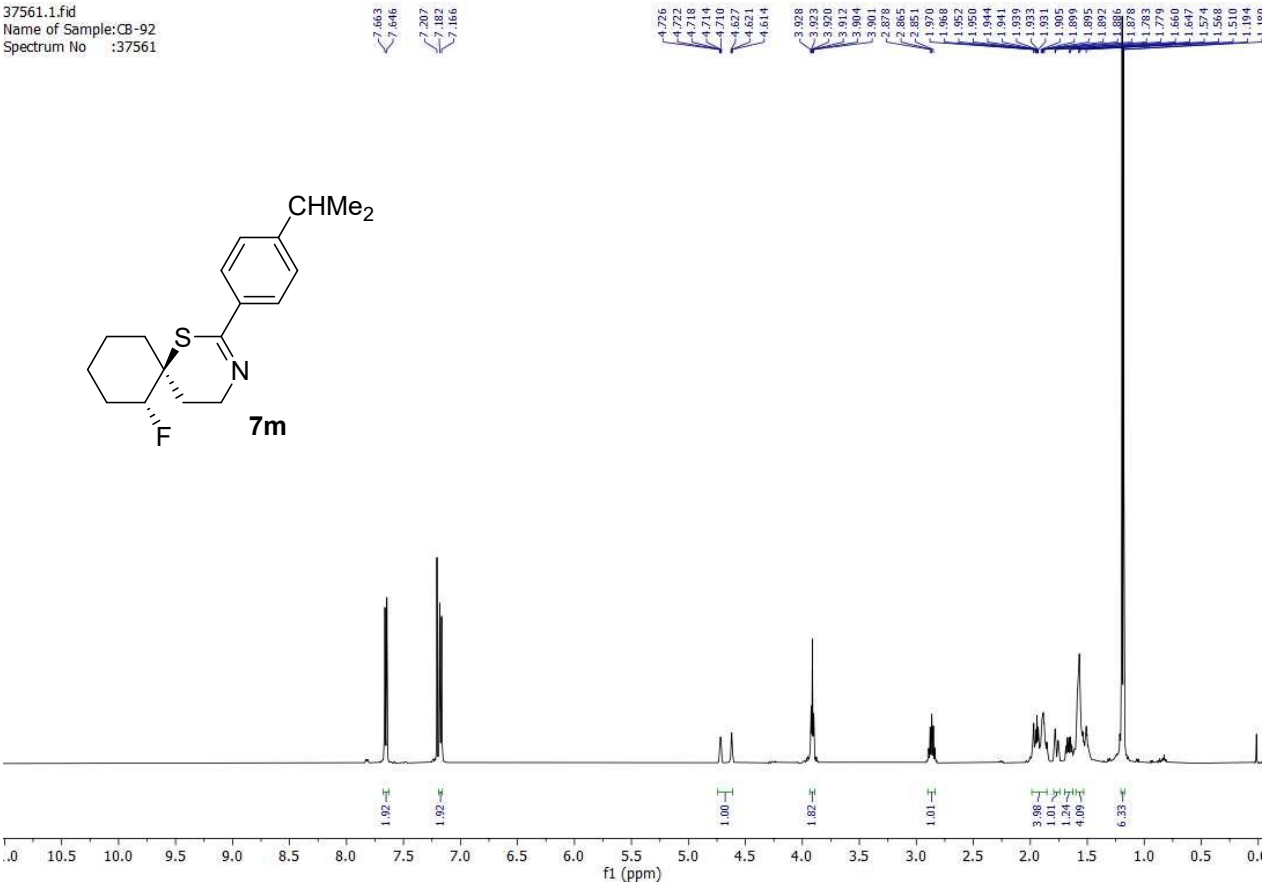
7l

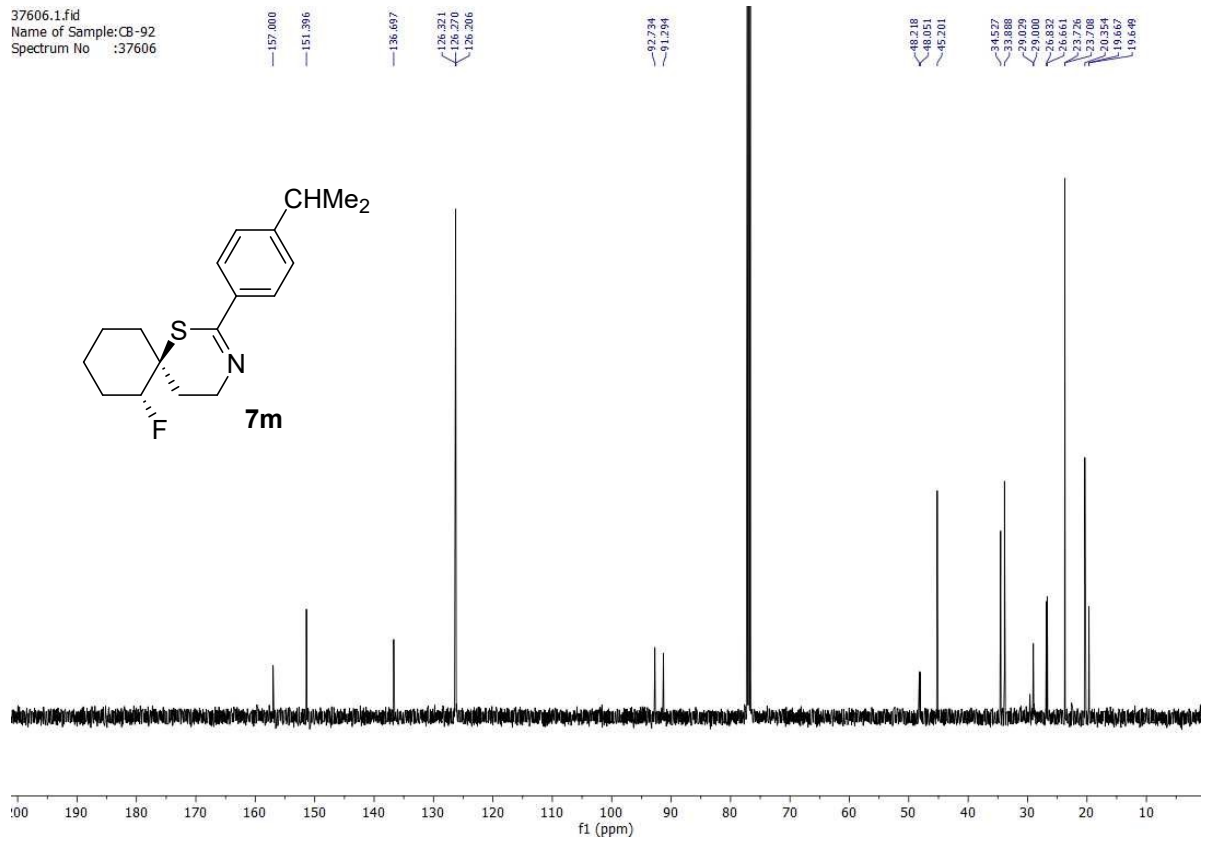


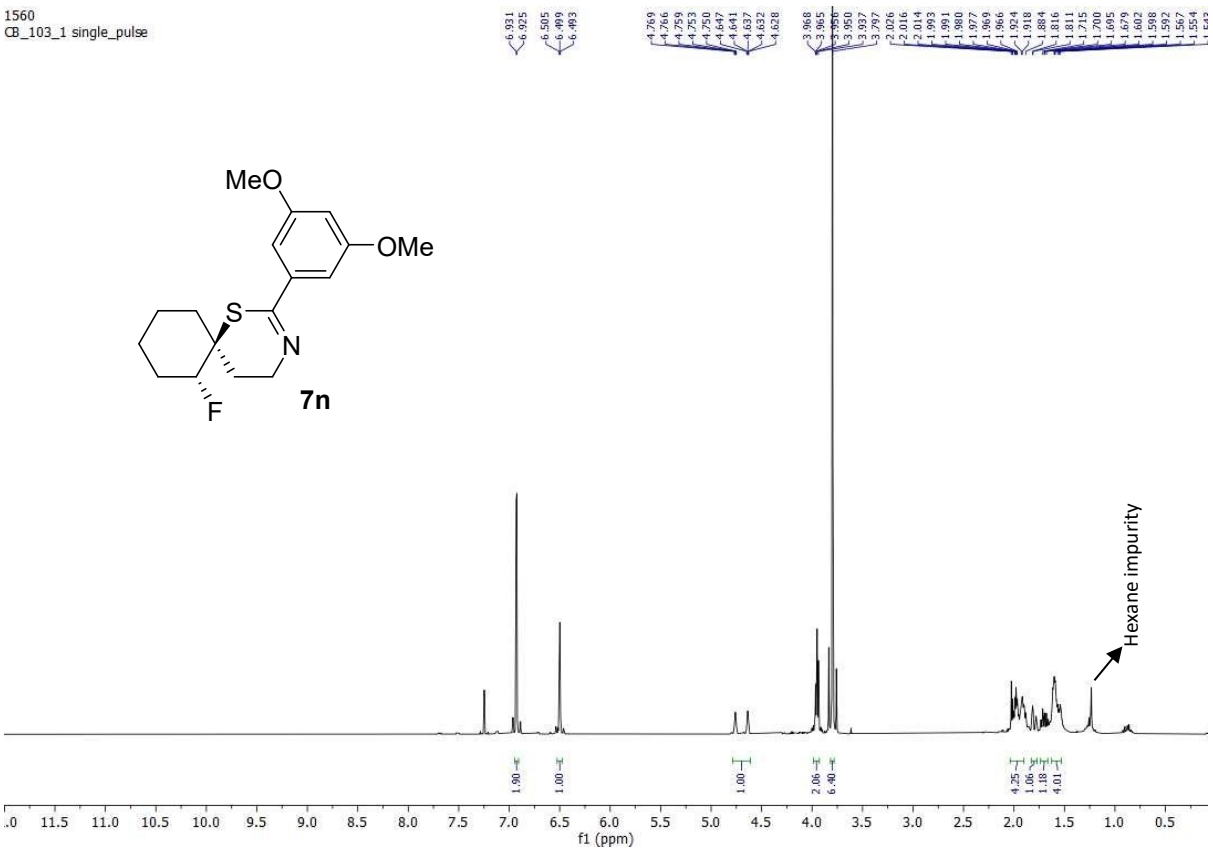
1843
CB_82_2 single pulse decoupled gated NOE

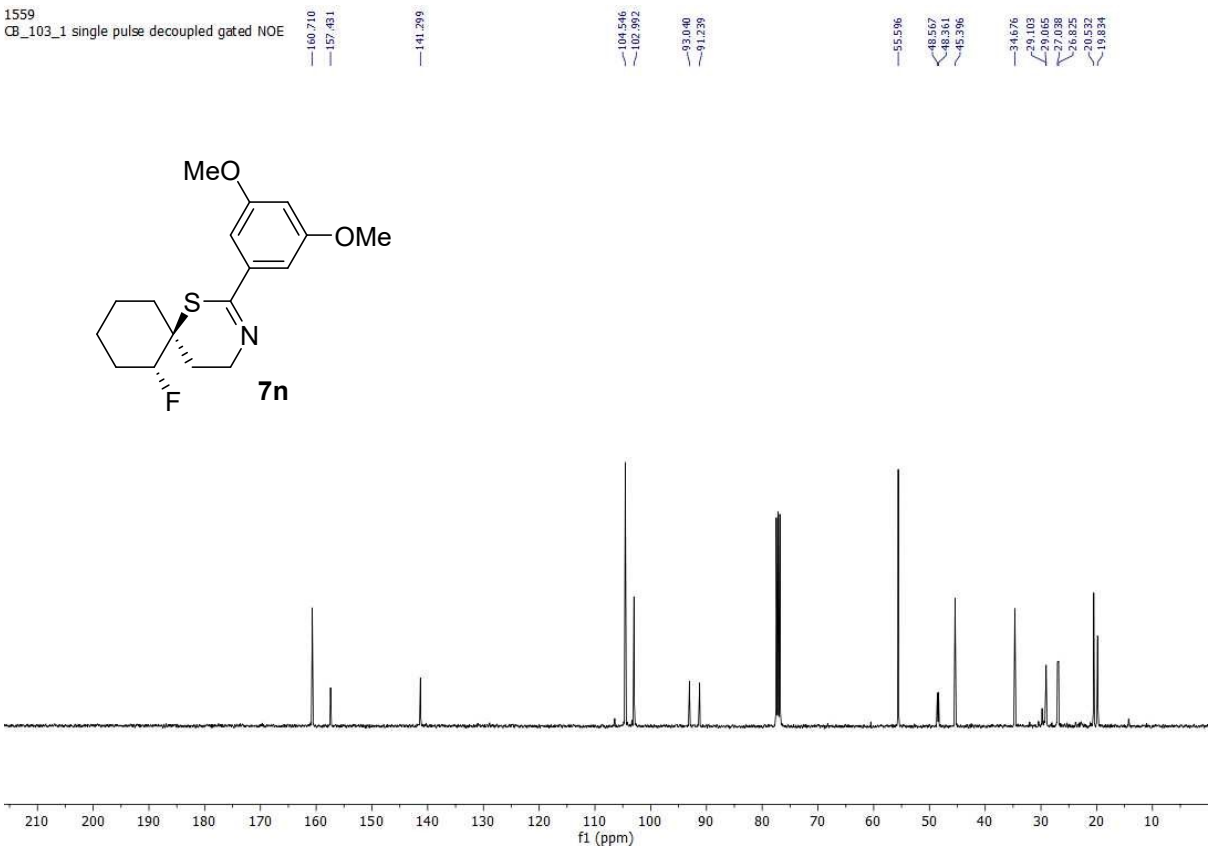


7l

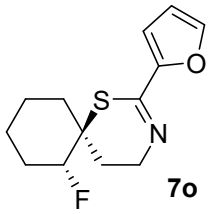

37561.1.fid
Name of Sample:CB-92
Spectrum No :37561
CHMe2
S
N
F
7m
f1 (ppm)


