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Supporting Information

Catalytic Intramolecular Aminofluorination, Oxyfluorination, and Carbofluorination with a Stable and Versatile Hypervalent Fluoroiodine Reagent**

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General Information

Hypervalent iodine **1**[1] and alkenes[2] were prepared according to literature procedures. All the other chemicals were obtained from commercial sources and used as received. ^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra were recorded in $CDCl_3$ (internal standard: 7.26 ppm, ^{1}H; 77.0 ppm, ^{13}C) using 400 MHz spectrometers. High resolution mass data (HRMS) were obtained using ESI technique. For column chromatography, silica gel (35-70 microns) was used. Unless otherwise stated the reactions were conducted under argon.

Experimental Procedures and Spectral Data

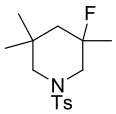
Method A for catalytic fluorocyclization

Fluoroiodine reagent **1** (92.0 mg, 0.33 mmol), the corresponding alkenes **2** (0.3 mmol), $Zn(BF_4)_2XH_2O$ (3.6 mg, 0.015 mmol) were mixed in DCM (0.5 mL). This reaction mixture was stirred at room temperature. After completion of the reaction, the resulting mixture was concentrated and purified by column chromatography on silca gel.

Method B for catalytic fluorocyclization

Fluoroiodine reagent **1** (126.0 mg, 0.45 mmol), the corresponding alkenes **2** (0.3 mmol), $Cu(MeCN)_4BF_4$ (9.4 mg, 0.03 mmol) were mixed in DCM (0.5 mL). This reaction mixture was stirred at 40 ^{o}C. After completion of the reaction, the resulting mixture was concentrated and purified by column chromatography on silca gel.

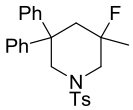
3-Fluoro-3,5,5-trimethyl-1-tosylpiperidine (3a) [3]



This compound was prepared according to method A. Compound **3a** was isolated as a white solid by chromatography using pentane:Et_2O (5:1) eluent system (67.4 mg, 75%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.90 (s, 3H); 1.08-1.23 (m, 4H); 1.30 (d, *J* = 20.6 Hz, 3H); 1.66-1.79 (m, 1H); 2.12 (d, *J* = 11.5 Hz, 1H); 2.32 (dd, $J_{H\text{-}F}$ = 31.6 Hz, $J_{H\text{-}H}$ = 12.6 Hz, 1H); 2.42 (s, 3H); 3.31 (dt, *J* = 11.6, 1.9 Hz, 1H); 3.67-3.76 (m, 1H); 7.31 (d, *J* = 8.2 Hz, 2H); 7.65 (d, *J* = 8.4 Hz, 2H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -145.00 (br); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 21.5; 25.6 (d, J_{CF} = 14.7 Hz); 25.7 (d, J_{CF} = 15.4 Hz); 29.0; 31.1; 46.8 (d, J_{CF} = 21.0 Hz); 53.9 (d, J_{CF} = 23.5 Hz); 56.5; 91.2 (d, J_{CF} = 176.8 Hz); 127.5; 129.6; 134.0; 143.4; HRMS (ESI): *m*/*z* calcd. for $[C_{15}H_{22}FNNaO_2S]^+$ 322.1247, found 322.1247; Mp: 130-131 ^{o}C.

The spectral data is in agreement with the literature values of ref [3].

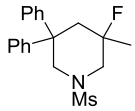
5-Fluoro-5-methyl-3,3-diphenyl-1-tosylpiperidine (3b) [3]



This compound was prepared according to method A. Compound **3b** was isolated as a white solid by chromatography using pentane:Et_2O (5:1) eluent system (92.8 mg, 73%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.17 (d, *J* = 22.8 Hz, 3H); 2.45 (s, 3H); 2.49-2.63 (m, 1H); 2.70 (t, *J* = 13.2 Hz, 1H); 3.02 (t, *J* = 10.3 Hz, 1H); 3.08-3.20 (m, 1H); 3.43 (d, *J* = 12.7 Hz, 1H); 3.71 (d, *J* = 12.6 Hz, 1H); 7.13-7.25 (m, 2H); 7.26-7.46 (m, 10H); 7.70 (d, *J* = 8.3 Hz, 2H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -137.98 (br); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 21.5; 25.1 (d, J_{CF} = 24.2 Hz); 44.9 (d, J_{CF} = 20.7 Hz); 45.5 (d, J_{CF} = 6.0 Hz); 53.8; 54.6 (d, J_{CF} = 29.4 Hz); 91.5 (d, J_{CF} = 174.6 Hz); 126.2; 126.4; 126.8; 127.1; 127.7; 128.3; 128.4; 129.8; 132.3; 143.9; 145.1; 145.4; HRMS (ESI): *m*/*z* calcd. for $[C_{25}H_{26}FNNaO_2S]^+$ 446.1560, found 446.1571; Mp: 160-162 ^{o}C.

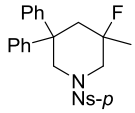
The spectral data is in agreement with the literature values of ref [3].

5-Fluoro-5-methyl-1-(methylsulfonyl)-3,3-diphenylpiperidine (3c)



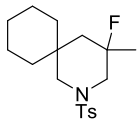
This compound was prepared according to method A. Compound **3c** was isolated as a white solid by chromatography using pentane:Et_2O (3:1) eluent system (85.5 mg, 82%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.28 (d, *J* = 22.1 Hz, 3H); 2.50 (dd, *J* = 23.8, 14.1 Hz, 1H); 2.67 (s, 3H); 2.90-3.06 (m, 1H); 3.25 (dd, *J* = 20.4, 12.5 Hz, 1H); 3.43 (t, *J* = 11.4 Hz, 1H); 3.78 (d, *J* = 13.0 Hz, 1 H); 4.08 (d, *J* = 13.1 Hz, 1H); 7.16-7.45 (m, 10H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -139.61 (br); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 25.0 (d, J_{CF} = 23.5 Hz); 35.9; 44.6 (d, J_{CF} = 21.0 Hz); 45.6 (d, J_{CF} = 4.4 Hz); 53.6; 54.4 (d, J_{CF} = 27.5 Hz); 91.4 (d, J_{CF} = 175.2 Hz); 126.3; 126.5; 126.6; 127.1; 128.2; 128.5; 144.7; 145.4; HRMS (ESI): *m*/*z* calcd. for $[C_{19}H_{22}FNNaO_2S]^+$ 370.1247, found 370.1255; Mp: 153-154 ^{o}C.

5-Fluoro-5-methyl-3,3-diphenyl-1-(*p*-nitro-benzenesulfonyl)piperidine (3d)



This compound was prepared according to method A. Compound **3d** was isolated as a white solid by chromatography using pentane:Et_2O (3:1) eluent system (114.4 mg, 84%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.22 (d, *J* = 22.1 Hz, 3H); 2.47 (dd, *J* = 21.5, 14.1 Hz, 1H); 2.73-2.87 (m, 1H); 3.02-3.27 (m, 2H); 3.56-3.80 (m, 2H); 7.18-7.37 (m, 10H); 7.95 (d, *J* = 8.8 Hz, 2H); 8.35 (d, *J* = 8.9 Hz, 2H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -139.39 (br); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 25.1 (d, J_{CF} = 23.5 Hz); 44.6 (d, J_{CF} = 20.5 Hz); 45.6 (d, J_{CF} = 5.2 Hz); 53.7; 54.4 (d, J_{CF} = 28.7 Hz); 91.1 (d, J_{CF} = 176.2 Hz); 124.4; 126.4; 126.7; 126.8; 126.9; 128.4; 128.6; 128.9; 141.7; 144.7; 145.0; 150.2; HRMS (ESI): *m*/*z* calcd. for $[C_{24}H_{23}FN_2NaO_4S]^+$ 477.1255, found 477.1247; Mp: 180-182 ^{o}C.

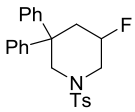
5-Cyclohexyl-3-fluoro-3-methyl-1-tosylpiperidine (3e)



This compound was prepared according to method A. Compound **3e** was isolated as a white solid by chromatography using pentane:Et_2O (5:1)

eluent system (85.5 mg, 84%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.97-1.15 (m, 1H); 1.15-1.23 (m, 2H); 1.30 (d, *J* = 20.5 Hz, 3H); 1.35-1.63 (m, 7H); 1.69-1.80 (m, 1H); 1.84-1.95 (m, 1H); 2.06 (d, *J* = 11.9 Hz, 1H); 2.29-2.47 (m, 4H); 3.57-3.67 (m, 1H); 3.68-3.79 (m, 1H); 7.32 (d, *J* = 8.3 Hz, 2H); 7.67 (d, *J* = 8.3 Hz, 2H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -143.41 (m); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 21.4; 21.5; 25.9 (d, J_{CF} = 24.3 Hz); 26.1; 33.1; 33.2; 33.7; 37.7; 45.4 (d, J_{CF} = 20.8 Hz); 53.8; 54.5 (d, J_{CF} = 24.3 Hz); 91.5 (d, J_{CF} = 176.2 Hz); 127.5; 129.6; 134.2; 143.4; HRMS (ESI): *m*/*z* calcd. for $[C_{18}H_{26}FNNaO_2S]^+$ 362.1560, found 362.1569; Mp: 114-115 ^{o}C.

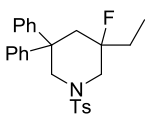
5-Fluoro-3,3-diphenyl-1-tosylpiperidine (3f)[3]



This compound was prepared according to method A. Compound **3f** was isolated as a white solid by chromatography using pentane:Et_2O (5:1) eluent system (87.2 mg, 71%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 2.14-2.25 (m, 1H); 2.28-2.38 (m, 1H); 2.39-2.55 (m, 4H); 2.91-3.07 (m, 1H); 3.99-4.15 (m, 1H); 4.43-4.70 (m, 2H); 7.13-7.44 (m, 10H); 7.50 (d, *J* = 7.8 Hz, 2H); 7.68 (d, *J* = 8.1 Hz, 2H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -185.46 (d, *J* = 47.9 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 21.5; 41.0 (d, J_{CF} = 18.5 Hz); 46.5 (d, J_{CF} = 11.0 Hz); 49.9 (d, J_{CF} = 31.2 Hz); 53.8; 85.6 (d, J_{CF} = 173.9 Hz); 126.4; 126.5; 126.9; 127.69; 127.73; 128.58; 128.66; 129.9; 132.1; 143.1; 144.0; 145.4; HRMS (ESI): *m*/*z* calcd. for $[C_{24}H_{24}FNNaO_2S]^+$ 432.1404, found 432.1398; Mp: 167-168 ^{o}C.

The spectral data is in agreement with the literature values of ref [3].

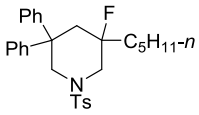
5-Ethyl -5-fluoro-3,3-diphenyl-1-tosylpiperidine (3g)



This compound was prepared according to method A. Compound **3g** was isolated as a white solid by chromatography using pentane:Et_2O (5:1) eluent system (94.5 mg, 72%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.82 (t, *J* =

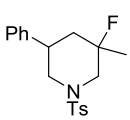
7.4 Hz, 3H); 1.34-1.54 (m, 2H); 2.36-2.57 (m, 4H); 2.61-2.75 (m, 1H); 2.98-3.17 (m, 2H); 3.47-3.66 (m, 2H); 7.13-7.24 (m, 2H); 7.25-7.43 (m, 10H); 7.69 (d, J = 8.3 Hz, 2H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -151.29 (m); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 6.8 (d, J_{CF} = 5.1 Hz); 21.5; 30.4 (d, J_{CF} = 22.2 Hz); 42.8 (d, J_{CF} = 20.6 Hz); 45.4 (d, J_{CF} = 5.9 Hz); 52.7 (d, J_{CF} = 28.7 Hz); 54.1; 93.0 (d, J_{CF} = 177.8 Hz); 126.2; 126.4; 126.9; 127.1; 127.7; 128.2; 128.4; 129.8; 132.4; 143.9; 145.47; 145.53; HRMS (ESI): *m*/*z* calcd. for $[C_{26}H_{28}FNNaO_2S]^+$ 460.1717, found 460.1716; Mp: 168-169 ^oC.

5-Fluoro-5-pentyl-3,3-diphenyl-1-tosylpiperidine (3h)



This compound was prepared according to method A. Compound **3h** was isolated as a white solid by chromatography using pentane:Et_2O (5:1) eluent system (89.1 mg, 62%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.85 (t, J = 7.3 Hz, 3H); 0.95-1.52 (m, 8H); 2.39-2.73 (m, 5H); 2.95-3.22 (m, 2H); 3.43-3.71 (m, 2H); 7.12-7.44 (m, 12H); 7.70 (d, J = 8.2 Hz, 2H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -148.59 (br); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 13.9; 21.5; 22.0 (d, J_{CF} = 3.7 Hz); 22.3; 31.7; 37.6 (d, J_{CF} = 22.1 Hz); 42.7 (d, J_{CF} = 20.5 Hz); 45.4 (d, J_{CF} = 5.9 Hz); 53.4 (d, J_{CF} = 29.1 Hz); 54.1; 93.0 (d, J_{CF} = 177.4 Hz); 126.2; 126.4; 126.9; 127.1; 127.7; 128.2; 128.4; 129.8; 132.4; 143.9; 145.4; 145.6; HRMS (ESI): *m*/*z* calcd. for $[C_{29}H_{34}FNNaO_2S]^+$ 502.2186, found 502.2172; Mp: 190-191 ^oC.