37606.1.fid
Name of Sample:CB-92
Spectrum No :37606
CHMe2
S
N
F
7m
f1 (ppm)

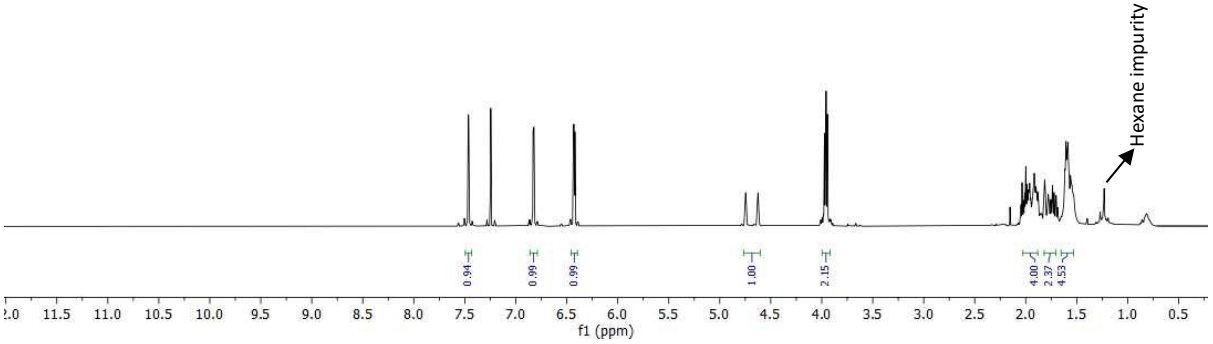

1560
CB_103_1 single_pulse
MeO
OMe
S
N
F
7n
Hexane impurity
f1 (ppm)


1559
CB_103_1 single pulse decoupled gated NOE
MeO
OMe
S
N
F
7n
f1 (ppm)

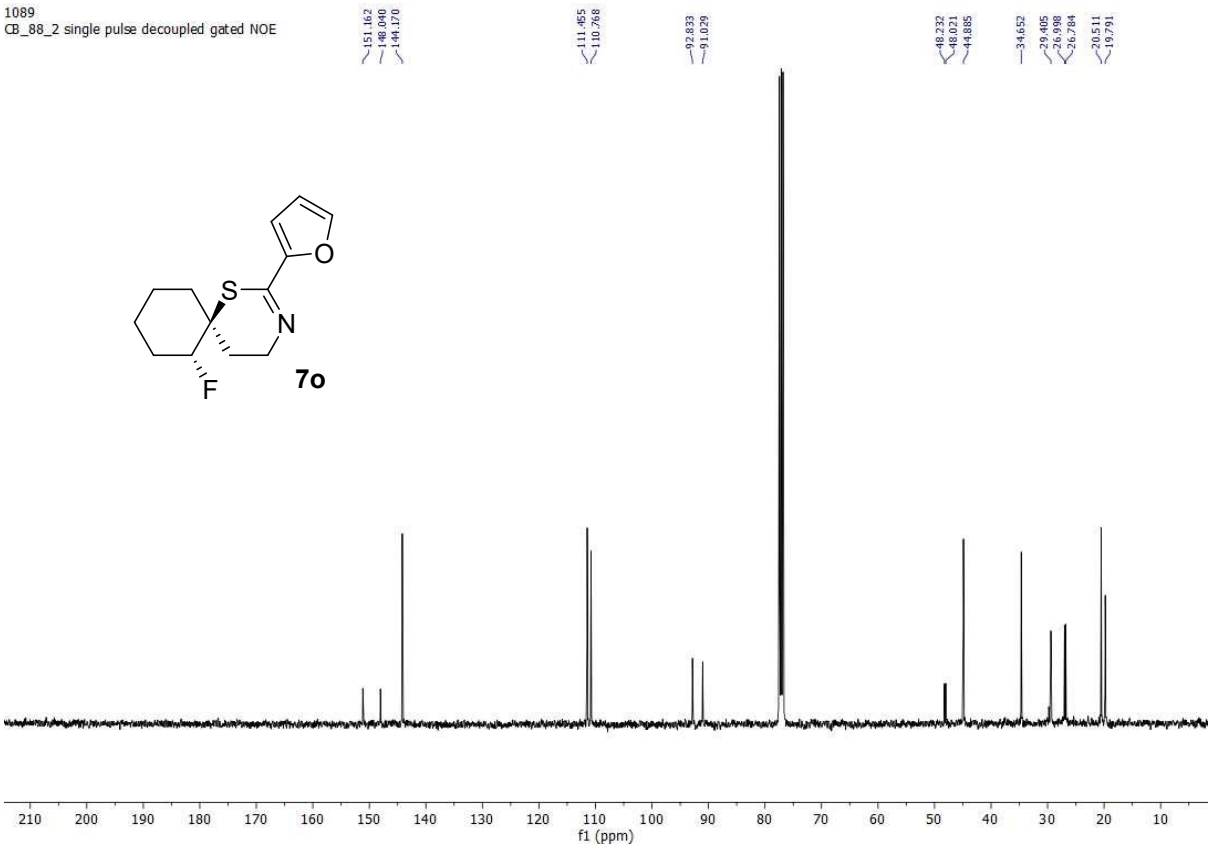
1090
CB_88_2 single_pulse



7o

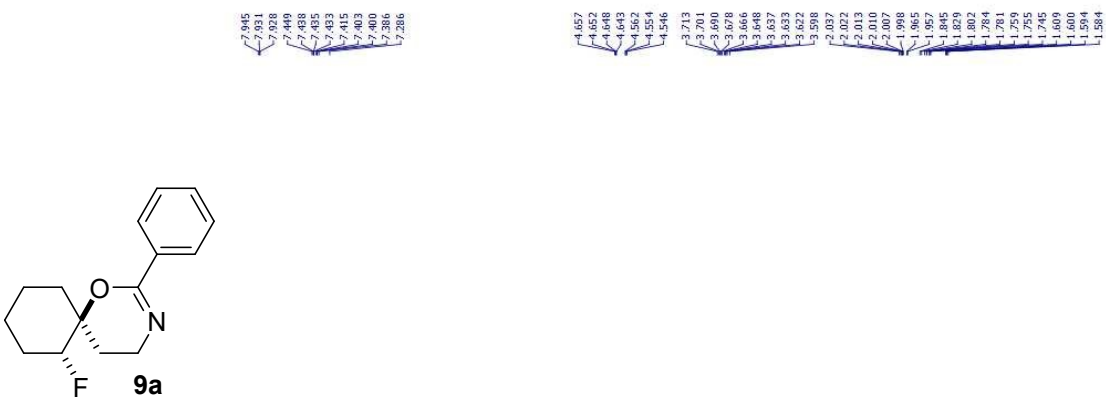


1089
CB_88_2 single pulse decoupled gated NOE

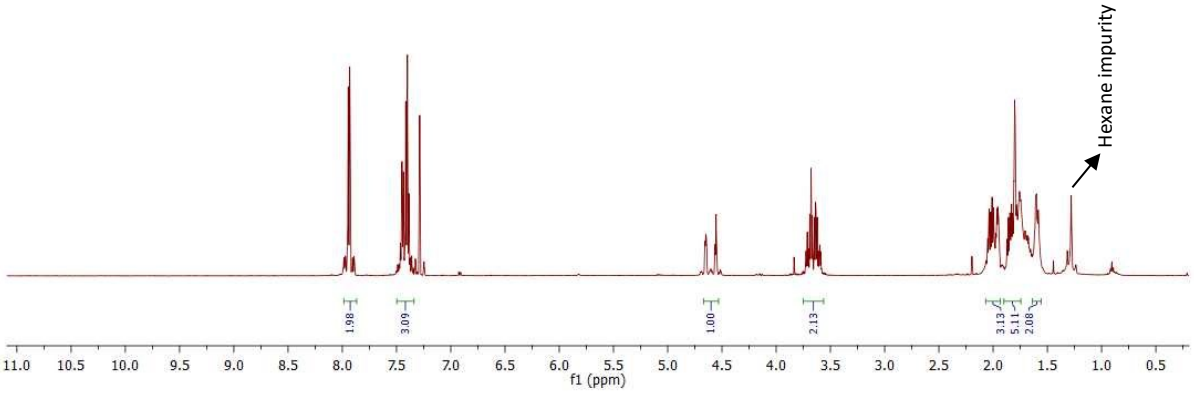


7o

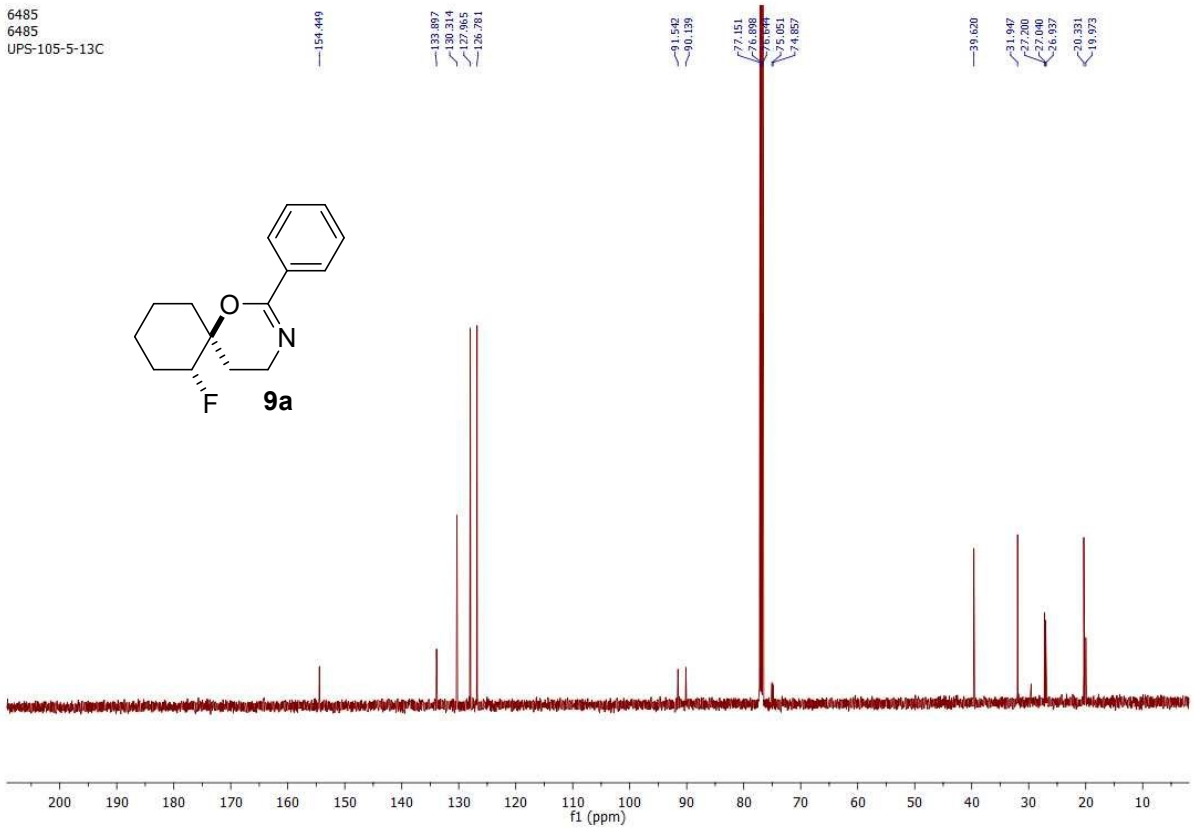
6484
6484
UPS-105-5



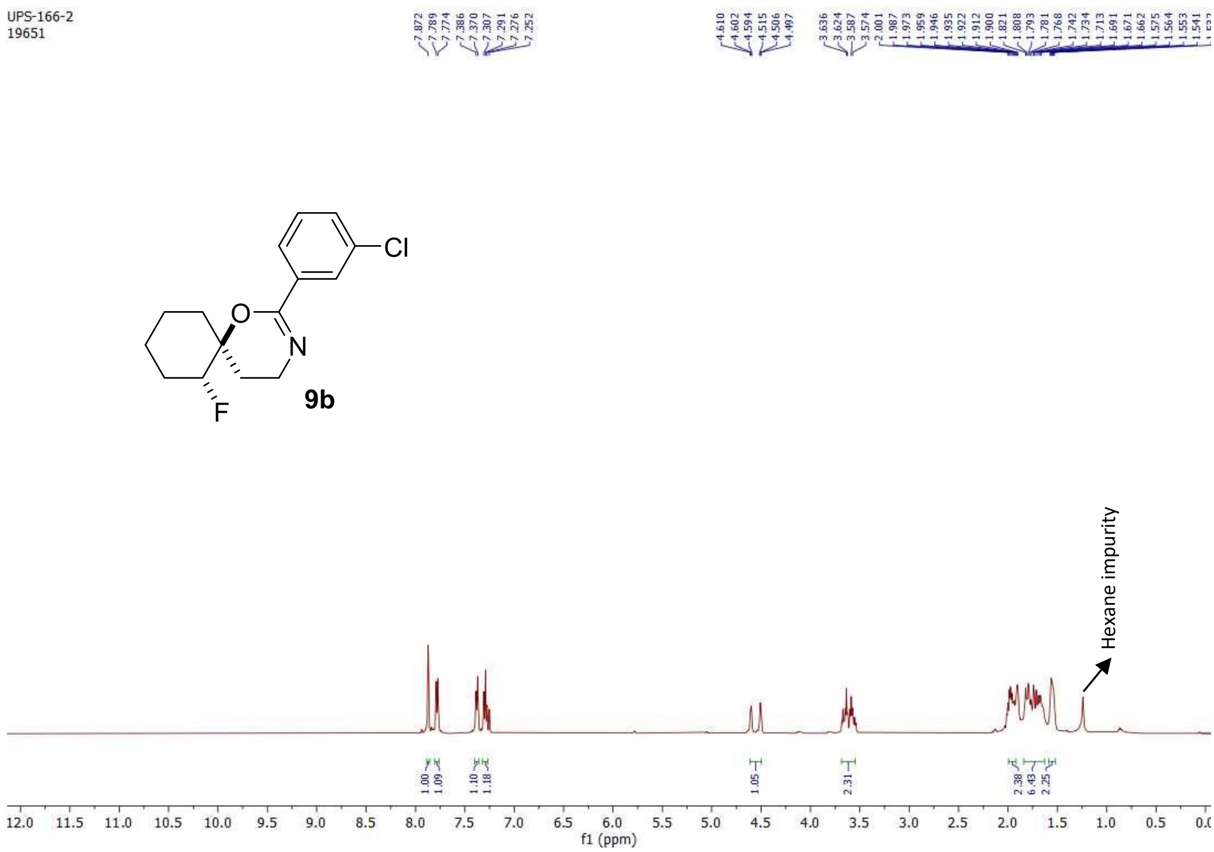
9a



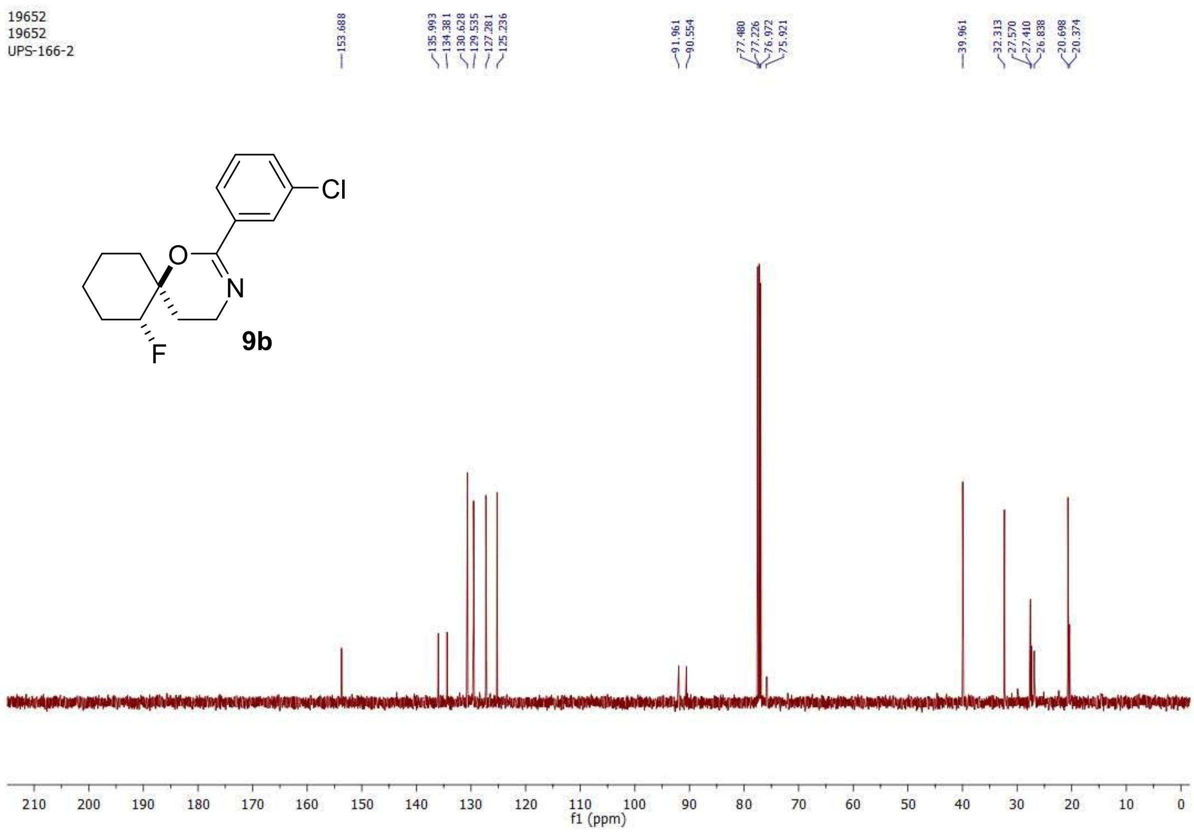
6485
6485
UPS-105-5-13C



9a

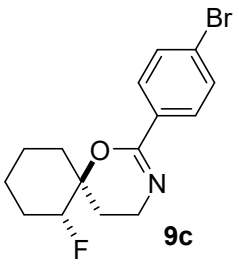


UPS-166-2
19651
9b
Hexane impurity
f1 (ppm)

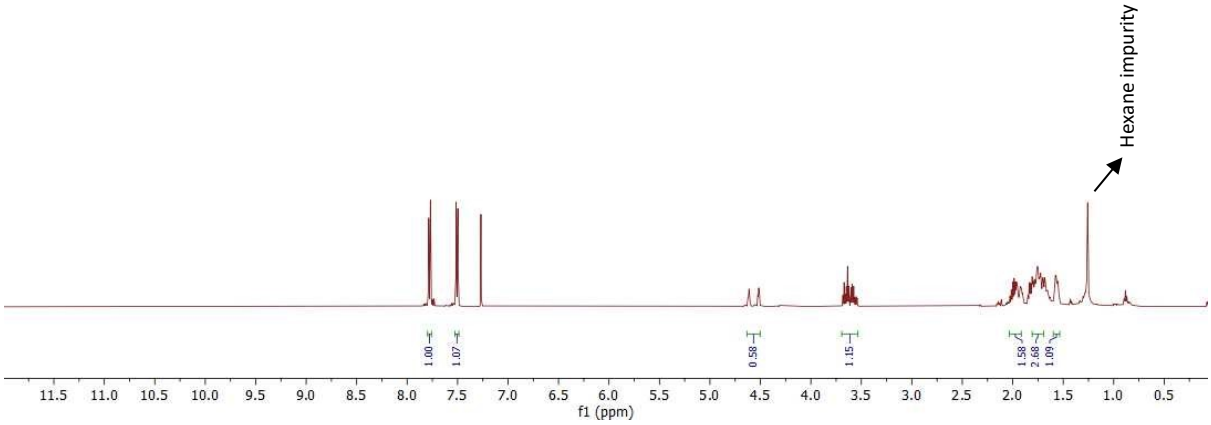


19652
19652
UPS-166-2
9b
f1 (ppm)

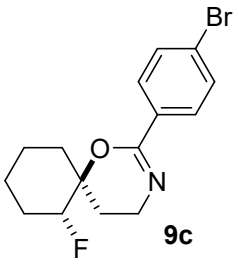
19210
UPS-162-1



9c

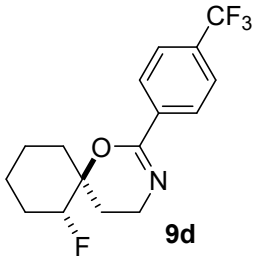


19211
19211
UPS-162-1

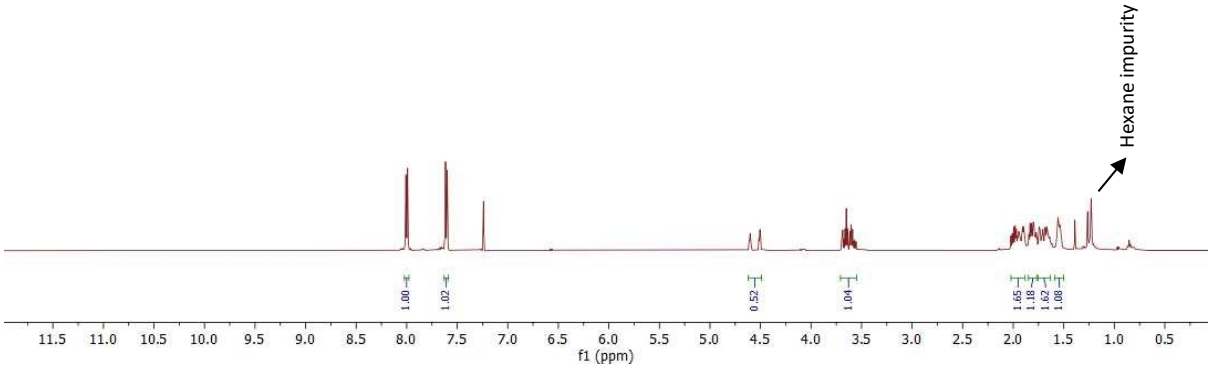


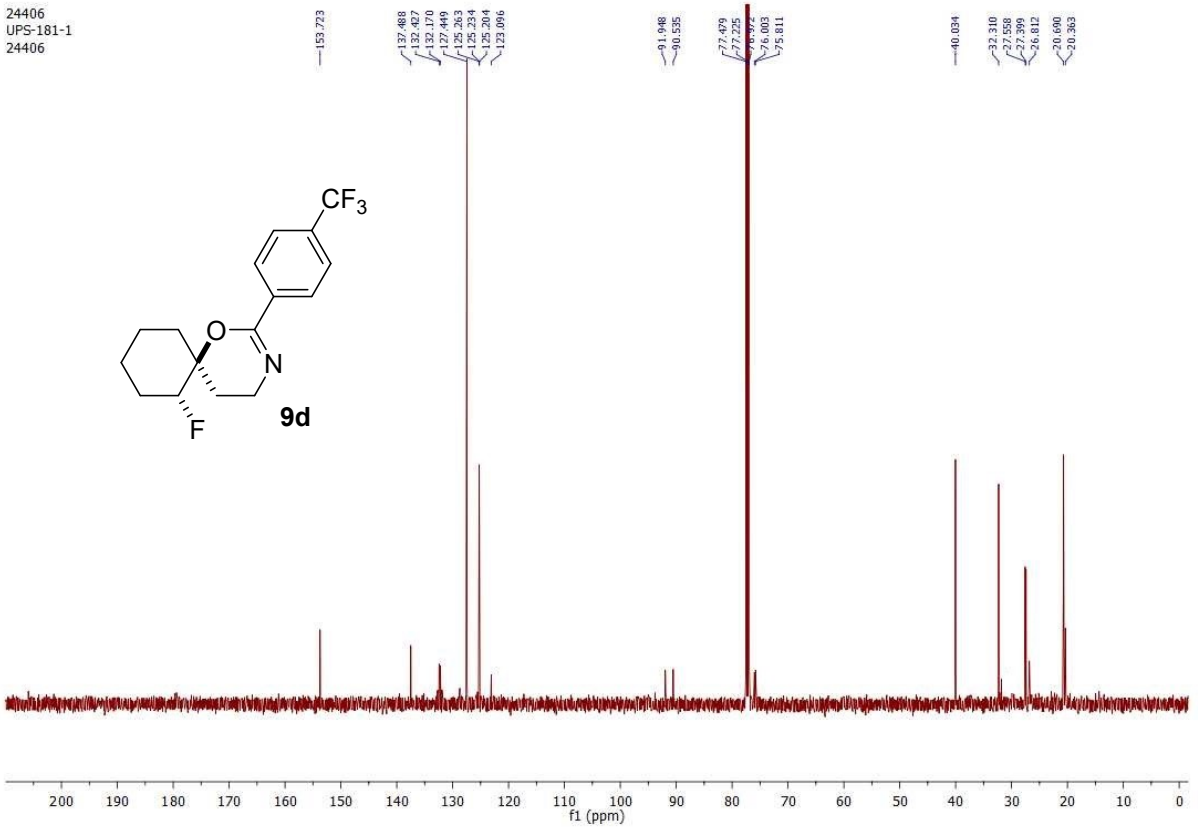
9c


24405
UPS-181-1


CF3
O
N
F

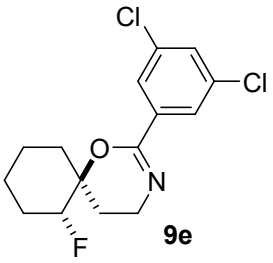
9d


Hexane impurity
1.00
1.02
0.52
1.04
1.65
1.18
1.62
1.08
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