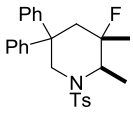
5-Fluoro-5-methyl-3-phenyl-1-tosylpiperidine (3i)



This compound was prepared according to method A. Compound **3i** was isolated as a white solid by chromatography using pentane:Et_2O (5:1) eluent system (64.6 mg, 62%). Spectral data for the separated major diastereomer: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.58 (d, J = 23.2 Hz, 3H); 1.65-1.84 (m, 1H); 1.99-2.14 (m, 2H); 2.28 (dd, J = 10.7, 6.8 Hz, 1H); 2.36 (s, 3H); 2.84-3.02 (m, 1H); 3.72-3.78 (m, 1H); 3.79-3.89 (m, 1H); 7.07-7.14 (m, 2H); 7.15-7.30 (m, 5H);

7.54 (d, J = 8.3 Hz, 2H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -138.50 (m); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 21.5; 23.4 (d, J_{CF} = 24.2 Hz); 40.2 (d, J_{CF} = 11.2 Hz); 42.2 (d, J_{CF} = 21.3 Hz); 52.5; 54.2 (d, J_{CF} = 34.6 Hz); 92.3 (d, J_{CF} = 172.0 Hz); 127.1; 127.4; 127.5; 128.8; 129.8; 133.0; 140.4; 143.8; HRMS (ESI): *m*/*z* calcd. for $[C_{19}H_{22}FNNaO_2S]^+$ 370.1247, found 370.1247; Mp: 154-155 ^oC.

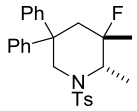
5-Fluoro-5,6-dimethyl-3,3-diphenyl-1-tosylpiperidine (3j)



This compound was prepared according to method A. Compound **3j** was isolated as a white solid by chromatography using pentane:Et_2O (5:1) eluent system (82.7 mg, 63%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.15 (d, J = 7.0 Hz, 3H); 1.35 (d, J = 21.2 Hz, 3H); 2.29-2.50 (m, 4H); 2.85-2.96 (m, 1H); 3.39 (d, J = 13.6 Hz, 1H); 3.94 (dq, $J_{H\text{-}F}$ = 13.3 $J_{H\text{-}H}$ = 7.1 Hz, 1H); 4.73 (dd, J = 13.7, 1.7 Hz, 1H); 7.10-7.24 (m, 8H); 7.26-7.32 (m, 2H); 7.40 (d, J = 7.8 Hz, 2H); 7.62 (d, J = 8.3 Hz, 2H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -139.81 (m); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 12.9 (d, J_{CF} = 8.1 Hz); 21.5; 25.4 (d, J_{CF} = 24.2 Hz); 40.0 (d, J_{CF} = 19.2 Hz); 45.3; 46.4; 56.3 (d, J_{CF} = 22.7 Hz); 94.0 (d, J_{CF} = 178.6 Hz); 125.6; 126.1; 126.5; 127.39; 127.42; 127.8; 128.6; 129.4; 136.6; 143.3; 144.9; 147.4; HRMS (ESI): *m*/*z* calcd. for $[C_{26}H_{28}FNNaO_2S]^+$ 460.1717, found 460.1722; Mp: 174-175 ^oC.

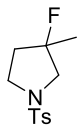
The stereochemistry was determined on the basis of the coupling constant between H(6) and F(5), which is 13.3 Hz. This coupling constant indicates a *cis* geometry for H(6) and F(5). In the case of *trans* geometry of H(6) and F(5) a larger coupling constant is expected (about 36 Hz, see ref [3] SI). Since H(6) and F(5) are *cis*, the two methyl groups Me(5) and Me(6) should also be in *cis* position. On the basis of this, the relative configuration of the Me(5) and Me(6) groups of **3k** was assigned as *trans*.

5-Fluoro-5,6-dimethyl-3,3-diphenyl-1-tosylpiperidine (3k)



This compound was prepared according to method A. Compound **3k** was isolated as a white solid by chromatography using pentane:Et_2O (5:1) eluent system (85.3 mg, 65%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.85 (dd, *J* = 6.8, 3.0 Hz, 3 H); 0.91 (d, *J* = 25.1 Hz, 3H); 2.42 (s, 3 H); 2.56-2.65 (m, 1H); 2.78 (t, *J* = 13.7 Hz, 1H); 2.99 (d, *J* = 13.4 Hz, 1H); 4.00 (q, *J* = 6.84 Hz, 1H); 4.78 (d, *J* = 13.6 Hz, 1H); 7.15-7.24 (m, 4H); 7.25-7.34 (m, 6H); 7.58 (d, *J* = 7.8 Hz, 2H); 7.74 (d, *J* = 8.3 Hz, 2H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -134.11 (m); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 8.3 (d, J_{CF} = 5.2 Hz); 21.5; 25.5 (d, J_{CF} = 24.4 Hz); 39.9 (d, J_{CF} = 22.9 Hz); 46.4; 46.6 (d, J_{CF} = 12.1 Hz); 56.2 (d, J_{CF} = 27.3 Hz); 93.6 (d, J_{CF} = 174.1 Hz); 126.1; 126.2; 126.7; 127.5; 127.6; 128.4; 128.7; 129.8; 136.8; 143.8; 144.4; 146.7; HRMS (ESI): *m*/*z* calcd. for $[C_{26}H_{28}FNNaO_2S]^+$ 460.1717, found 460.1714; Mp: 172-173 ^{o}C.

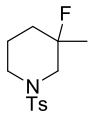
3-Fluoro-3-methyl-1-tosylpyrrolidine (3l) [4]



This compound was prepared according to method A. Compound **3l** was isolated as a white solid by chromatography using pentane:Et_2O (5:1) eluent system (54.0 mg, 70%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.45 (d, *J* = 20.5 Hz, 3H); 1.75-1.95 (m, 1H); 2.03-2.15 (m, 1H); 2.43 (s, 3H); 3.23-3.40 (m, 2H); 3.43-3.61 (m, 2H); 7.32 (d, *J* = 8.0 Hz, 2H); 7.71 (d, *J* = 8.4 Hz, 2H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -140.89 (m); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 21.5; 21.8 (d, J_{CF} = 21.5 Hz); 37.7 (d, J_{CF} = 23.6 Hz); 46.9; 58.3 (d, J_{CF} = 25.7 Hz); 100.3 (d, J_{CF} = 176.0 Hz); 127.5; 129.6; 133.7; 143.6; HRMS (ESI): *m*/*z* calcd. for $[C_{12}H_{16}FNNaO_2S]^+$ 280.0778, found 280.0788; Mp: 105-107 ^{o}C.

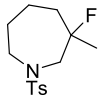
The spectral data is in agreement with the literature values of ref [4].

3-Fluoro-3-methyl-1-tosylpiperidine (3m)



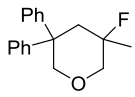
This compound was prepared according to method A. Compound **3m** was isolated as a white solid by chromatography using pentane:Et_2O (5:1) eluent system (59.4 mg, 73%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.32-1.54 (m, 4H); 1.55-1.65 (m, 1H); 1.72-1.92 (m, 2H); 2.43 (s, 3H); 2.66-2.83 (m, 2H); 3.23-3.41 (m, 2H); 7.32 (d, *J* = 8.0 Hz, 2H); 7.65 (d, *J* = 8.3 Hz, 2H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -148.97 (br); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 21.2 (d, J_{CF} = 4.41 Hz); 21.5; 24.3 (d, J_{CF} = 23.5 Hz); 34.5 (d, J_{CF} = 22.7 Hz); 45.5; 53.9 (d, J_{CF} = 26.4 Hz); 90.6 (d, J_{CF} = 174.6 Hz); 127.6; 129.6; 133.7; 143.5; HRMS (ESI): *m*/*z* calcd. for $[C_{13}H_{18}FNNaO_2S]^+$ 294.0934, found 294.0949; Mp: 104-105 ^{o}C.

3-Fluoro-3-methyl-1-tosylazepane (3n)



This compound was prepared according to method A. Compound **3n** was isolated as a white solid by chromatography using pentane:Et_2O (5:1) eluent system (59.9 mg, 70%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.46 (d, *J* = 21.9 Hz, 3H); 1.51-2.01 (m, 6H); 2.42 (s, 3H); 2.86-3.00 (m, 1H); 3.15-3.28 (m, 1H); 3.42-3.56 (m, 2H); 7.30 (d, *J* = 8.2 Hz, 2H); 7.67 (d, *J* = 8.3 Hz, 2H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -139.15 (m); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 21.0 (d, J_{CF} = 5.2 Hz); 21.5; 26.1 (d, J_{CF} = 24.3 Hz); 30.0; 39.1 (d, J_{CF} = 23.5 Hz); 50.9; 57.5 (d, J_{CF} = 33.8 Hz); 96.9 (d, J_{CF} = 170.2 Hz); 127.1; 129.7; 136.0; 143.3; HRMS (ESI): *m*/*z* calcd. for $[C_{14}H_{20}FNNaO_2S]^+$ 308.1091, found 308.1096; Mp: 95-96 ^{o}C.

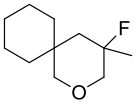
5-Fluoro-5-methyl-3,3-diphenyltetrahydro-2*H*-pyran (5a)



This compound was prepared according to method A. Compound **5a** was isolated as a white solid by chromatography using pentane:Et_2O (15:1) eluent system (50.3 mg, 62%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.22 (d, *J* = 21.7 Hz, 3H); 2.59 (dd, *J* = 26.4, 13.5 Hz, 1H); 2.82-2.96 (m, 1H); 3.57 (dd, *J* = 24.0, 12.0 Hz,

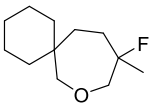
1H); 3.79-3.94 (m, 2H); 4.41 (dd, *J* = 12.3, 1.9 Hz, 1H); 7.15-7.45 (m, 10H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -145.57 (br); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 24.1 (d, J_{CF} = 24.2 Hz); 44.4 (d, J_{CF} = 19.8 Hz); 45.7 (d, J_{CF} = 2.2 Hz); 74.23 (d, J_{CF} = 25.7 Hz); 74.32; 90.8 (d, J_{CF} = 174.1 Hz); 125.9; 126.3; 127.0; 127.5; 128.0; 128.4; 145.6; 145.8; HRMS (ESI): *m*/*z* calcd. for $[C_{18}H_{19}FNaO]^+$ 293.1312, found 293.1301; Mp: 138-139 ^{o}C.

5-Cyclohexyl-3-fluoro-3-methyltetrahydro-2*H*-pyran (5b)



This compound was prepared according to method A. Compound **5b** was isolated as a liquid by chromatography using pentane:Et_2O (15:1) eluent system (36.3 mg, 65%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.12-1.17 (m, 2H); 1.23 (d, *J* = 20.1 Hz, 3H); 1.34-1.54 (m, 7H); 1.64-1.74 (m, 2H); 1.99-2.11 (m, 1H); 2.99 (d, *J* = 11.4 Hz, 1H); 3.23 (dd, *J* = 35.1, 12.5 Hz, 1H); 3.78-3.93 (m, 2H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -146.74 (m); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 21.5 (d, J_{CF} = 16.9 Hz); 24.1; 24.3; 26.4; 32.8 (d, J_{CF} = 5.2 Hz); 36.8; 43.9 (d, J_{CF} = 20.7 Hz); 74.6; 74.8; 76.0; 91.8 (d, J_{CF} = 172.4 Hz); HRMS (ESI): *m*/*z* calcd. for $[C_{11}H_{19}FNaO]^+$ 209.1312, found 209.1321.

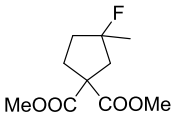
6-Cyclohexyl-3-fluoro-3-methyloxepane (5c)



This compound was prepared according to method A. Compound **5c** was isolated as a liquid by chromatography using pentane:Et_2O (15:1) eluent system (36.1 mg, 60%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.22-1.48 (m, 14H); 1.66-1.96 (m, 3H); 3.29 (d, *J* = 12.3 Hz, 1H); 3.44 (dd, *J* = 30.0, 13.6 Hz, 1H); 3.62 (d, *J* = 12.4 Hz, 1H); 3.89 (dd, *J* = 18.6, 13.5 Hz, 1H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -142.33 (m); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 21.6 (d, J_{CF} = 1.5 Hz); 24.9 (d, J_{CF} = 25.1 Hz); 26.4; 30.9; 32.9 (d, J_{CF} = 23.5 Hz); 33.6; 35.9; 37.5; 80.5; 80.7; 82.0;

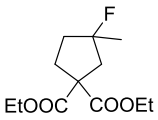
97.7 (d, J_{CF} = 170.2 Hz); HRMS (ESI): *m*/*z* calcd. for $[C_{12}H_{21}FNaO]^+$ 223.1469, found 223.1473.

Dimethyl 3-fluoro-3-methylcyclopentane-1,1-dicarboxylate (7a)



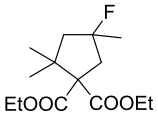
This compound was prepared according to method B. Compound **7a** was isolated as an oil by chromatography using pentane:Et_2O (10:1) eluent system (36.0 mg, 55%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.49 (d, *J* = 21.0 Hz, 3H); 1.66-1.86 (m, 1H); 2.00-2.14 (m, 1H); 2.17-2.27 (m, 1H); 2.30-2.46 (m, 1H); 2.56-2.74 (m, 2H); 3.72 (s, 3H); 3.74 (s, 3H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -135.03 (m); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 23.8 (d, J_{CF} = 26.4 Hz); 32.7; 38.4 (d, J_{CF} = 23.5 Hz); 46.4 (d, J_{CF} = 24.3 Hz); 52.9 (d, J_{CF} = 11.0 Hz); 59.6; 103.6 (d, J_{CF} = 171.0 Hz); 171.8; 172.8; HRMS (ESI): *m*/*z* calcd. for $[C_{10}H_{15}FNaO_4]^+$ 240.0847, found 240.0870.

Diethyl 3-fluoro-3-methylcyclopentane-1,1-dicarboxylate (7b)



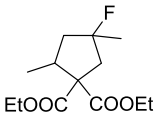
This compound was prepared according to method B. Compound **7b** was isolated as an oil by chromatography using pentane:Et_2O (10:1) eluent system (44.3 mg, 60%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.20-1.27 (m, 6H); 1.48 (d, *J* = 21.0 Hz, 3H); 1.66-1.85 (m, 1H); 2.00-2.13 (m, 1H); 2.14-2.24 (m, 1H); 2.30-2.46 (m, 1H); 2.55-2.71 (m, 2H); 4.13-4.23 (m, 4H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -134.83 (m); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 14.0; 23.9 (d, J_{CF} = 26.0 Hz); 32.6; 38.4 (d, J_{CF} = 24.2 Hz); 46.3 (d, J_{CF} = 24.6 Hz); 59.7; 61.6 (d, J_{CF} = 14.7 Hz); 103.6 (d, J_{CF} = 171.1 Hz); 171.3; 172.3; HRMS (ESI): *m*/*z* calcd. for $[C_{12}H_{19}FNaO_4]^+$ 269.1160, found 269.1162.

Diethyl 4-fluoro-2,2,4-trimethylcyclopentane-1,1-dicarboxylate (7c)



This compound was prepared according to method B. Compound **7c** was isolated as an oil by chromatography using pentane:Et_2O (10:1) eluent system (62.5 mg, 76%). ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.19 (s, 3H); 1.24-1.35 (m, 9H); 1.54 (d, *J* = 22.0 Hz, 3H); 2.02-2.29 (m, 2H); 2.47 (dd, *J* = 18.1, 16.0 Hz, 1H); 2.84-2.99 (m, 1H); 4.11-4.30 (m, 4H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -118.28 (m); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 14.0; 24.8; 25.6; 27.0; 27.3; 30.9; 45.5; 47.3 (d, J_{CF} = 27.2 Hz); 53.6 (d, J_{CF} = 22.9 Hz); 61.1 (d, J_{CF} = 24.2 Hz); 66.9; 101.6 (d, J_{CF} = 171.5 Hz); 170.1; 171.6; HRMS (ESI): *m*/*z* calcd. for $[C_{14}H_{23}FNaO_4]^+$ 297.1473, found 297.1479.

Diethyl 4-fluoro-2,4-dimethylcyclopentane-1,1-dicarboxylate (7d)



This compound was prepared according to method B. Compound **7d** was isolated as an oil by chromatography using pentane:Et_2O (10:1) eluent system (46.9 mg, 60%). The ^{1}H-NMR and ^{19}F-NMR data of major diastereomer: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.02 (d, *J* = 7.1 Hz, 3H); 1.20-1.29 (m, 6H); 1.49 (d, *J* = 21.1 Hz, 3H); 1.90-2.30 (m, 2H); 2.40-2.78 (m, 2H); 3.04-3.20 (m, 1H); 4.07-4.28 (m, 4H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -130.68 (m); the ^{1}H-NMR and ^{19}F-NMR data of minor diastereomer: ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.10 (d, *J* = 7.2 Hz, 1.5H); 1.20-1.29 (m, 3H); 1.42 (d, *J* = 21.7 Hz, 1.5H); 1.90-2.30 (m, 1H); 2.40-2.78 (m, 1H); 2.90-3.04 (m, 0.5H); 4.07-4.28 (m, 2H); ^{19}F-NMR (376 MHz, $CDCl_3$) δ -124.74 (m); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 13.96; 13.98; 14.06; 14.08; 15.9; 16.1; 24.1 (d, J_{CF} = 26.4 Hz); 27.0 (d, J_{CF} = 28.0 Hz); 38.8; 39.5; 46.3 (d, J_{CF} = 22.8 Hz); 46.4 (d, J_{CF} = 22.8 Hz); 46.5 (d, J_{CF} = 17.7 Hz); 47.2 (d, J_{CF} = 24.3 Hz); 61.27; 61.28 (d, J_{CF} = 18.7 Hz); 63.0; 101.4 (d, J_{CF} = 175.5 Hz); 102.3 (d, J_{CF} = 170.0 Hz); 170.0; 171.3; 171.6; 171.8; HRMS (ESI): *m*/*z* calcd. for $[C_{13}H_{21}FNaO_4]^+$ 283.1316, found 283.1325.

References

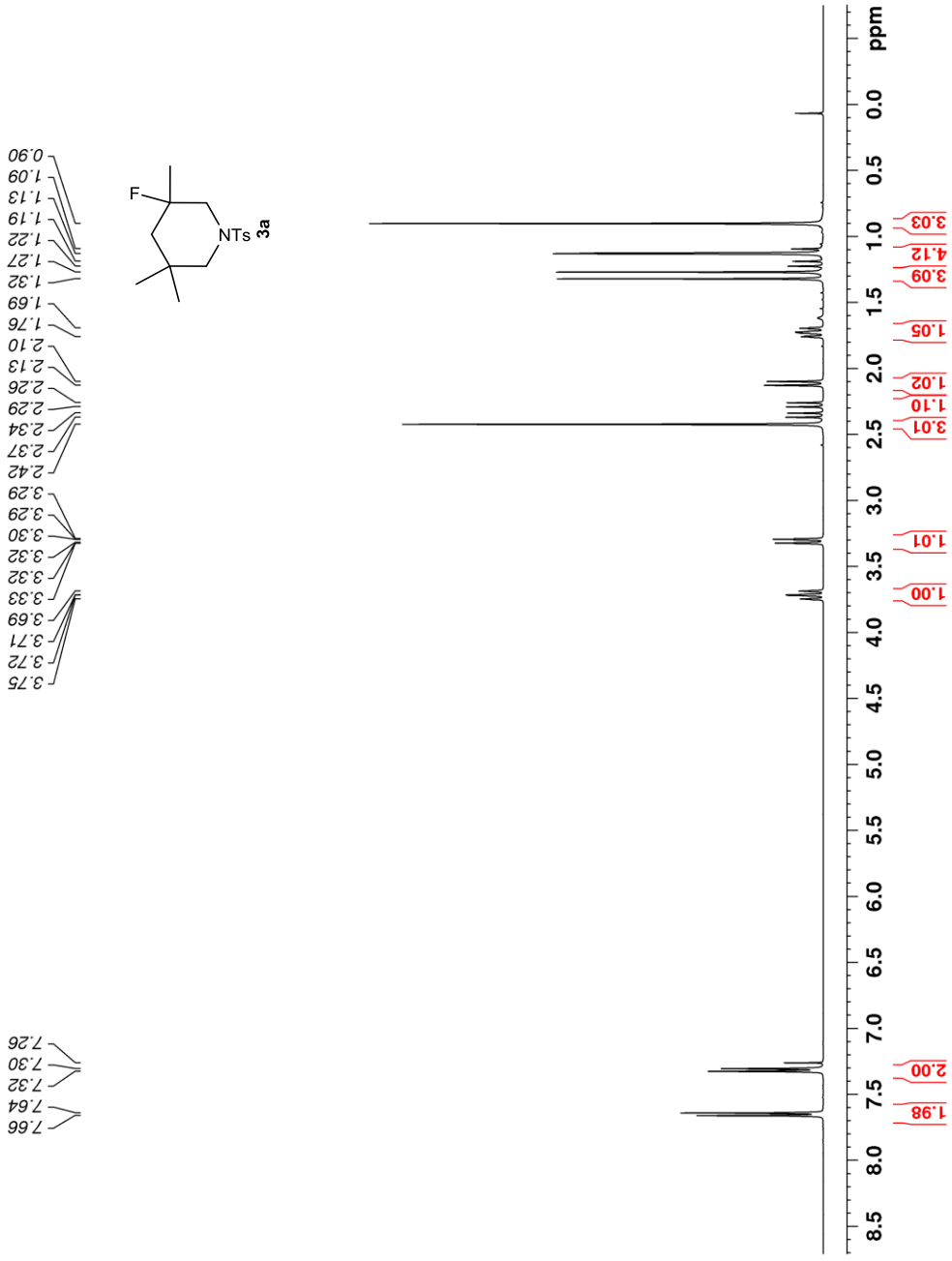
[1] a) G. C. Geary, E. G. Hope, K. Singh, A. M. Stuart, *Chem. Commun.* **2013**, *49*, 9263; b) V. Matoušek, E. Pietrasiak, R. Schwenk, A. Togni, *J. Org. Chem.* **2013**, *78*, 6763; c) P. Eisenberger, I. Kieltsch, R. Koller, K. Stanek, A. Togni, *Org. Synth.* **2011**, *88*, 168.

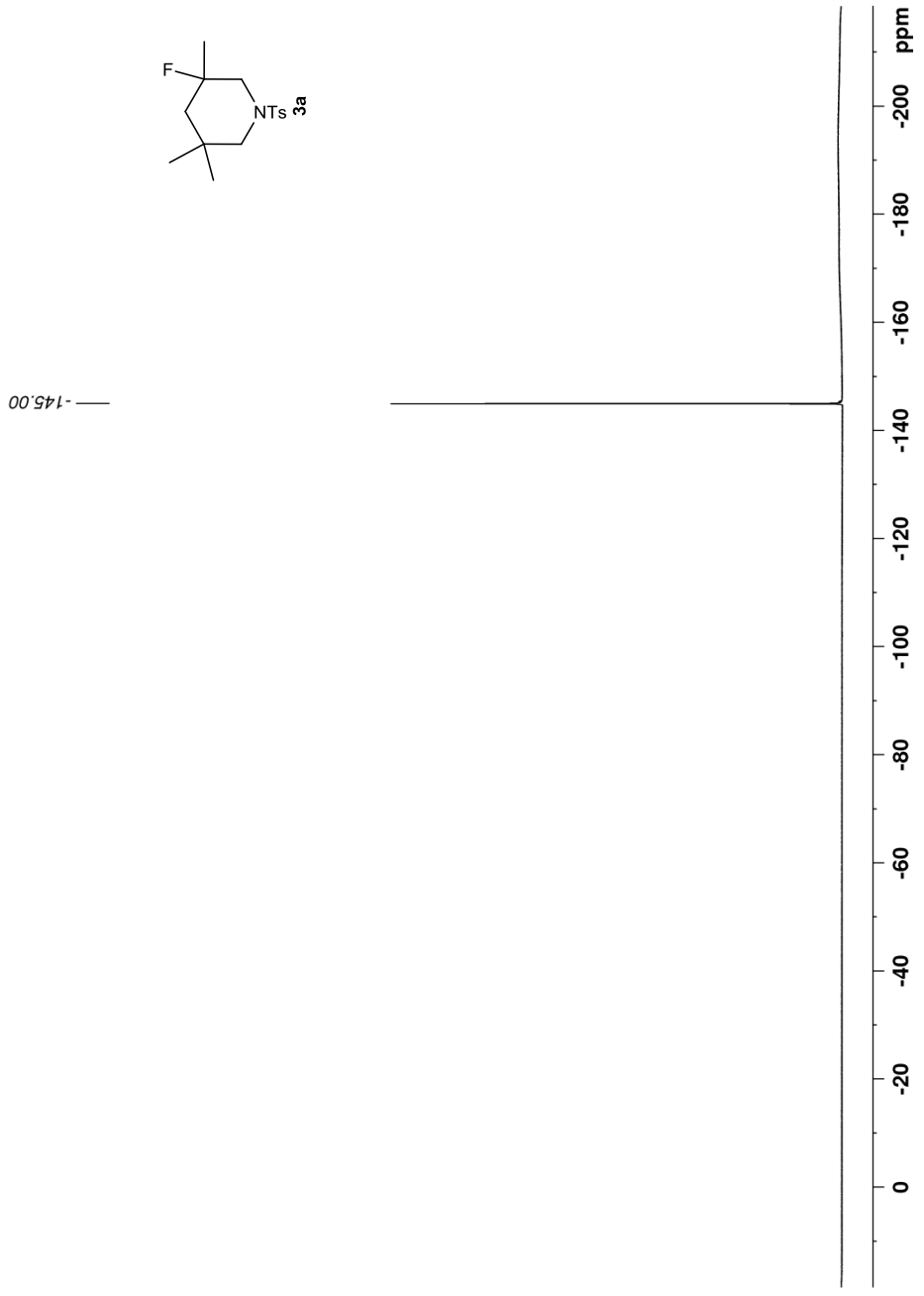
[2] a) W. Q. Kong, P. Feige, T. D. Haro, C. Nevado, *Angew. Chem. Int. Ed.* **2013**, *52*, 2469; b) A. P. Dobbs, S. J. J. Guesné, R. J. Parker, J. Skidmore, R. A. Stephenson, M. B. Hursthouse, *Org. Biomol. Chem.* **2010**, *8*, 1064; c) Y. Jeong, D-Y. Kim, Y. Choi, J-S. Ryu, *Org. Biomol. Chem.* **2011**, *9*, 374; d) J. Kempson, W. J. Pitts, J. Barbosa, J. Guo, O. Omotoso, A. Watson, K. Stebbins, G. C. Starling, J. H. Dodd, J. C. Barrish, R. Felix, K. Fischer, *Bioorg. Med. Chem. Lett.* **2005**, *15*, 1829.

[3] T. Wu, G. Y. Yin, G. S. Liu, *J. Am. Chem. Soc.* **2009**, *131*, 16354.

[4] J. Cui, Q. Jia, R-Z. Feng, S-S. Liu, T. He, C. Zhang, *Org. Lett.* **2014**, *16*, 1442.