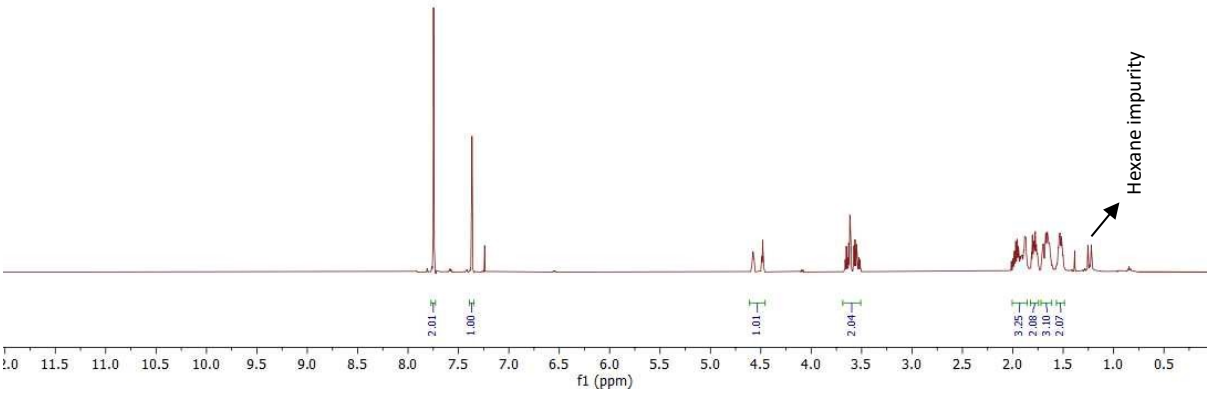

24406
UPS-181-1
24406
153.723
137.488
132.427
132.170
127.449
125.263
125.234
125.204
123.096
91.948
90.535
77.479
77.225
76.972
76.003
75.811
40.034
32.310
27.558
27.399
26.812
20.690
20.363
CF3
O
N
F
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