NMR spectra of products 3a-3n, 5a-5c and 7a-7d

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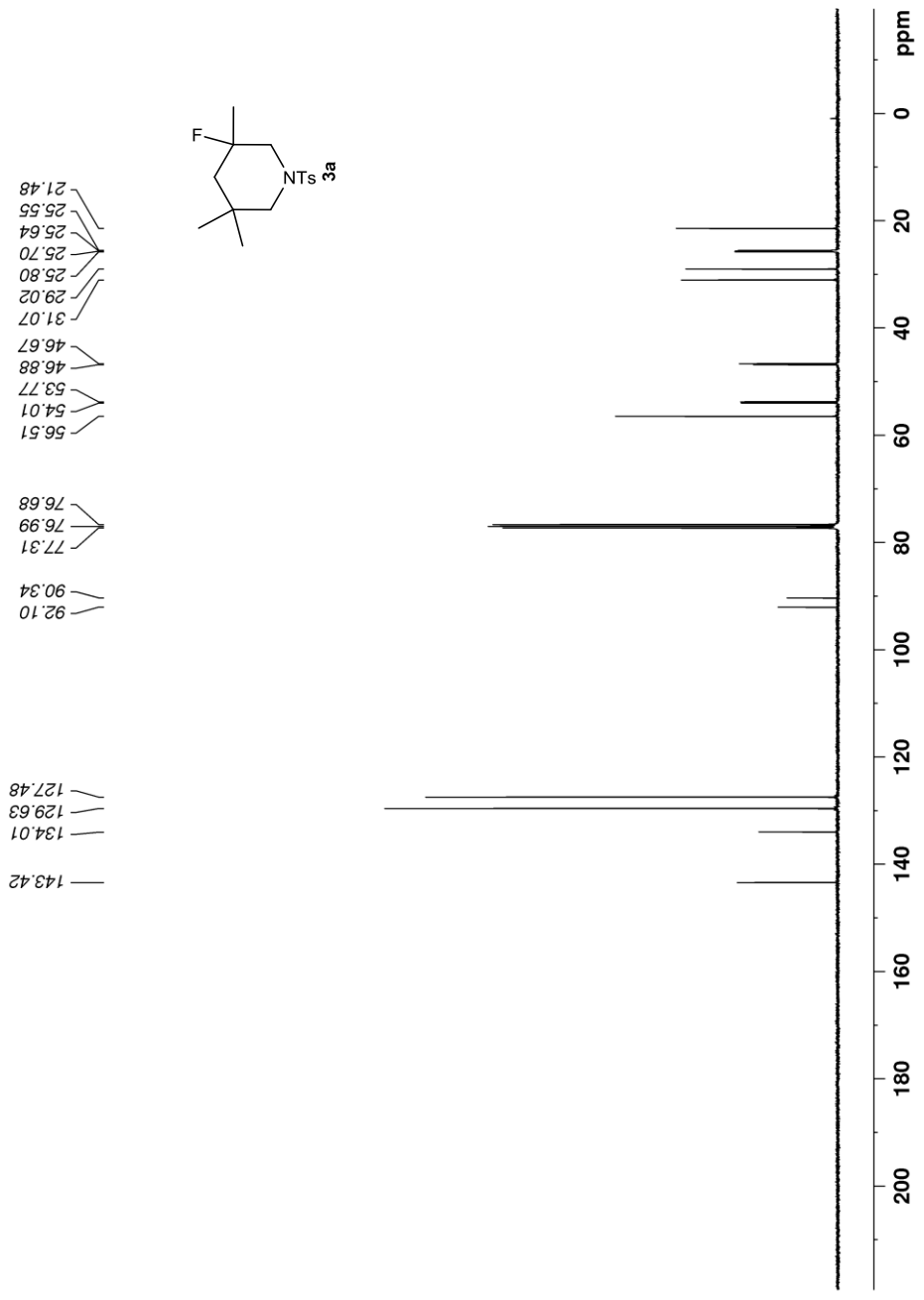
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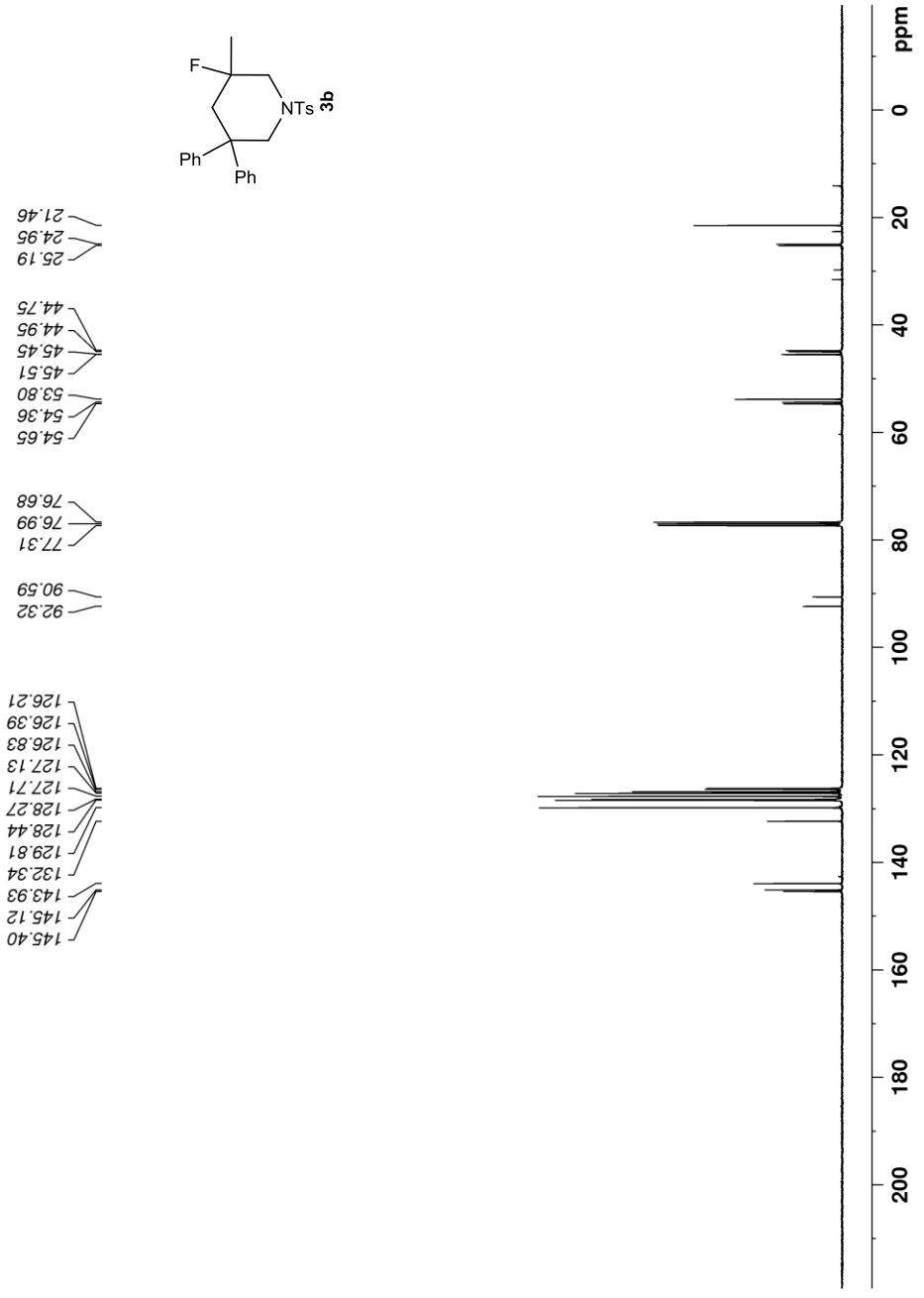
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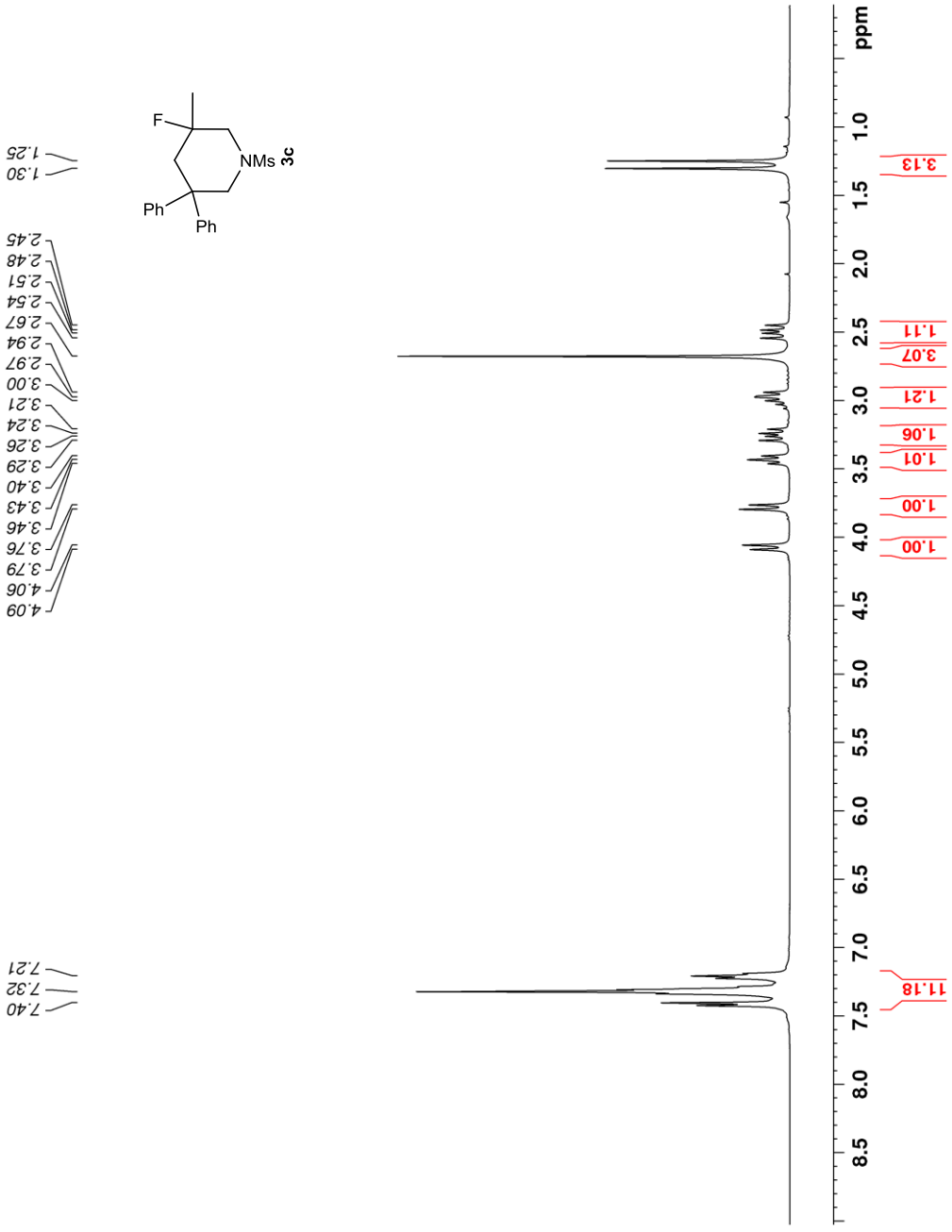
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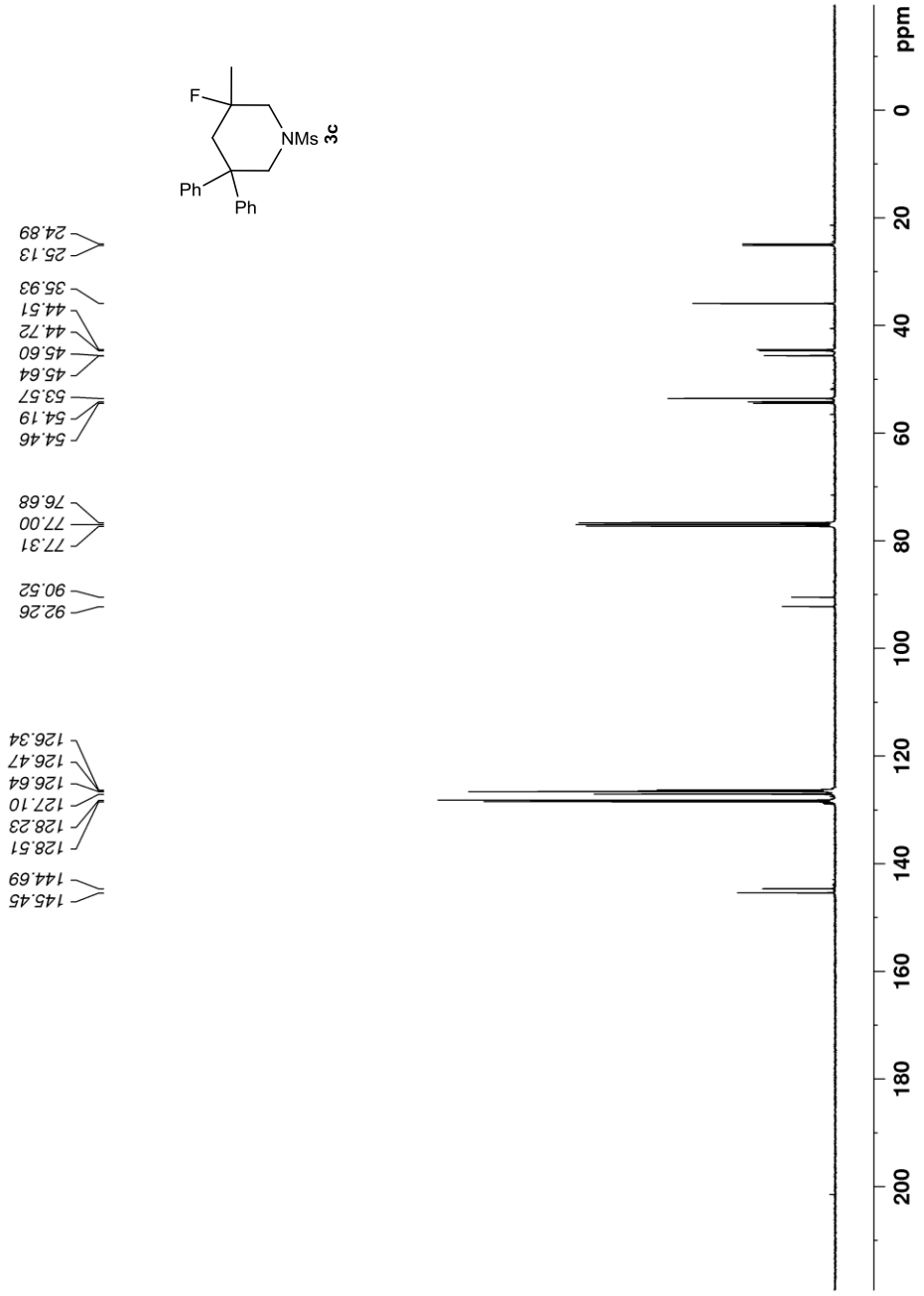
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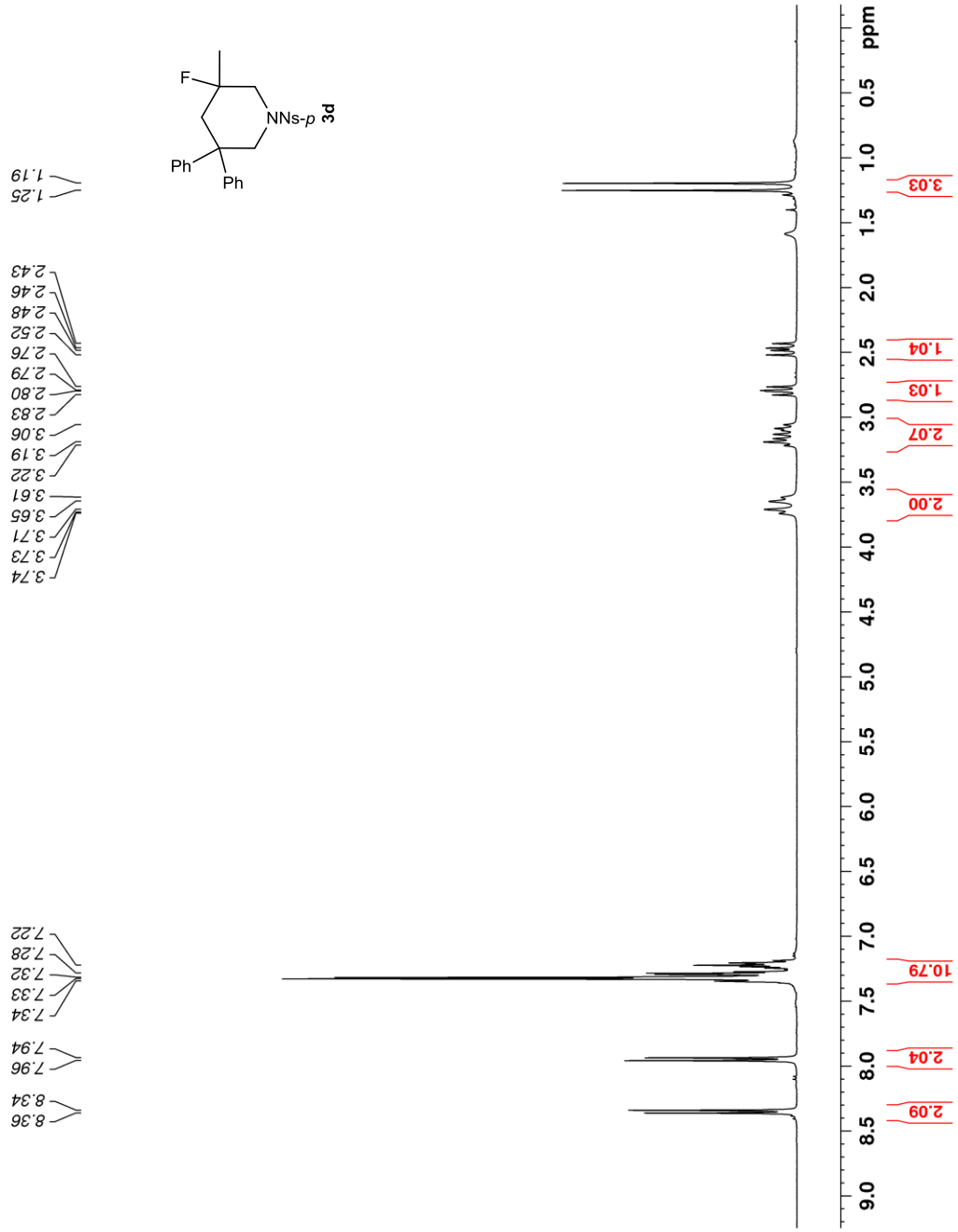
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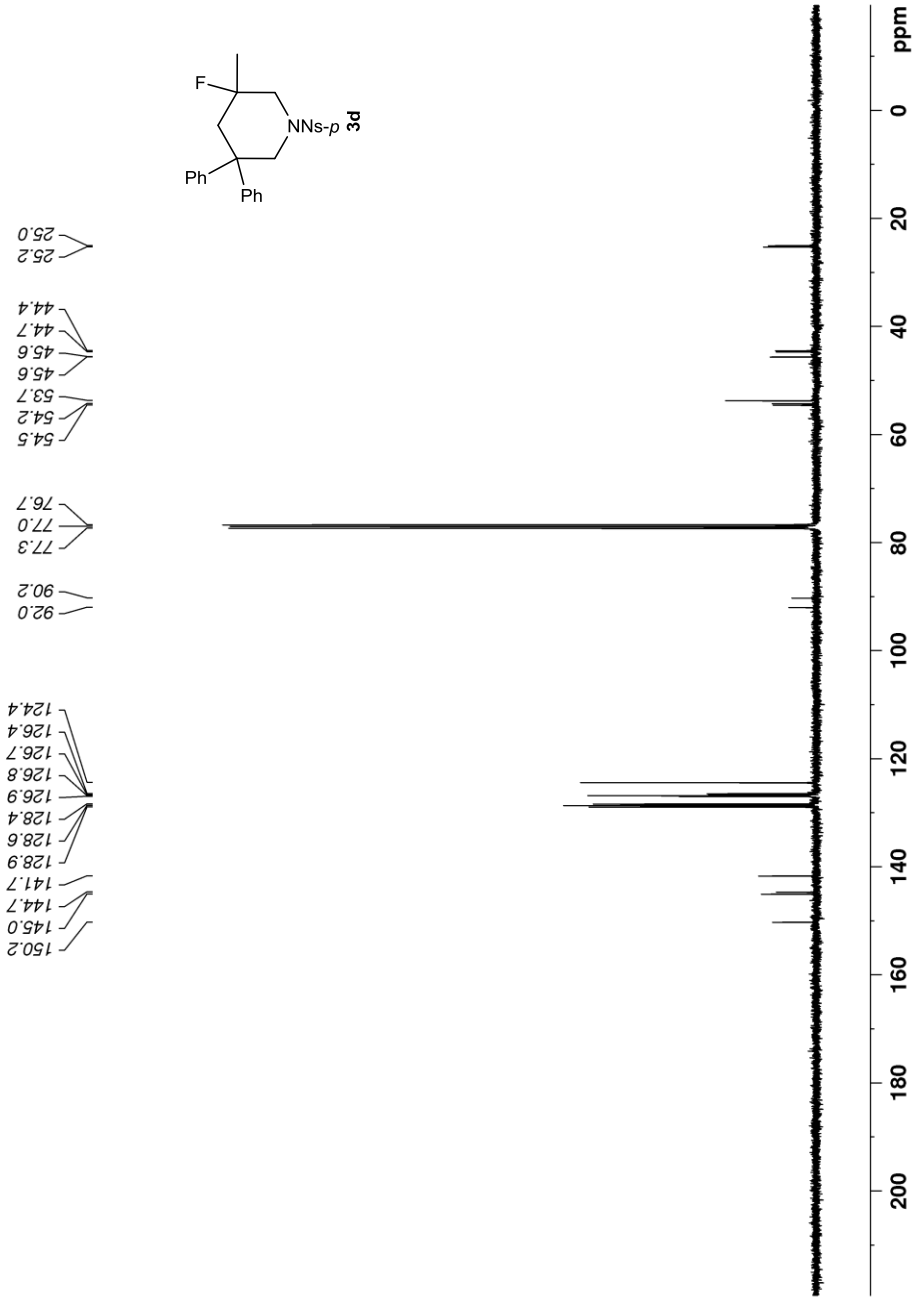
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126.9
128.4
128.6
128.9
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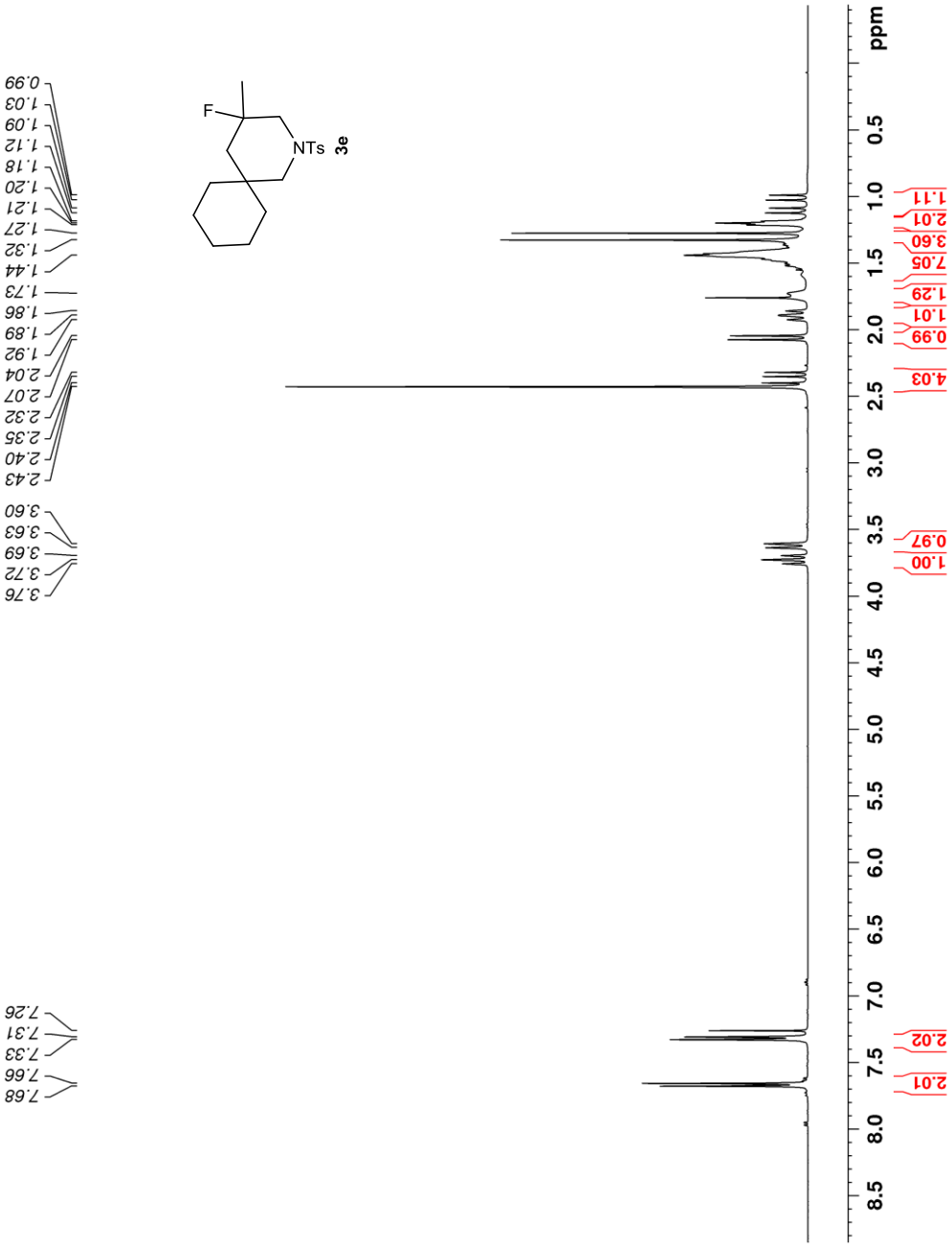
180
200