9d

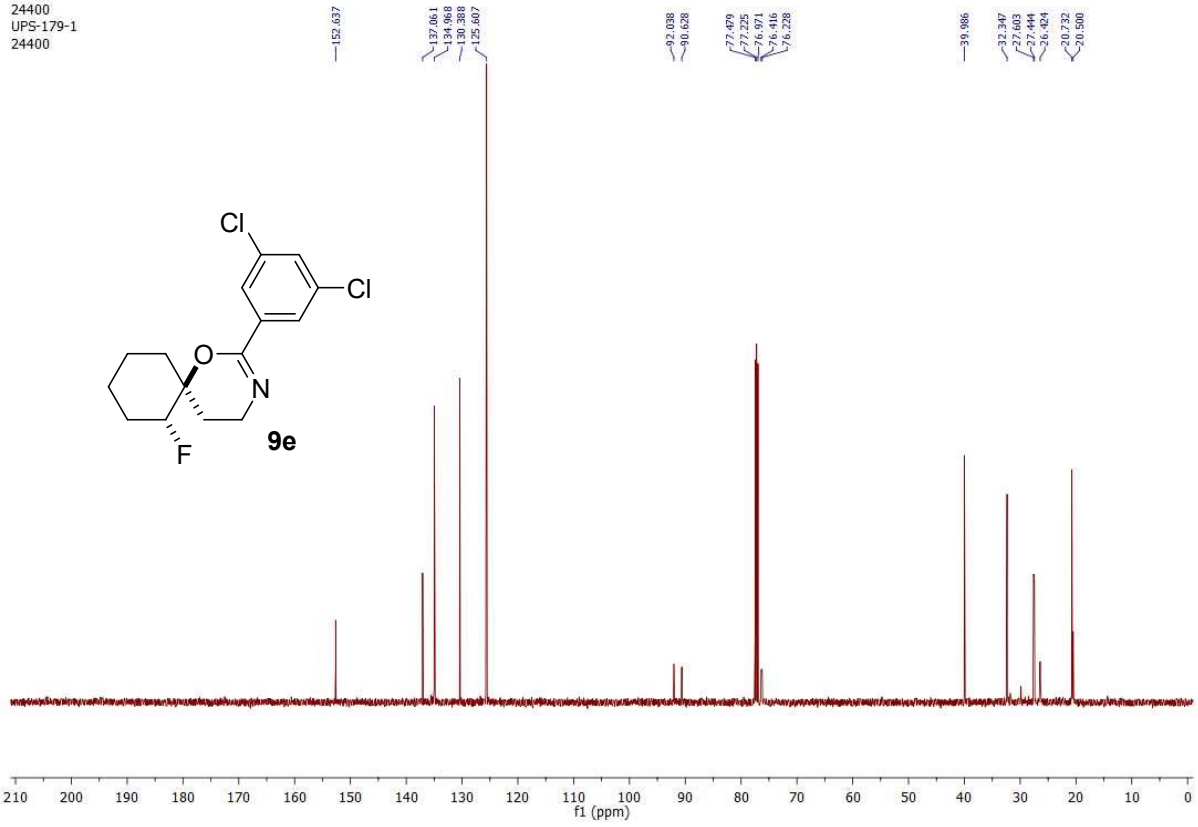
24399
UPS-179-1



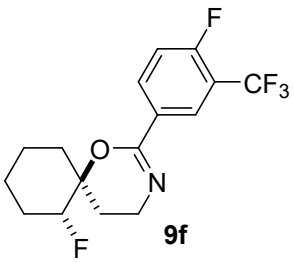
9e



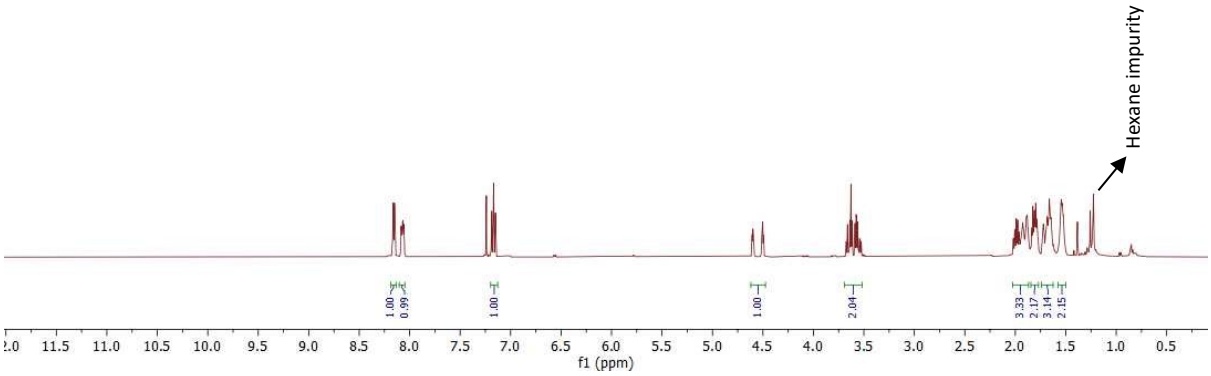
24400
UPS-179-1
24400

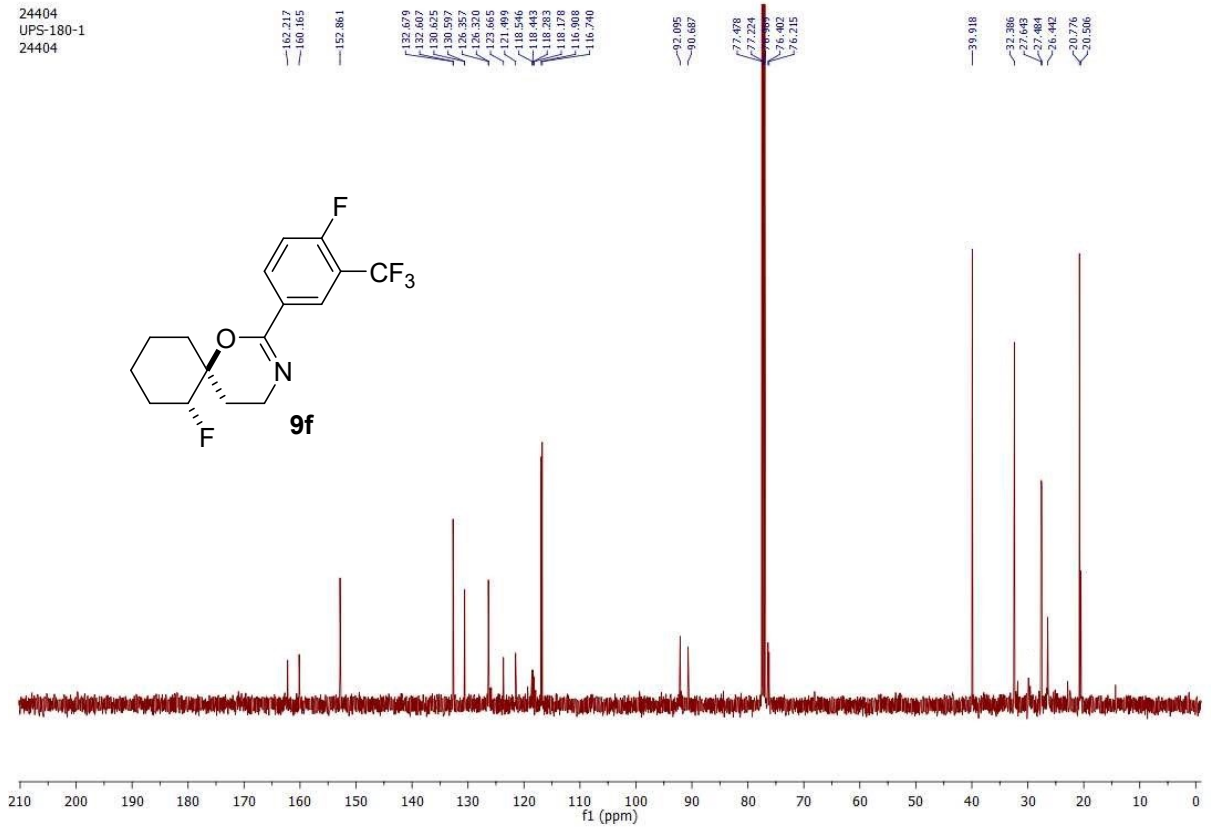


9e

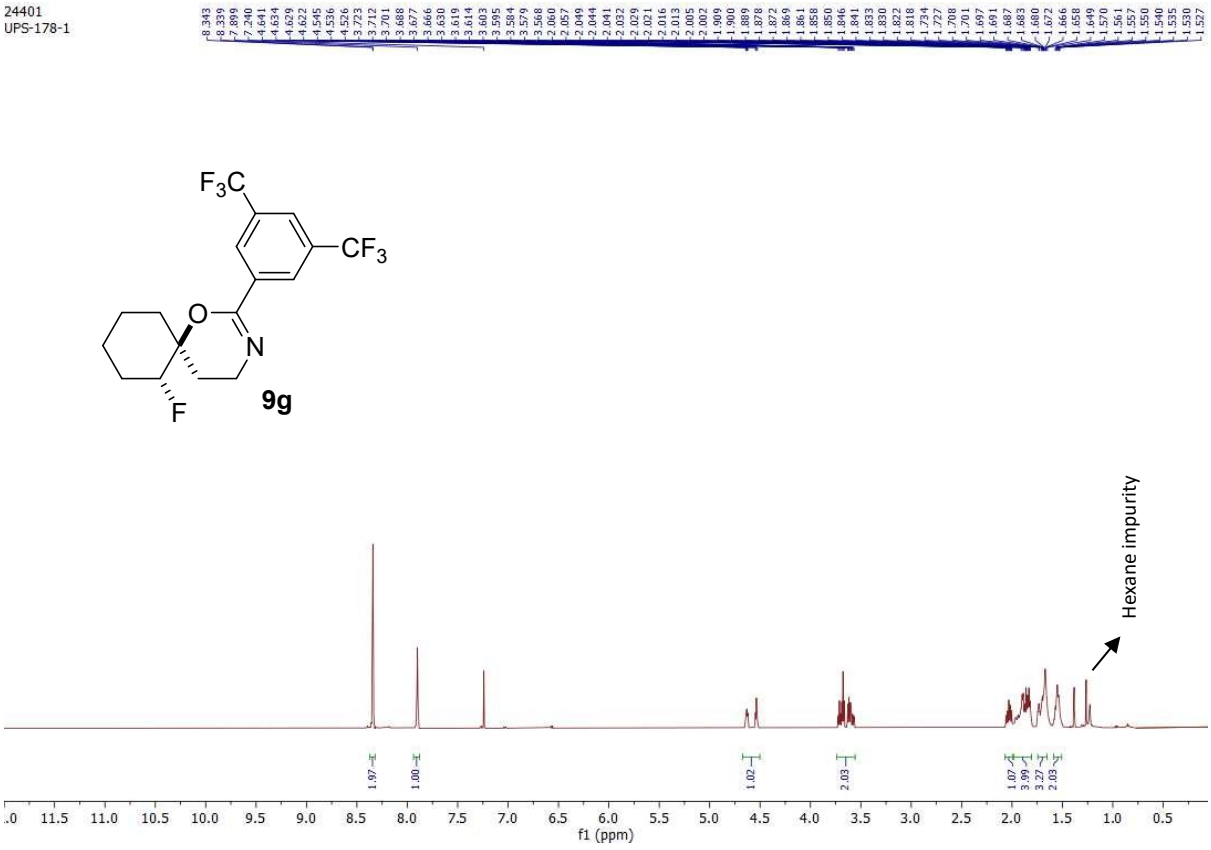

F
CF3
O
N
F

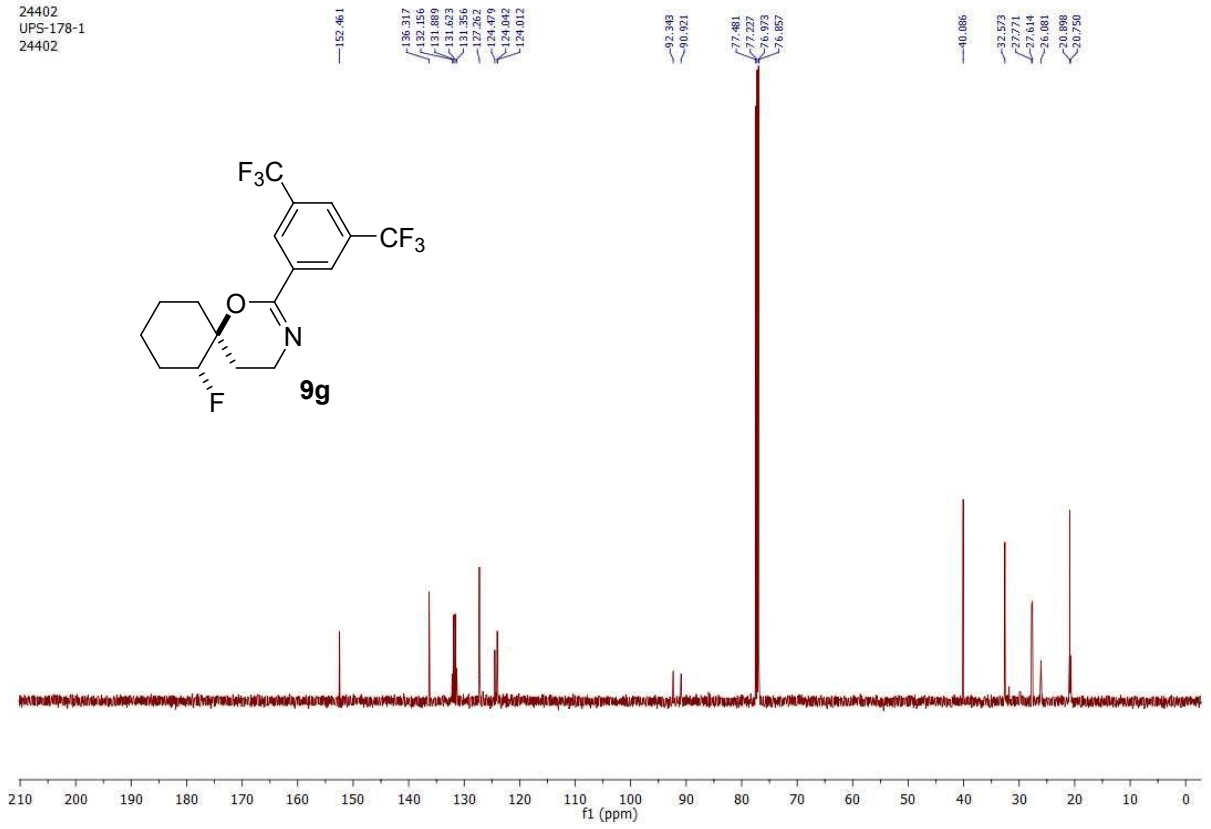
9f

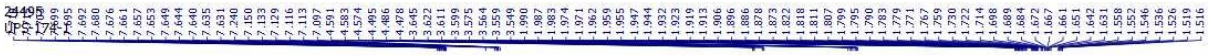

Hexane impurity
1.00
0.99
1.00
1.00
2.04
3.33
2.17
3.14
2.15
f1 (ppm)

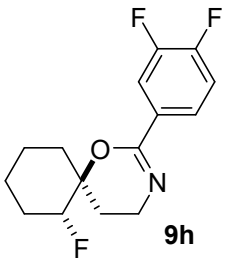

24404
UPS-180-1
24404
F
CF3
O
N
F
f1 (ppm)

9f

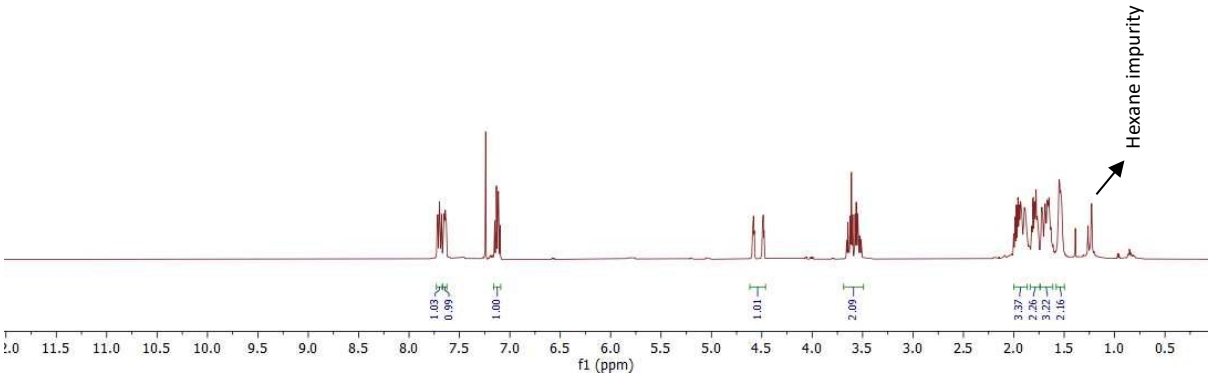

24401
UPS-178-1
F3C
CF3
O
N
F
9g
Hexane impurity
f1 (ppm)

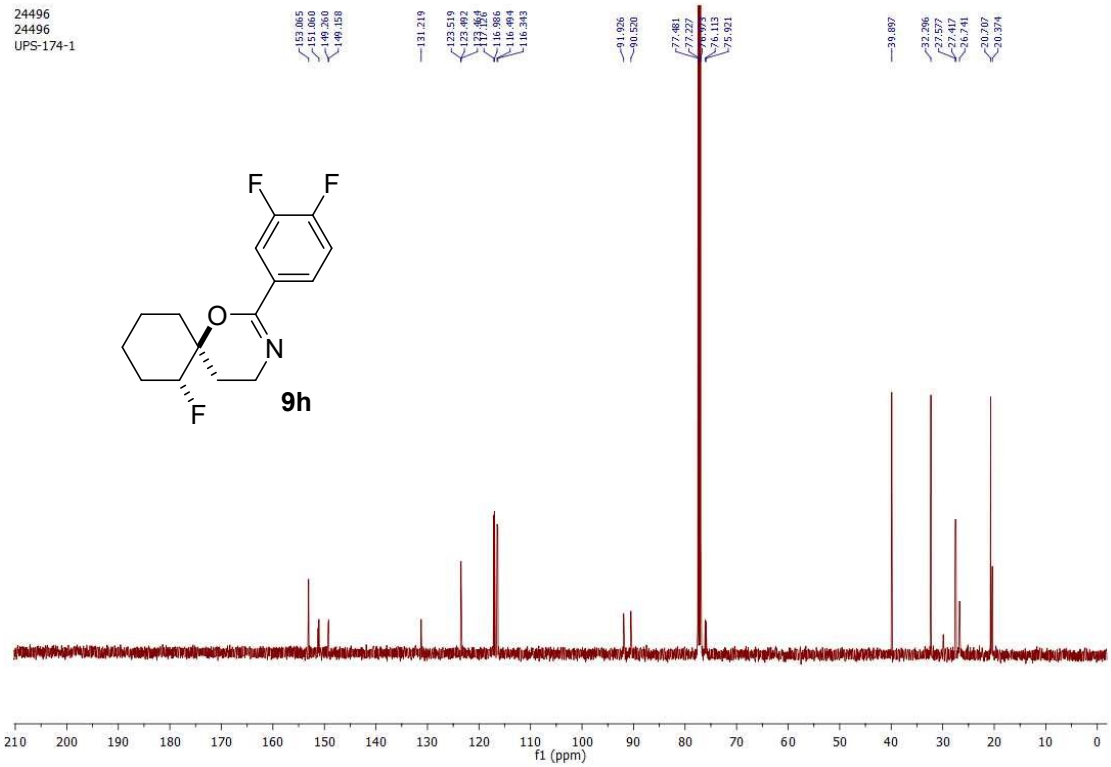

24402
UPS-178-1
24402
F3C
CF3
O
N
F
9g
f1 (ppm)




F
F
O
N
F

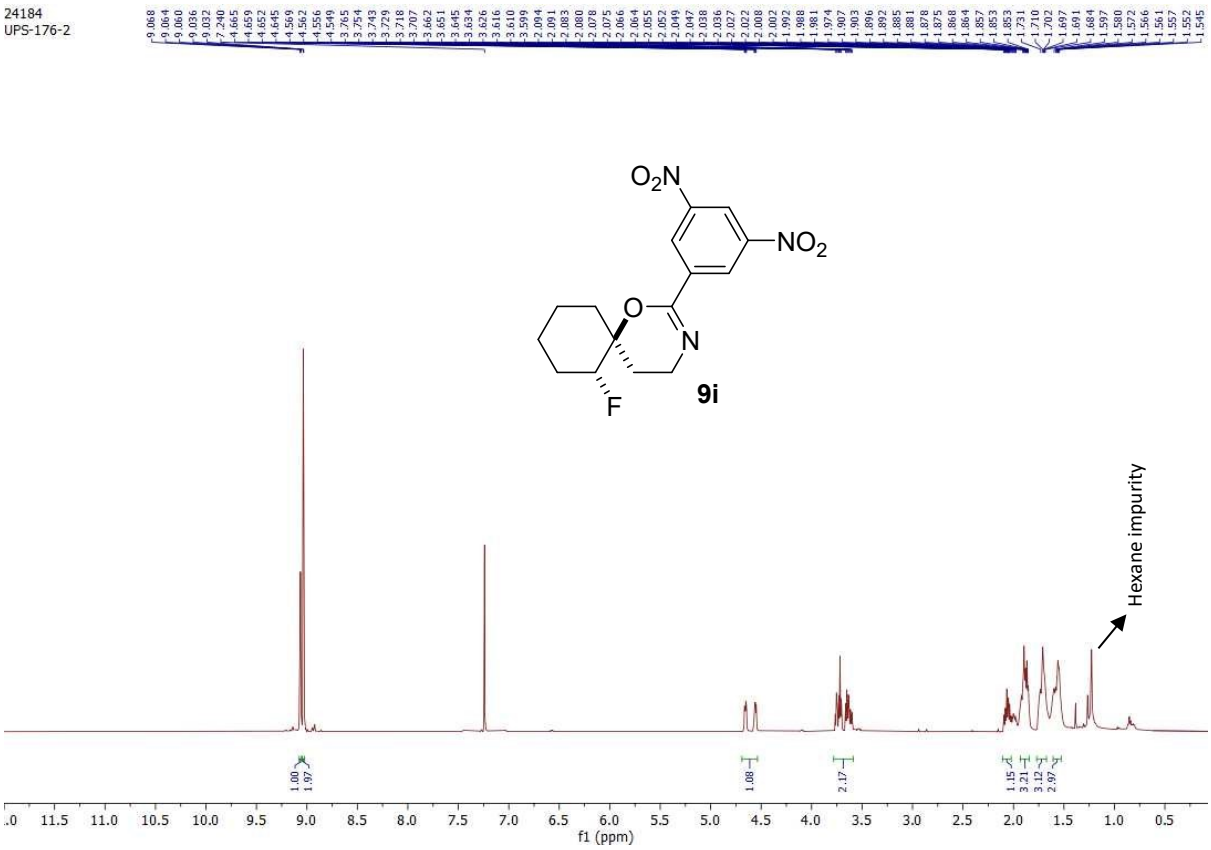
9h

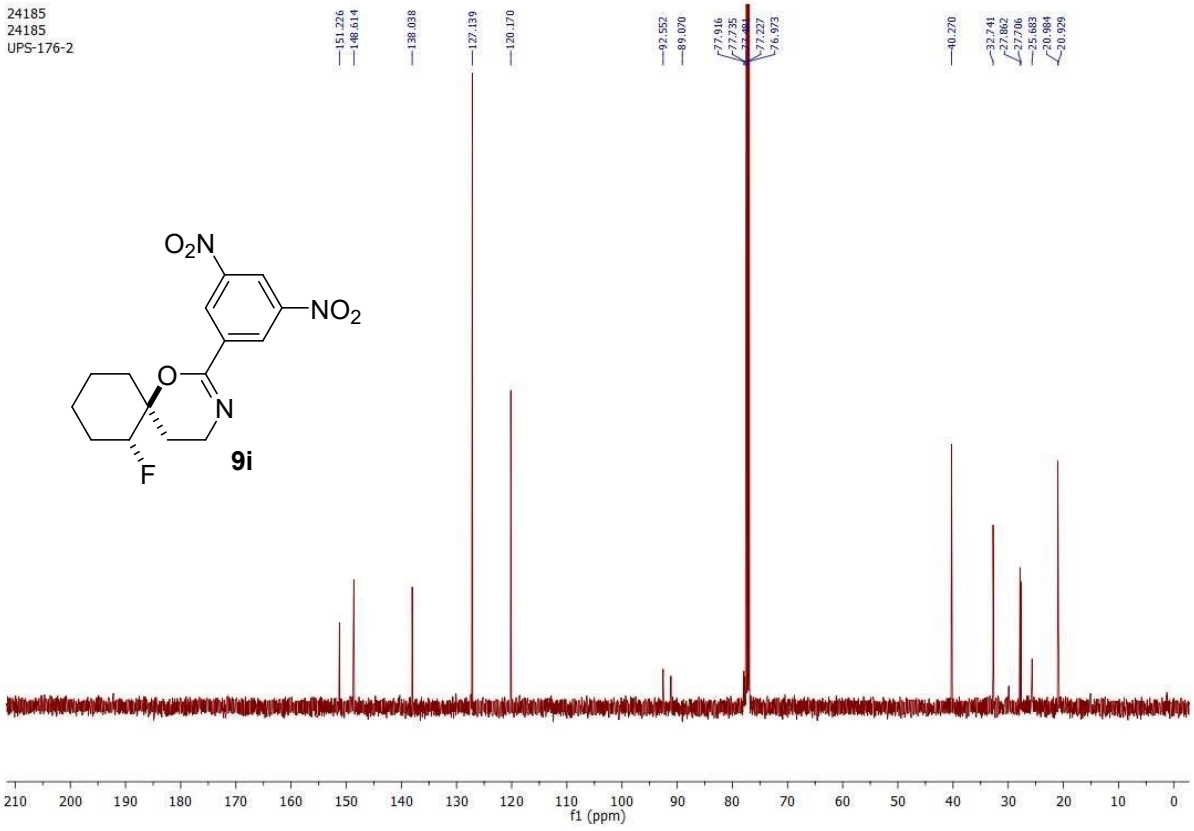

Hexane impurity
1.03
0.99
1.00
1.01
2.09
3.37
2.26
3.22
2.16
f1 (ppm)