F
NTs
3e
ppm
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1.09
1.12
1.18
1.20
1.21
1.27
1.32
1.44
1.73
1.86
1.89
1.92
2.04
2.07
2.32
2.35
2.40
2.43
3.60
3.63
3.69
3.72
3.76
7.26
7.31
7.33
7.66
7.68
1.11
2.01
3.60
7.05
1.29
1.01
0.99
4.03
0.97
1.00
2.02
2.01

F
NTs
3e

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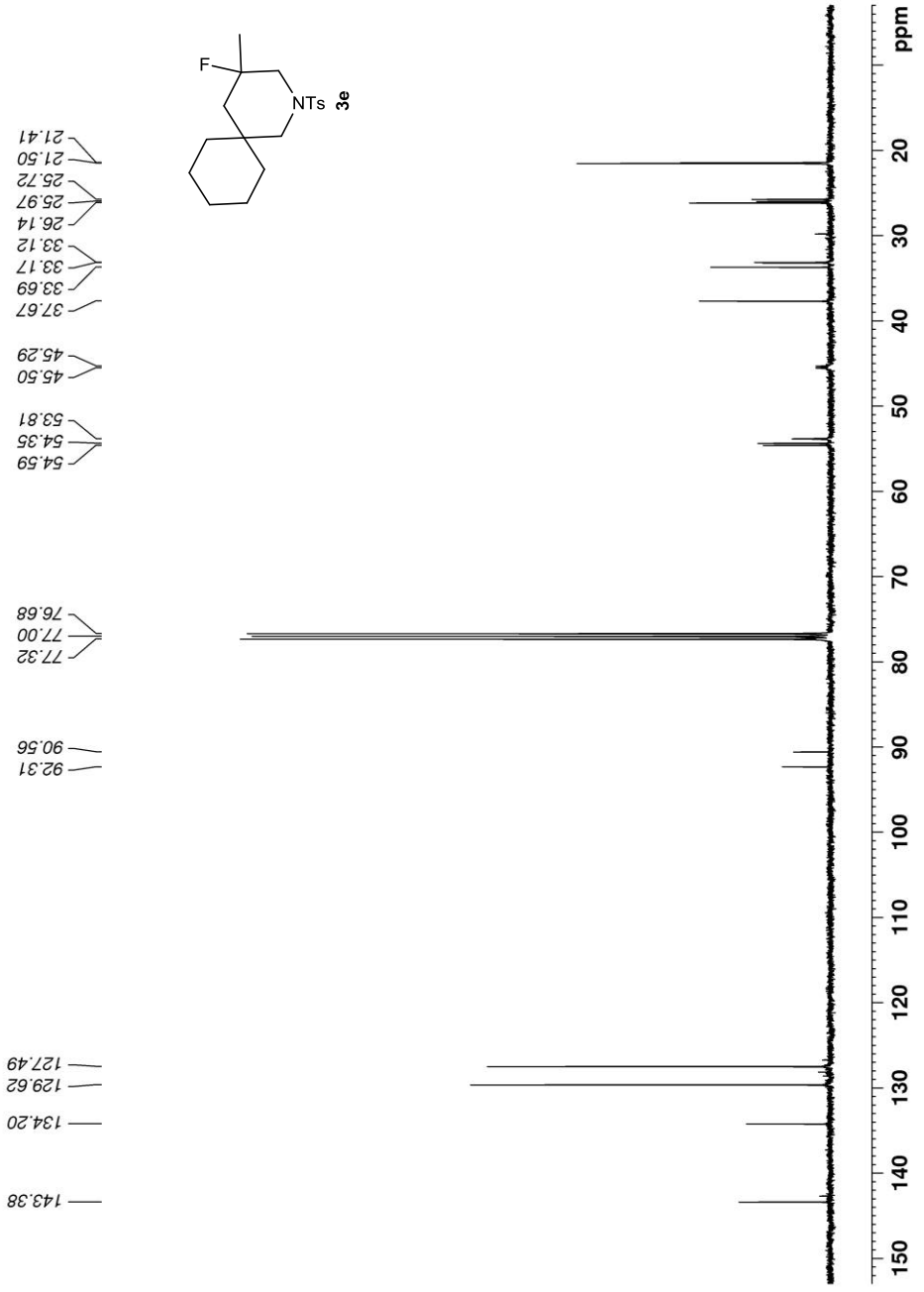
-200
ppm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NTs
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21.41
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26.14
33.12
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33.69
37.67
45.29
45.50
53.81
54.35
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76.68
77.00
77.32
90.56
92.31
127.49
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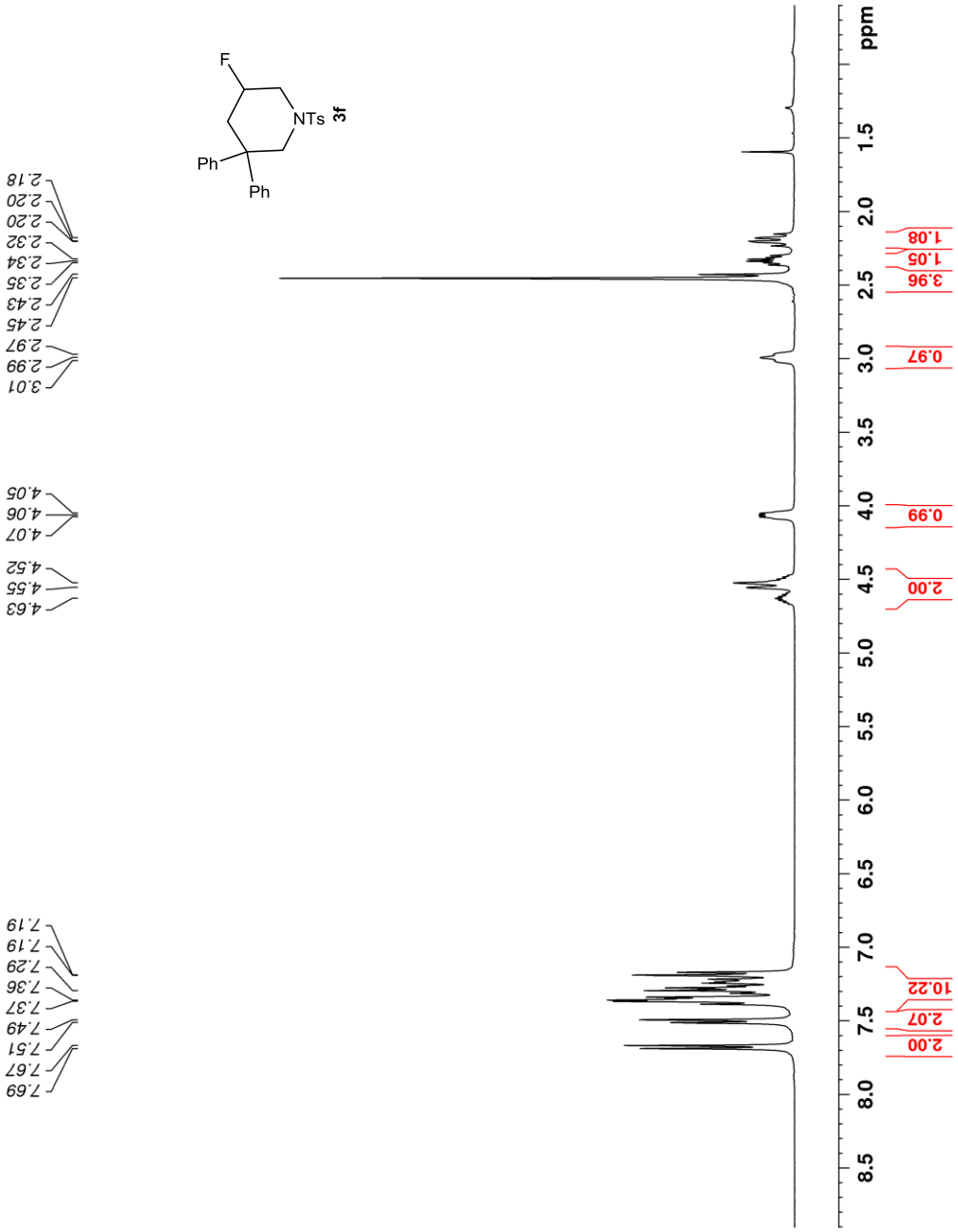
134.20
143.38
ppm


F
NTs
3f
Ph
Ph
2.18
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2.20
2.32
2.34
2.35
2.43
2.45
2.97
2.99
3.01
4.05
4.06
4.07
4.52
4.55
4.63
7.19
7.19
7.29
7.36
7.37
7.49
7.51
7.67
7.69
ppm
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
1.08
1.05
3.96
0.97
0.99
2.00
10.22
2.07
2.00

F
NTs
3f
Ph
Ph
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ppm
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-40
-20
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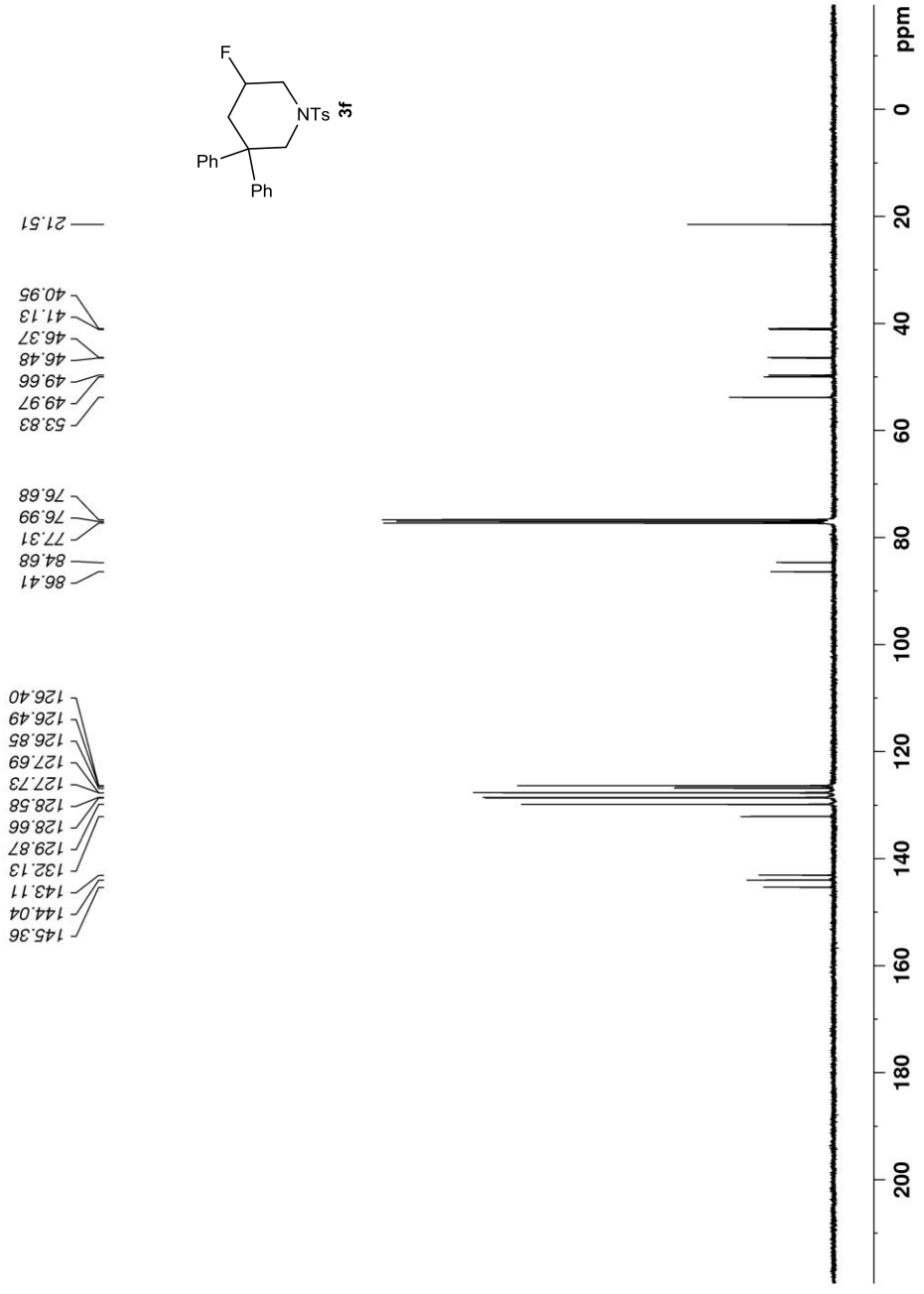
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Ph
Ph
21.51
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49.66
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53.83
76.68
76.99
77.31
84.68
86.41
126.40
126.49
126.85
127.69
127.73
128.58
128.66
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132.13
143.11
144.04
145.36
ppm
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60
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100
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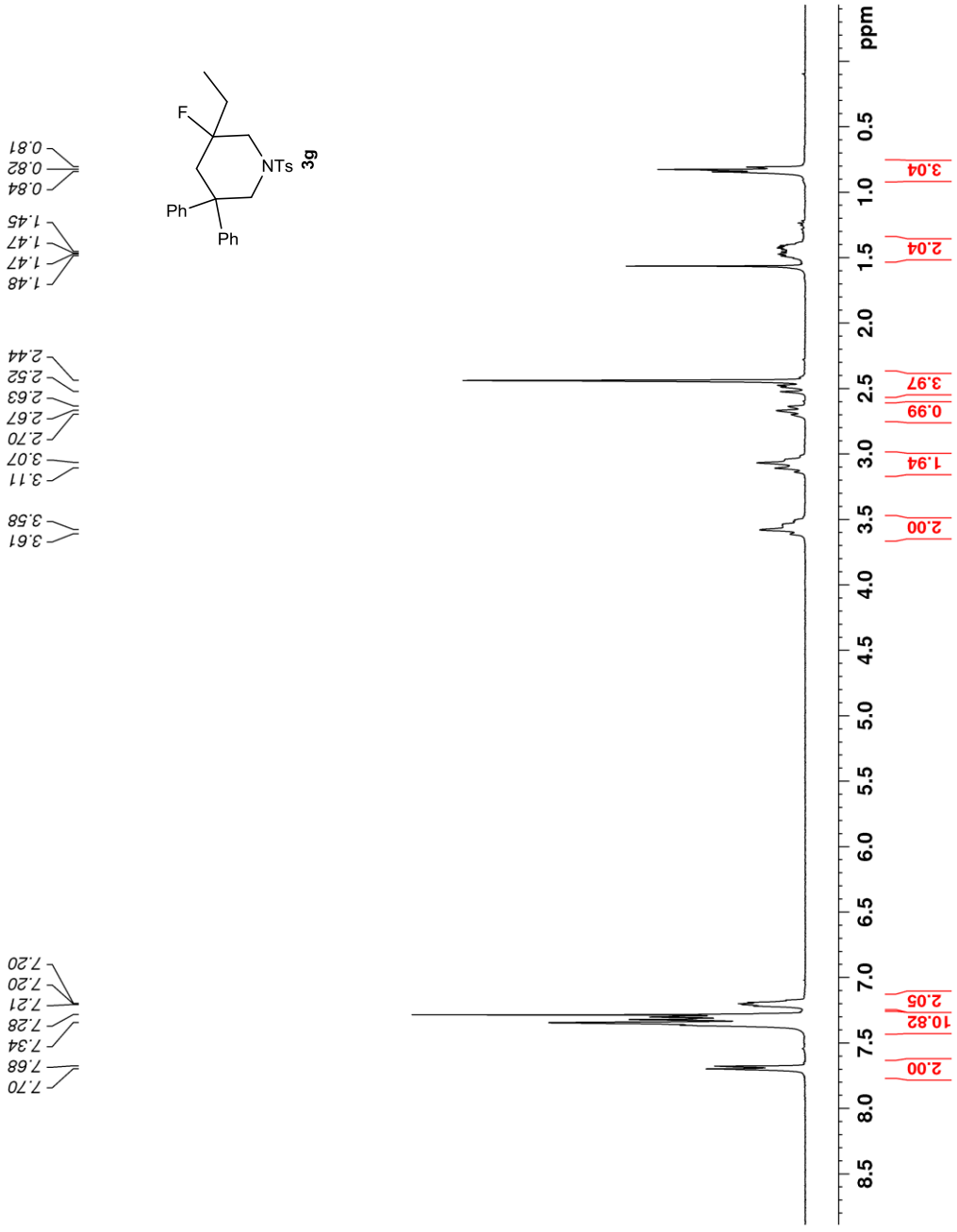
140
160
180
200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NTs
3g
Ph
Ph
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0.82
0.84
1.45
1.47
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1.48
2.44
2.52
2.63
2.67
2.70
3.07
3.11
3.58
3.61
7.20
7.20
7.21
7.28
7.34
7.68
7.70
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
3.04
2.04
3.97
0.99
1.94
2.00
2.05
10.82
2.00