24496
24496
UPS-174-1
f1 (ppm)

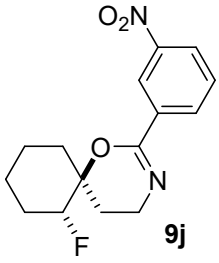
F
F
O
N
F

9h

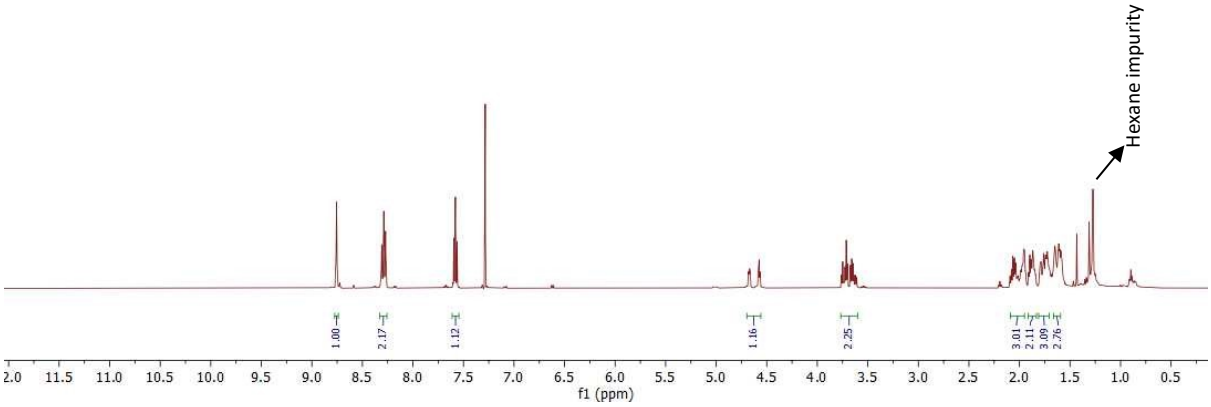

24184
UPS-176-2
O2N
NO2
O
N
F
9i
Hexane impurity
f1 (ppm)

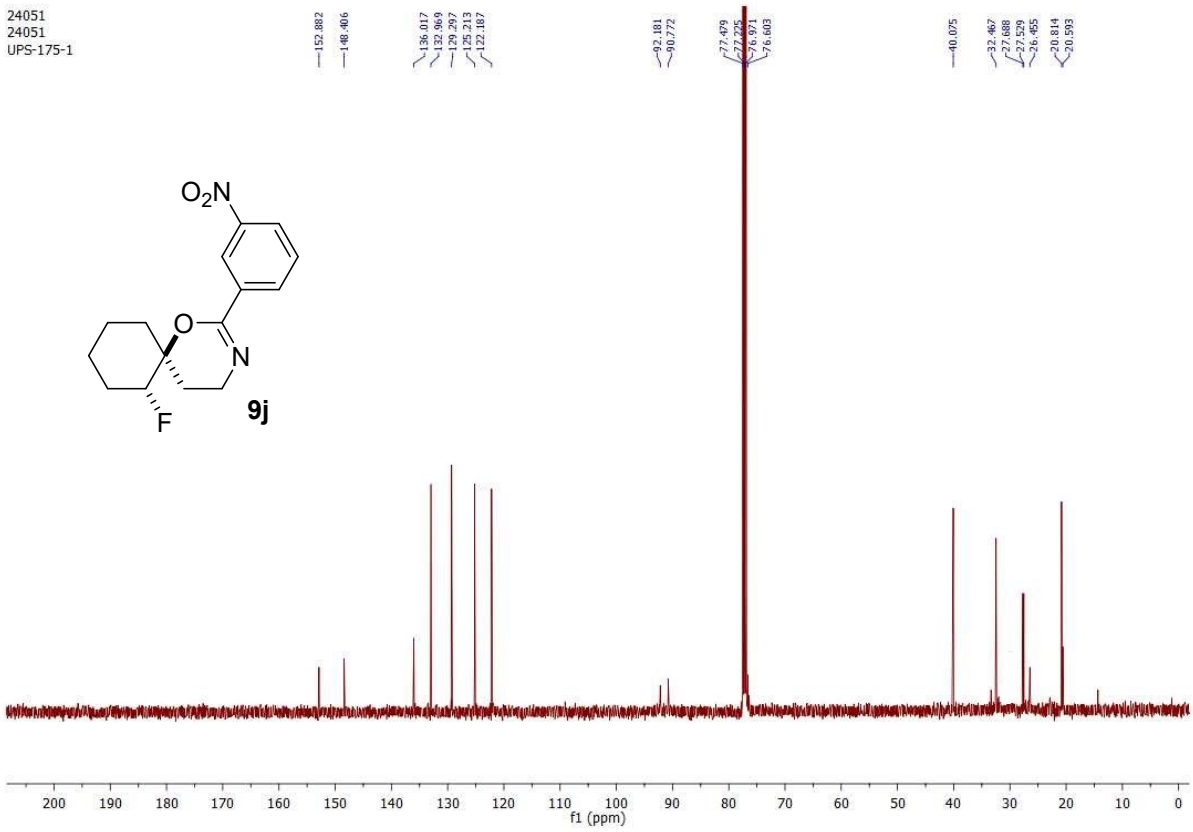

24185
24185
UPS-176-2
O2N
NO2
O
N
F
9i
f1 (ppm)


UPS-175-1
24050


O2N
O
N
F

9j

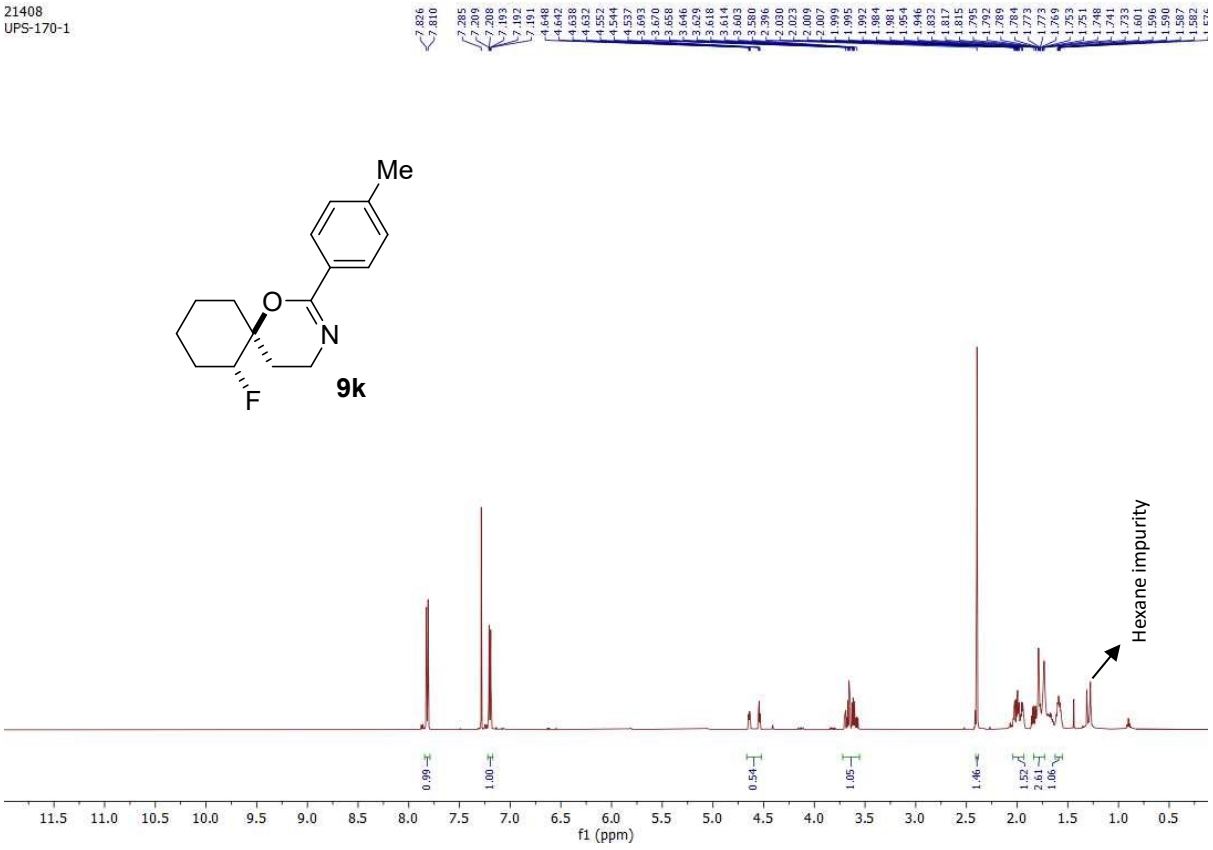

Hexane impurity
1.00
2.17
1.12
1.16
2.25
3.01
2.11
3.09
2.76
f1 (ppm)


24051
24051
UPS-175-1
152.882
148.406
136.017
132.969
129.297
125.213
122.187
92.181
90.772
77.479
77.275
76.971
76.603
40.075
32.467
27.688
27.529
26.455
20.814
20.593
f1 (ppm)

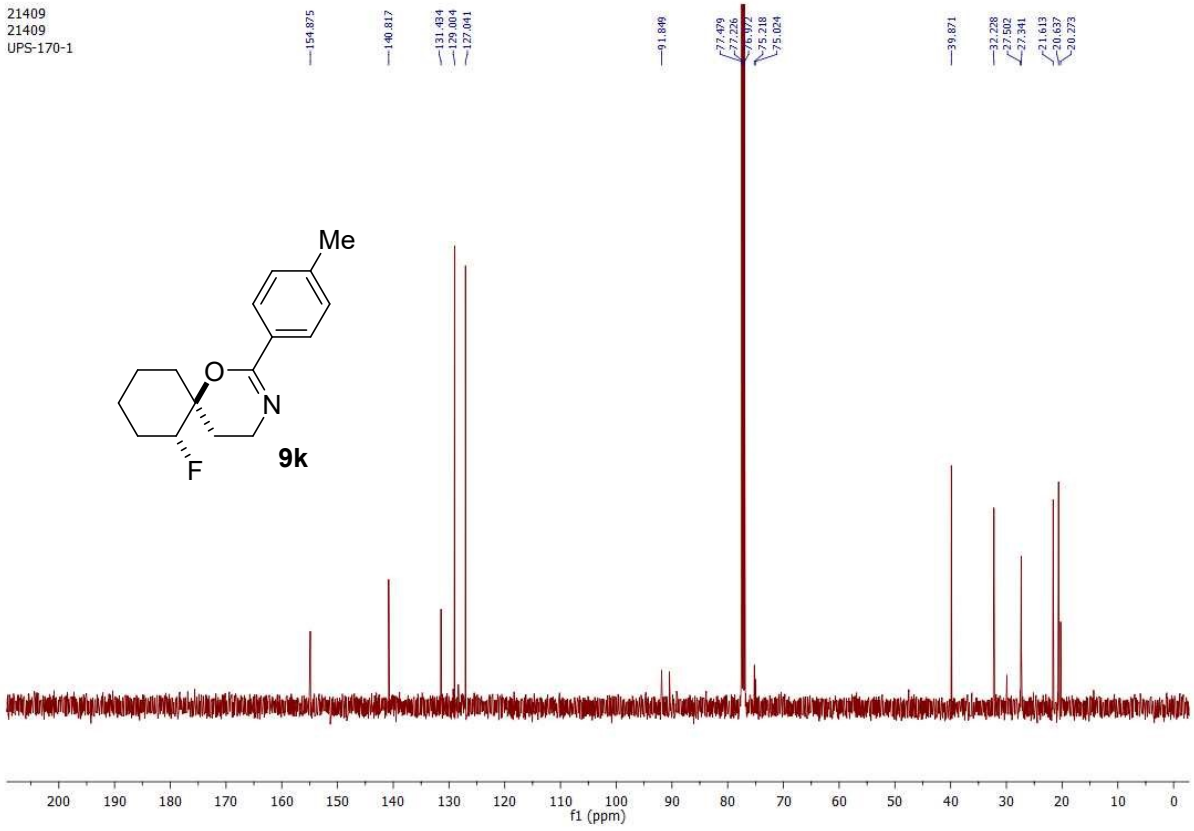
O2N
O
N
F

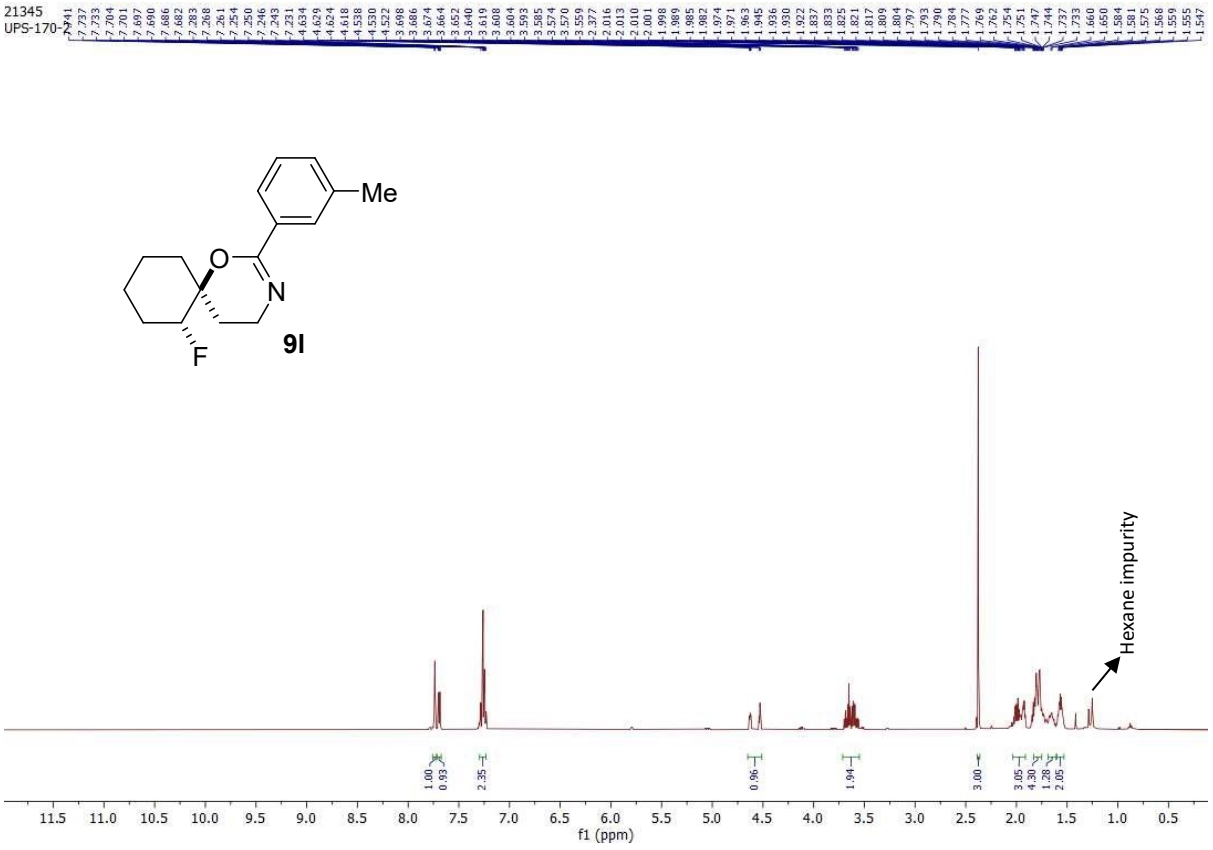
9j

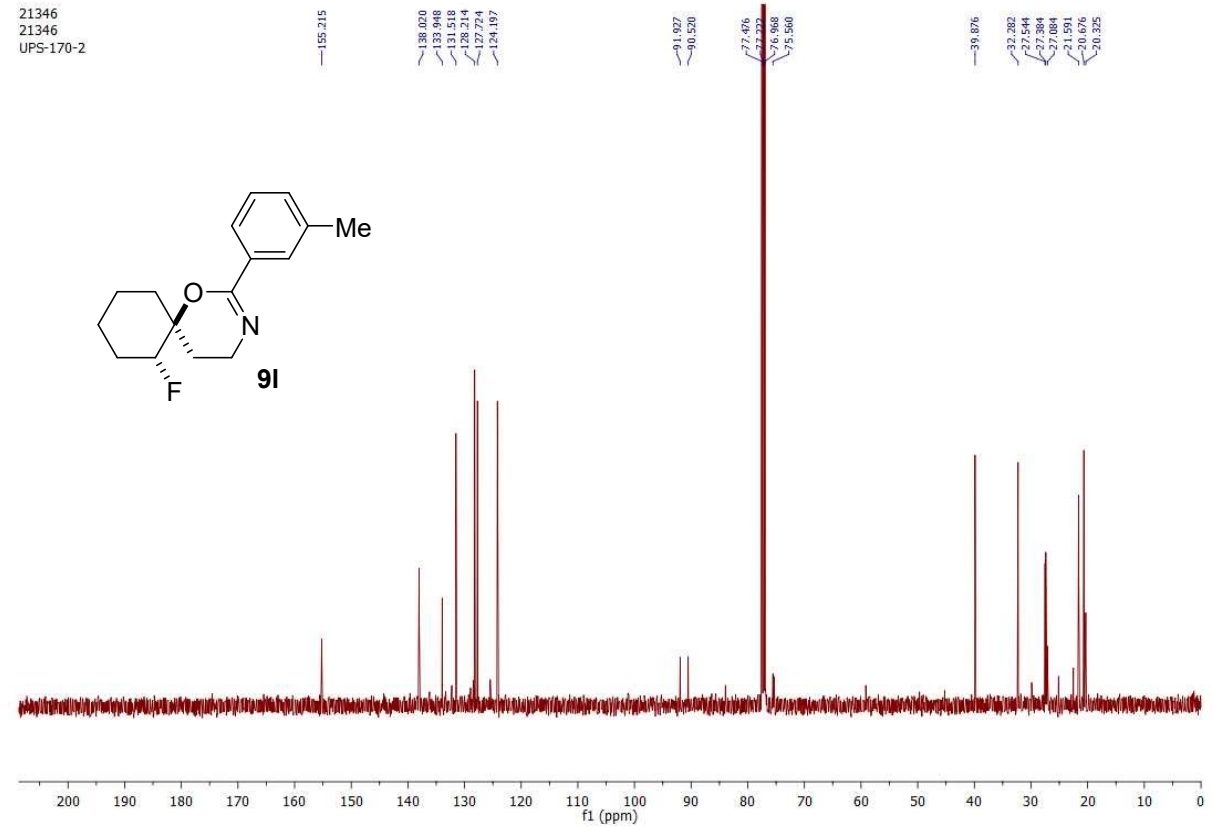
21408
UPS-170-1

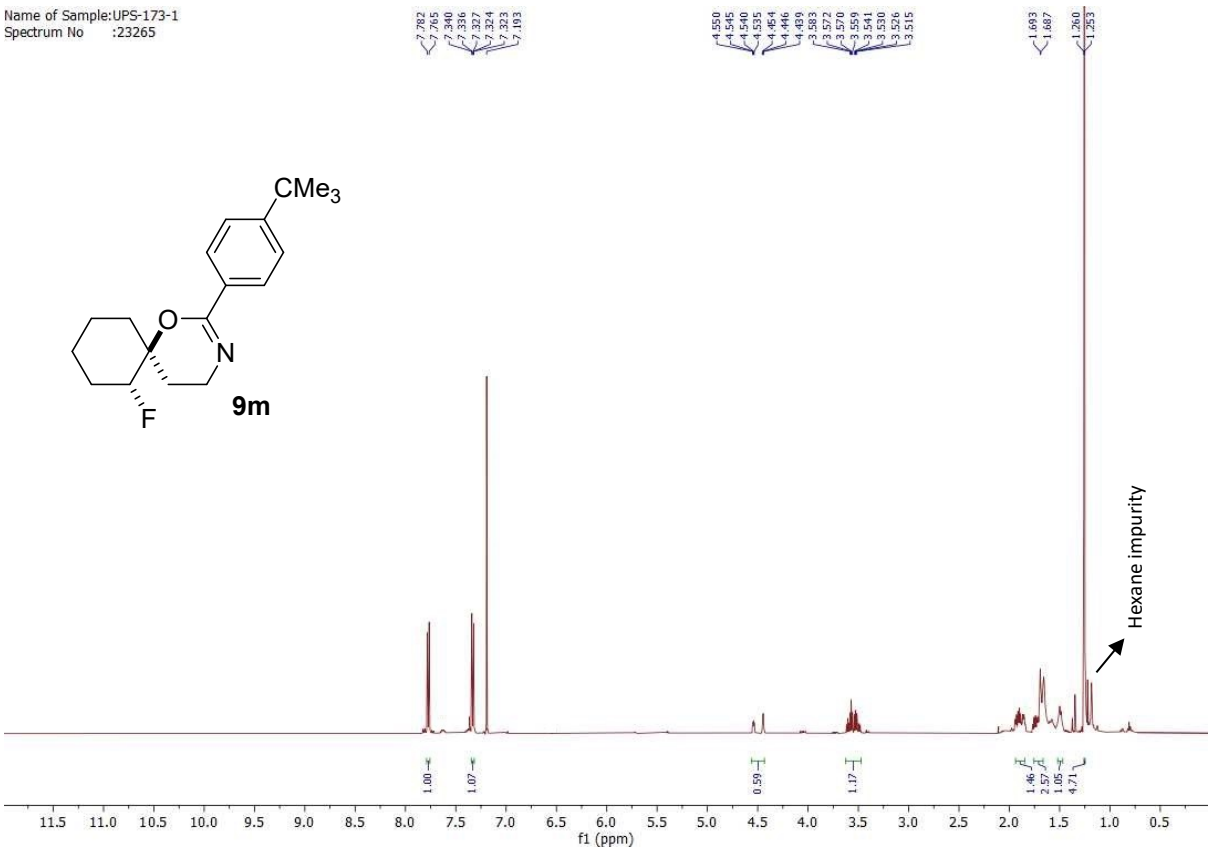


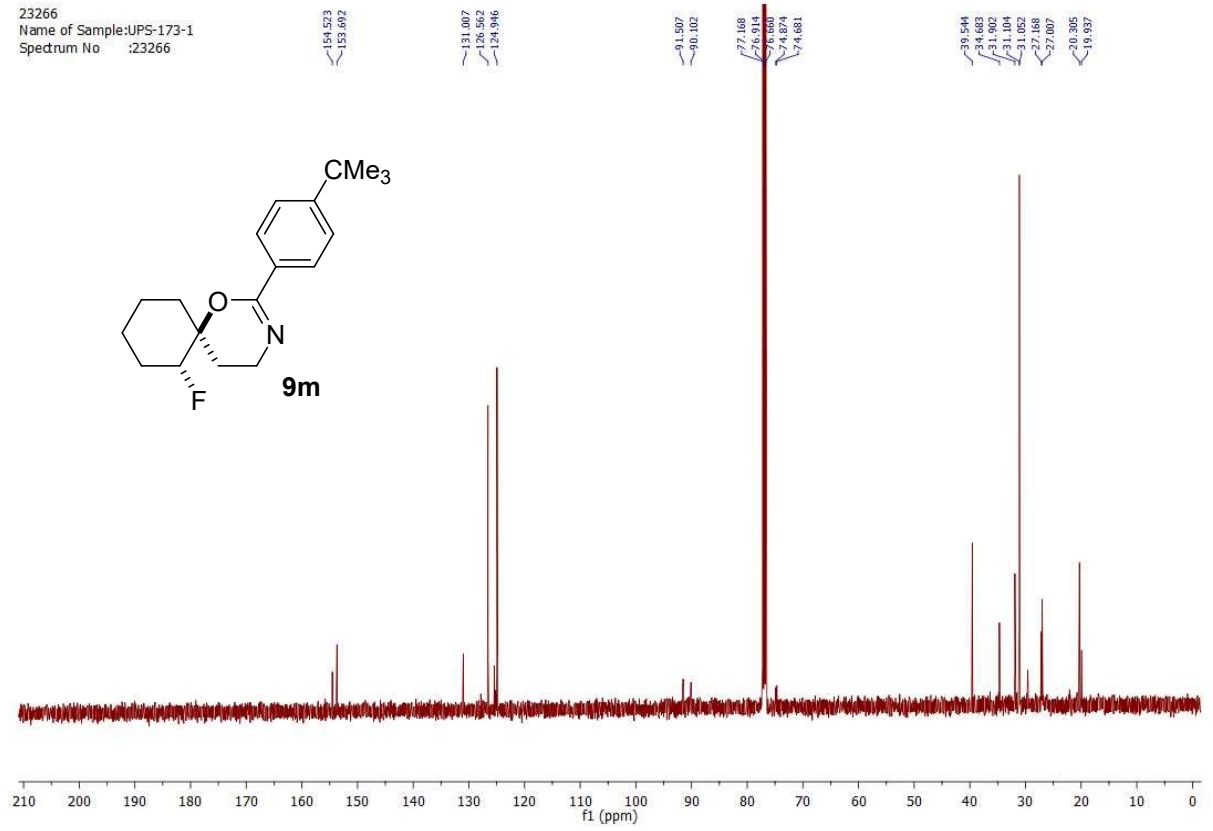
21409
21409
UPS-170-1

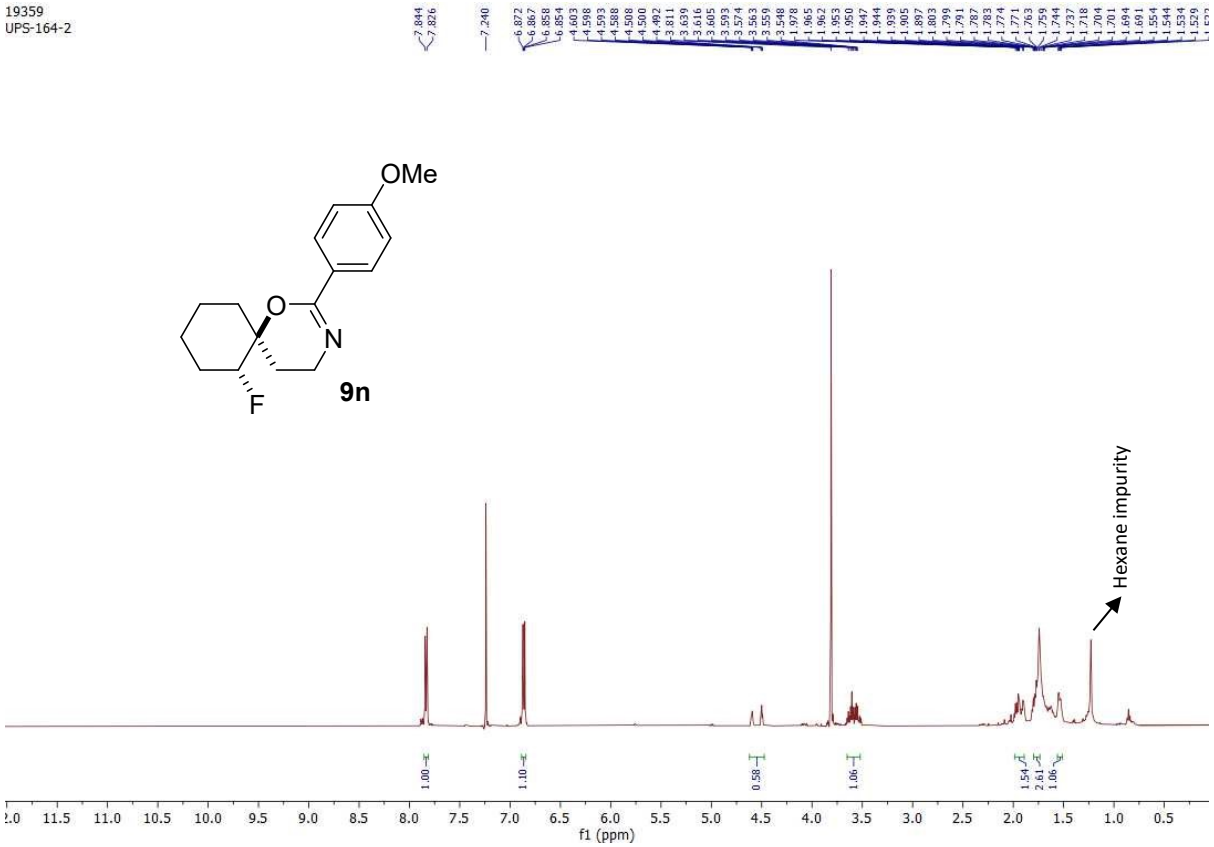


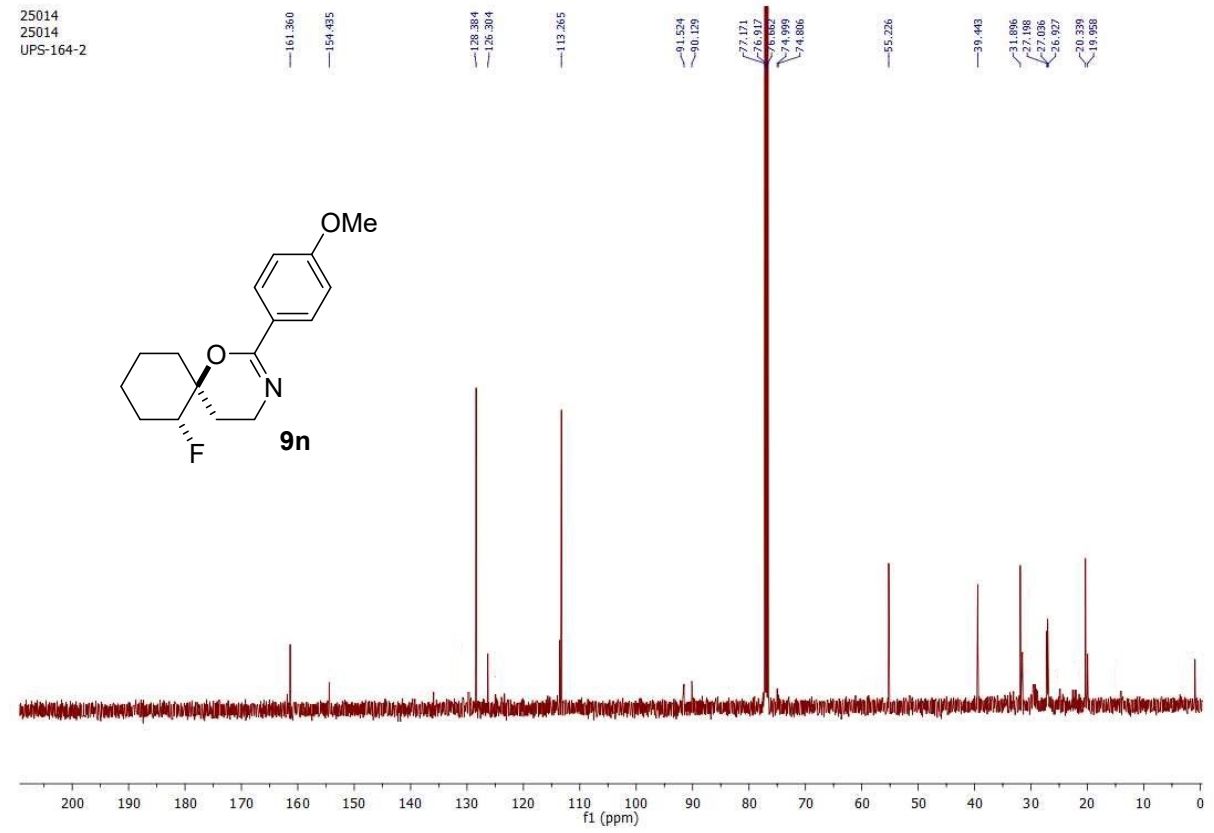

21345
UPS-170-2
Me
O
N
F
9l
Hexane impurity
f1 (ppm)

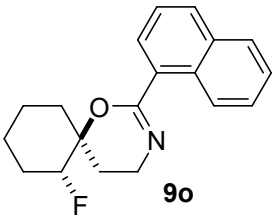

21346
21346
UPS-170-2
Me
O
N
F
9l
f1 (ppm)


Name of Sample:UPS-173-1
Spectrum No :23265
CMe3
O
N
F
9m
Hexane impurity
f1 (ppm)