F
NTs
Ph
Ph
3g
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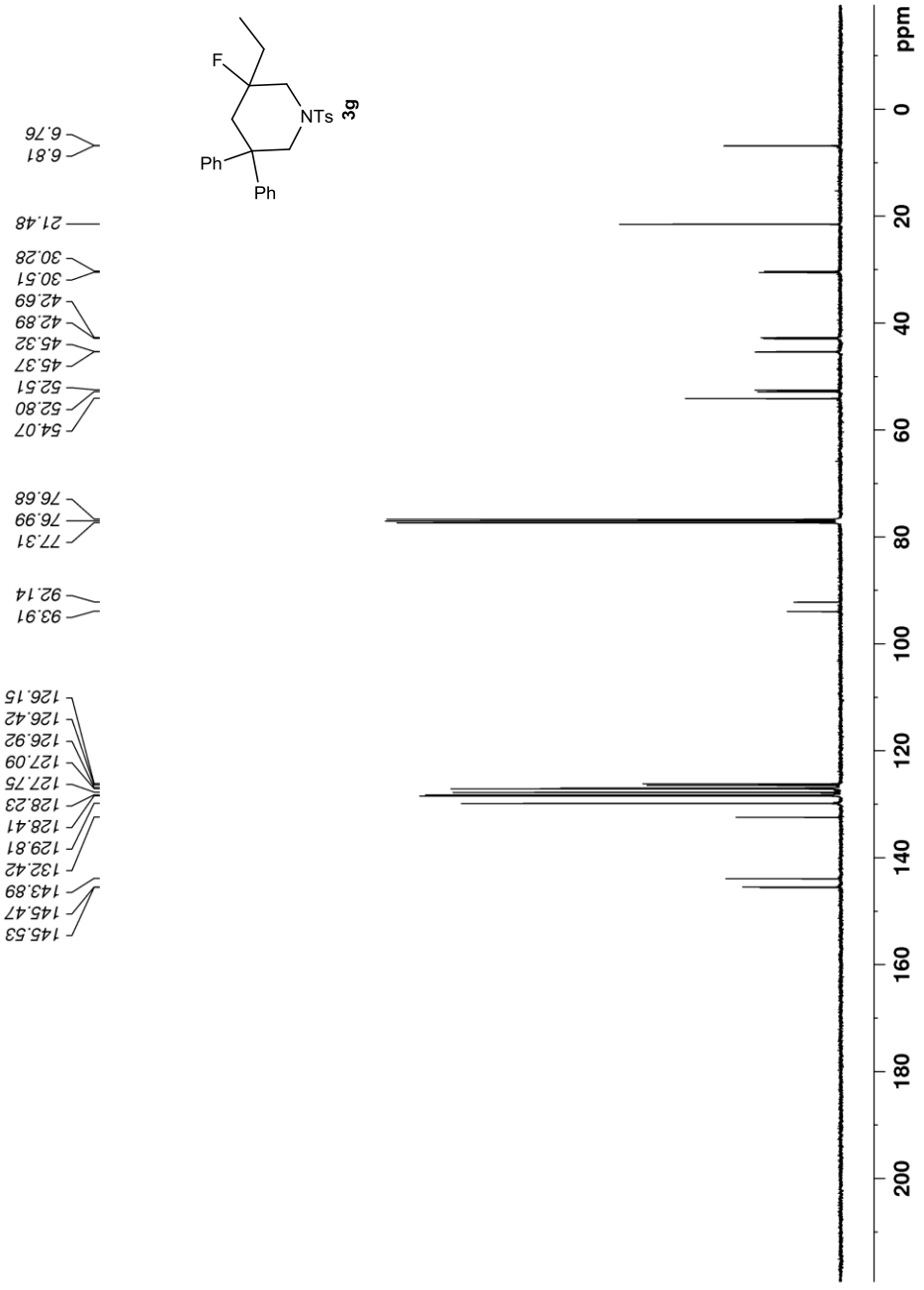
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
ppm


F
NTs
3g
Ph
Ph
6.76
6.81
21.48
30.28
30.51
42.69
42.89
45.32
45.37
52.51
52.80
54.07
76.68
76.99
77.31
92.14
93.91
126.15
126.42
126.92
127.09
127.75
128.23
128.41
129.81
132.42
143.89
145.47
145.53
ppm
0
20
40
60
80
100
120
140
160
180
200

0.83
0.85
0.87
1.19
1.21
1.36
2.44
2.62
2.65
3.05
3.08
3.12
3.49
3.52
3.61
3.64
7.20
7.30
7.35
7.69
7.71
F
C5H11-n
NTs
3h
Ph
Ph
3.03
8.76
5.19
1.96
2.00
13.02
2.02
ppm

F
C_5H_{11}-*n*
NTs
Ph
Ph
3h

-148.59
ppm
-200
-180
-160
-140
-120
-100
-80
-60
-40
-20
0

13.89
21.47
21.96
22.00
22.29
31.67
37.45
37.67
42.64
42.85
45.34
45.40
53.23
53.52
54.07
76.68
76.99
77.31
92.12
93.88
126.16
126.39
126.90
127.14
127.74
128.23
128.37
129.80
132.39
143.88
145.40
145.59
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C5H11-n
NTs
3h
Ph
Ph
ppm
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60
80
100
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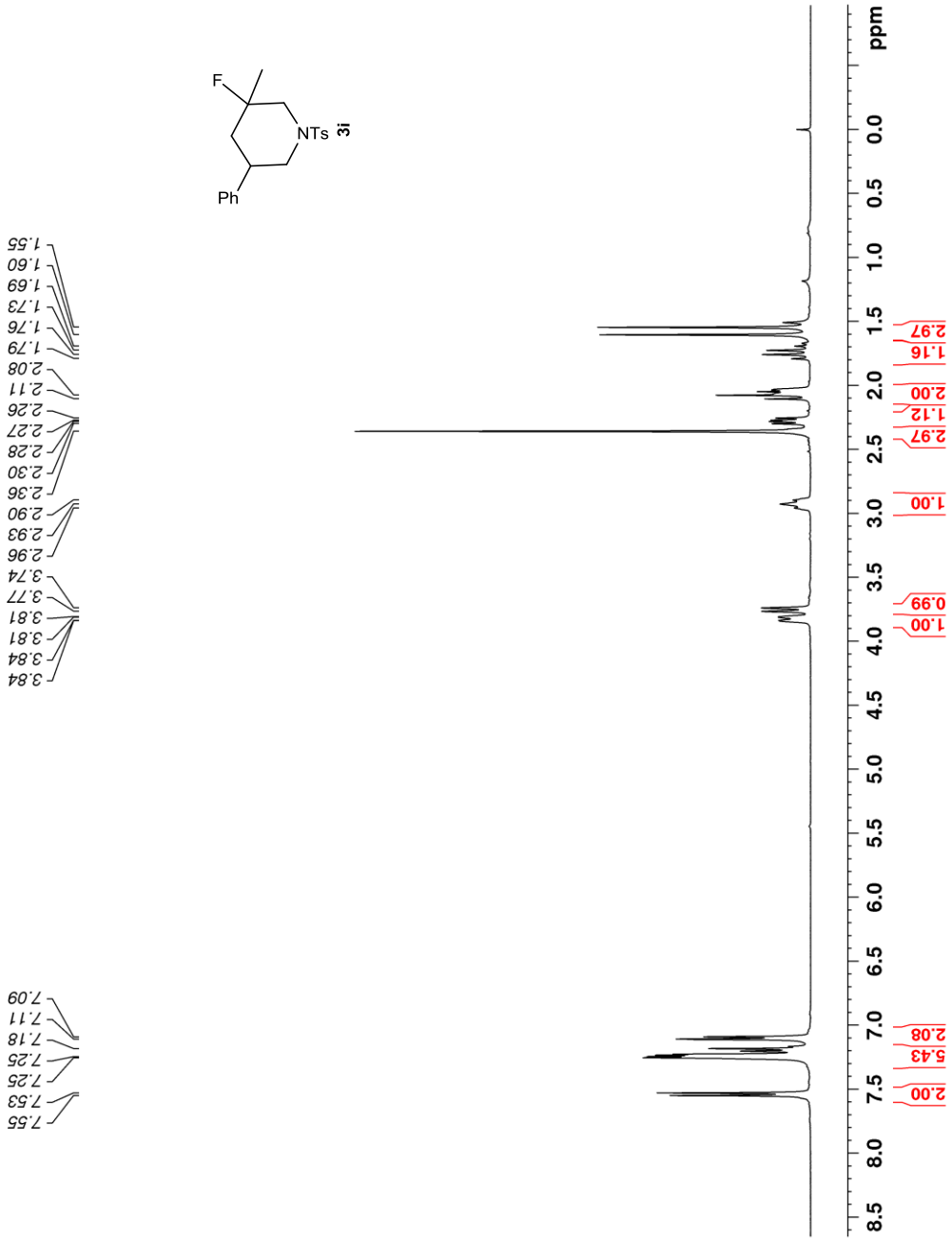
140
160
180
200


F
NTs
3i
Ph
ppm
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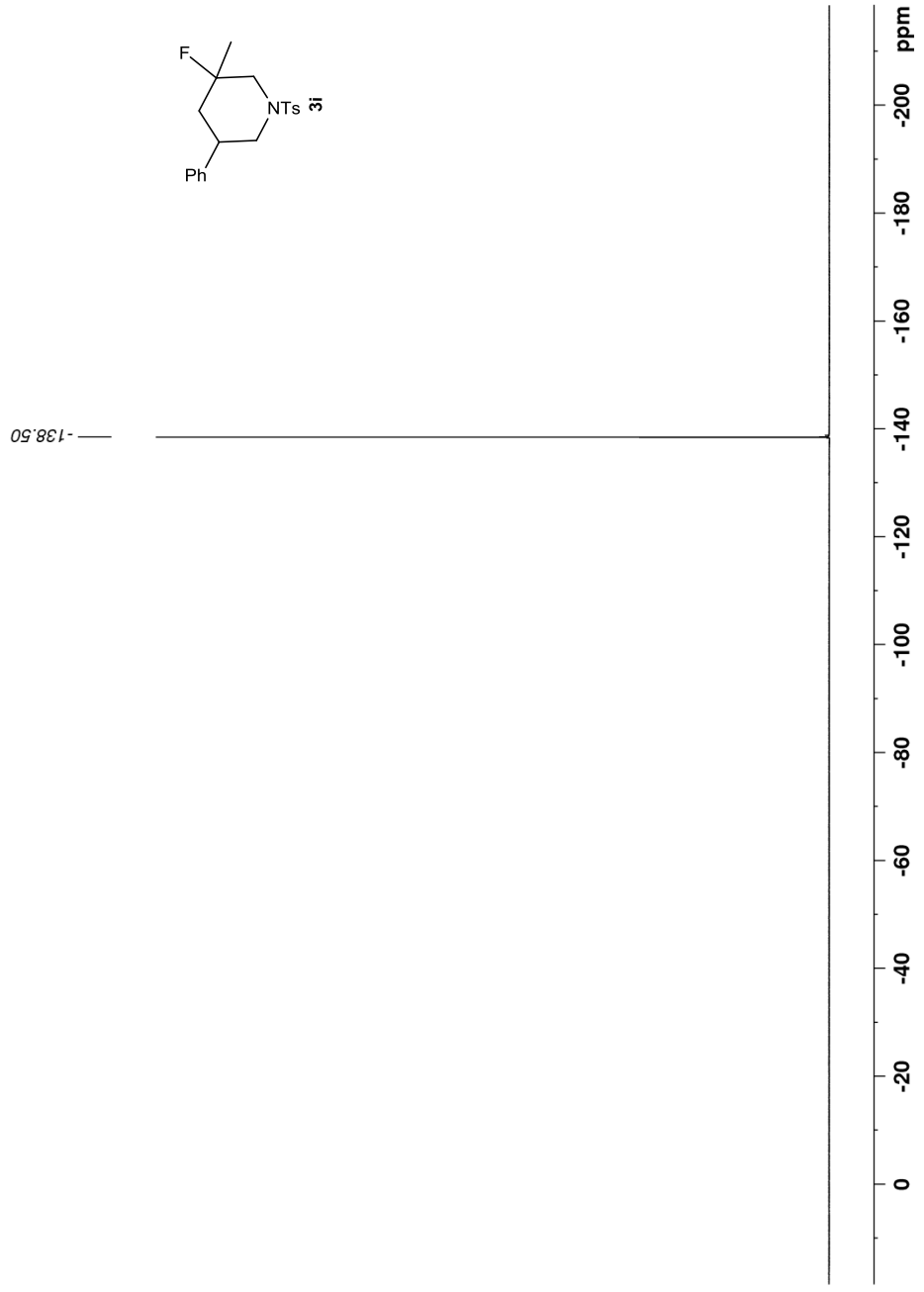
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
2.97
1.16
2.00
1.12
2.97
1.00
0.99
1.00
2.08
5.43
2.00
1.55
1.60
1.69
1.73
1.76
1.79
2.08
2.11
2.26
2.27
2.28
2.30
2.36
2.90
2.93
2.96
3.74
3.77
3.81
3.81
3.84
3.84
7.09
7.11
7.18
7.25
7.25
7.53
7.55