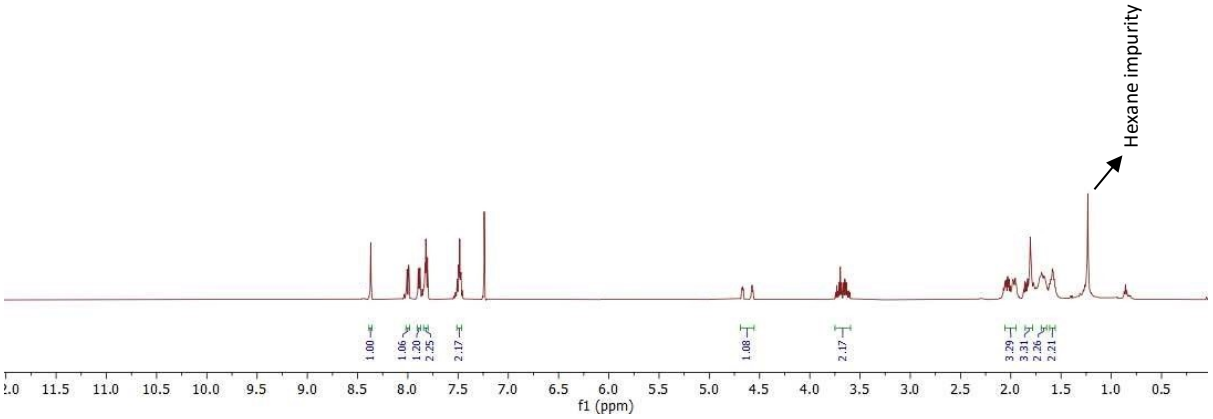

23266
Name of Sample:UPS-173-1
Spectrum No :23266
CMe3
O
N
F
9m
f1 (ppm)

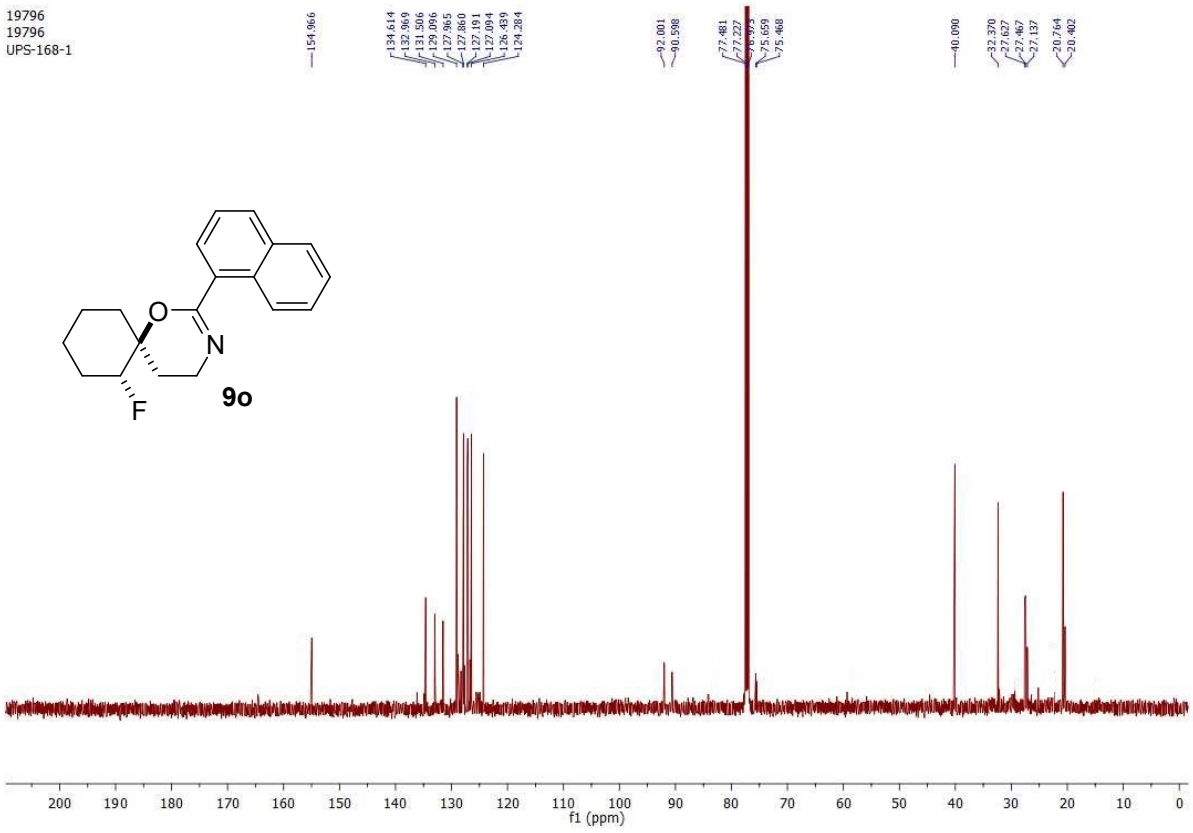

19359
UPS-164-2
OMe
O
N
F
9n
Hexane impurity
f1 (ppm)


25014
25014
UPS-164-2
OMe
O
N
F
9n
f1 (ppm)


O
N
F

9o


Hexane impurity
1.00
1.06
1.20
2.25
2.17
1.08
2.17
3.29
3.31
2.26
2.21
f1 (ppm)


19796
19796
UPS-168-1
154.966
134.614
132.969
131.506
129.096
127.965
127.860
127.191
127.094
126.439
124.284
92.001
90.598
77.481
77.227
76.973
75.659
75.468
40.090
32.370
27.627
27.467
27.137
20.764
20.402
f1 (ppm)

O
N
F

9o