F
NTs
3i
Ph
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0
-20
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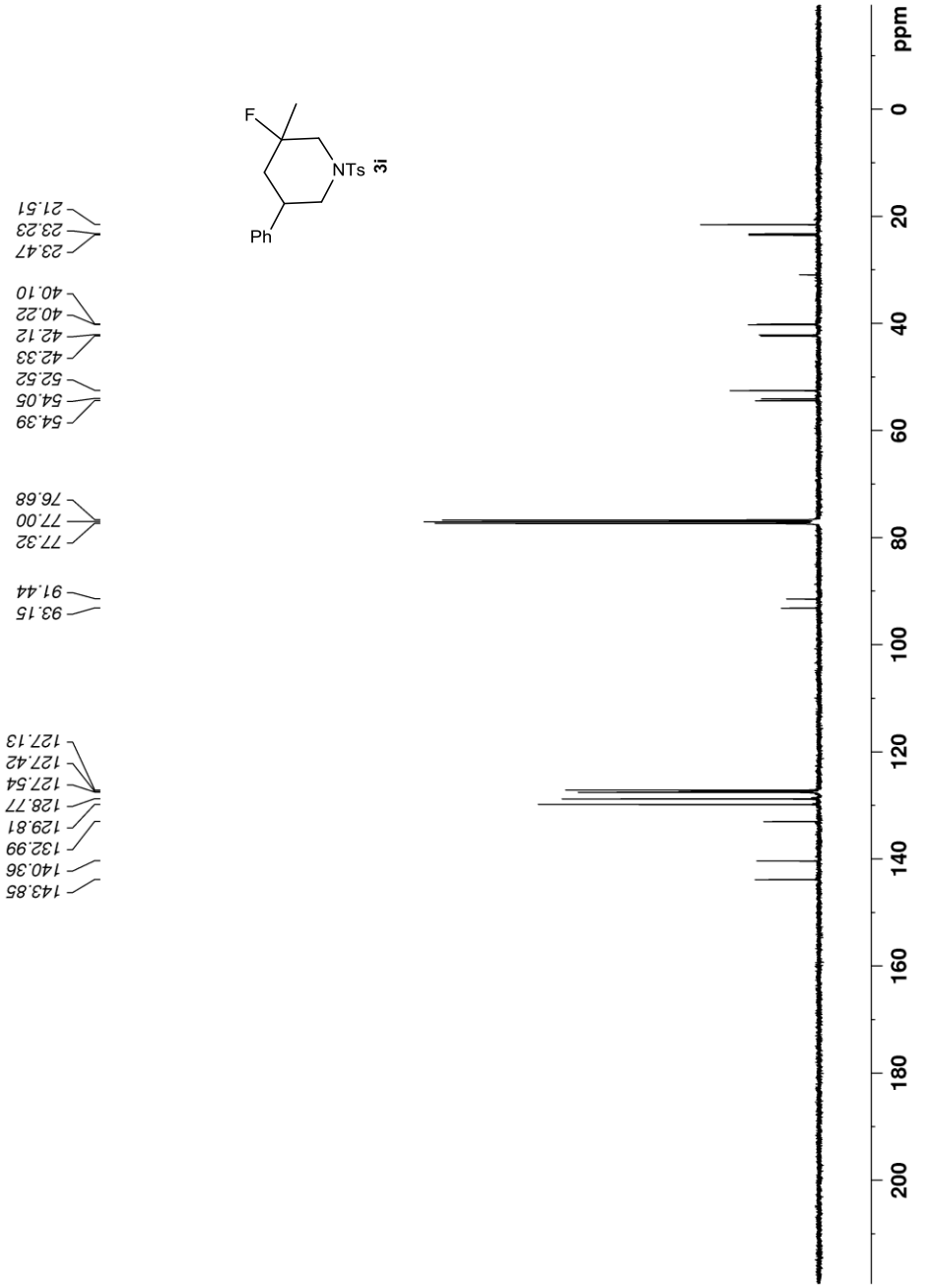
-60
-80
-100
-120
-140
-160
-180
-200
ppm


F
NTs
3i
Ph
21.51
23.23
23.47
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40.22
42.12
42.33
52.52
54.05
54.39
76.68
77.00
77.32
91.44
93.15
127.13
127.42
127.54
128.77
129.81
132.99
140.36
143.85
ppm
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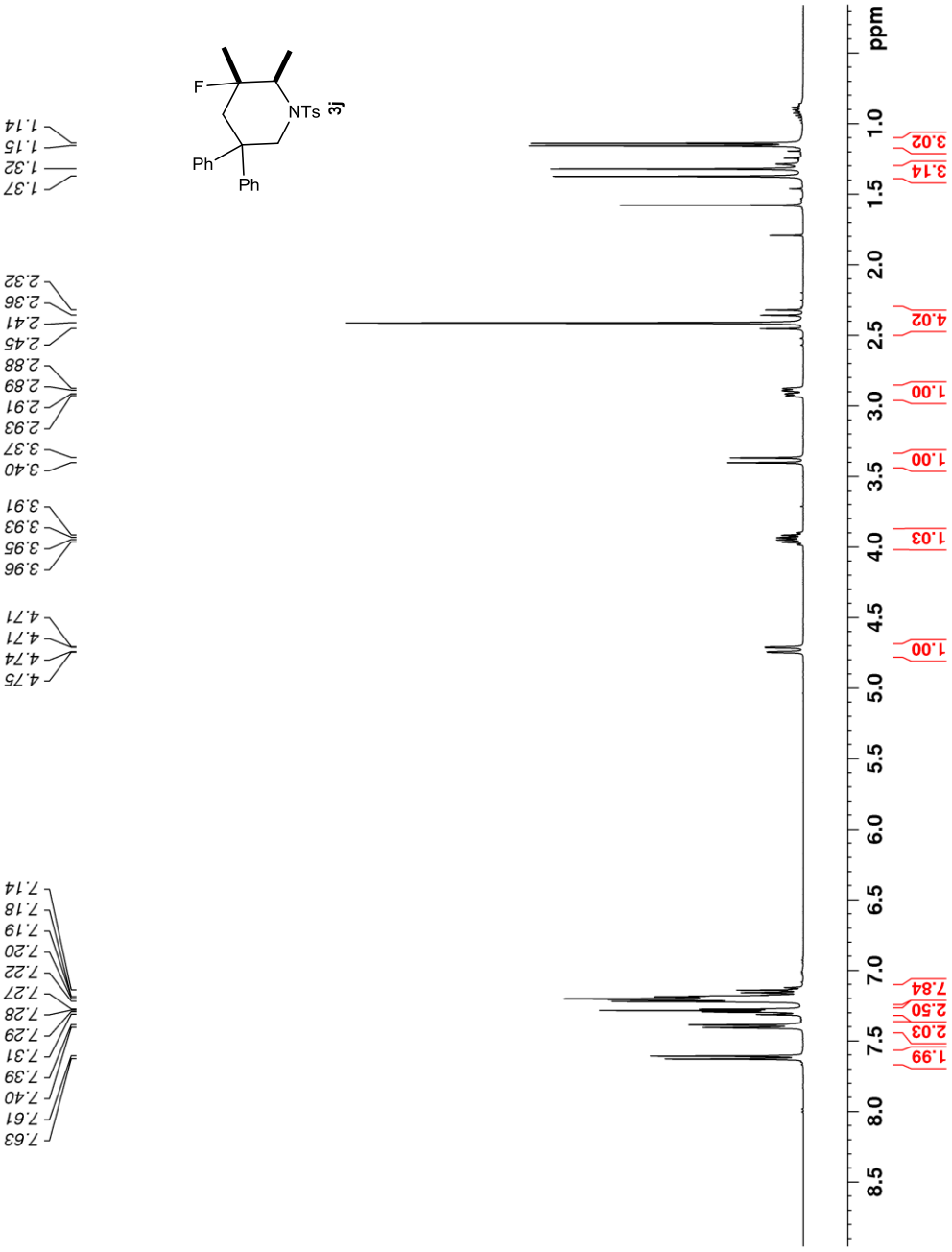
60
80
100
120
140
160
180
200


F
NTs
3j
Ph
Ph
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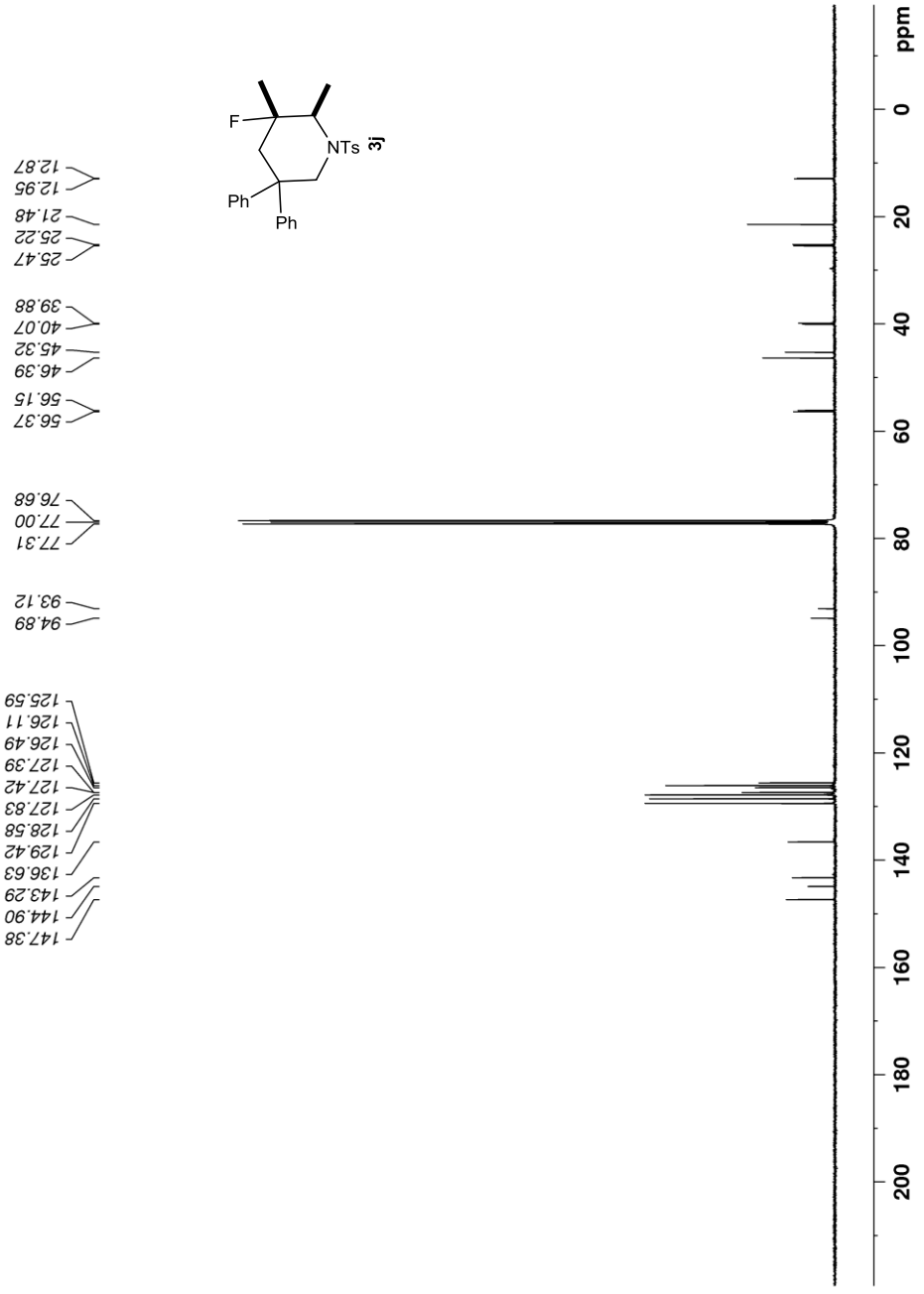
1.32
1.37
2.32
2.36
2.41
2.45
2.88
2.89
2.91
2.93
3.37
3.40
3.91
3.93
3.95
3.96
4.71
4.71
4.74
4.75
7.14
7.18
7.19
7.20
7.22
7.27
7.28
7.29
7.31
7.39
7.40
7.61
7.63
ppm
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
3.02
3.14
4.02
1.00
1.00
1.03
1.00
7.84
2.50
2.03
1.99

F
NTs
3j
Ph
Ph
-139.81
ppm
-200
-180
-160
-140
-120
-100
-80
-60
-40
-20
0


F
NTs
3j
Ph
Ph
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21.48
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25.47
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40.07
45.32
46.39
56.15
56.37
76.68
77.00
77.31
93.12
94.89
125.59
126.11
126.49
127.39
127.42
127.83
128.58
129.42
136.63
143.29
144.90
147.38
ppm
0
20
40
60
80
100
120
140
160
180
200

0.83
0.84
0.85
0.85
0.88
0.94
2.42
2.58
2.59
2.62
2.74
2.78
2.81
2.97
3.00
3.97
3.99
4.01
4.02
4.76
4.79
7.20
7.22
7.26
7.29
7.31
7.57
7.59
7.73
7.75
F
NTs
3k
Ph
Ph
ppm
0
1
2
3
4
5
6
7
8
9
3.15
3.57
3.01
1.03
1.00
0.99
1.00
1.00
2.02
1.97
6.41
4.04

-134.11
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NTs
3k
Ph
Ph
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-60
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-120
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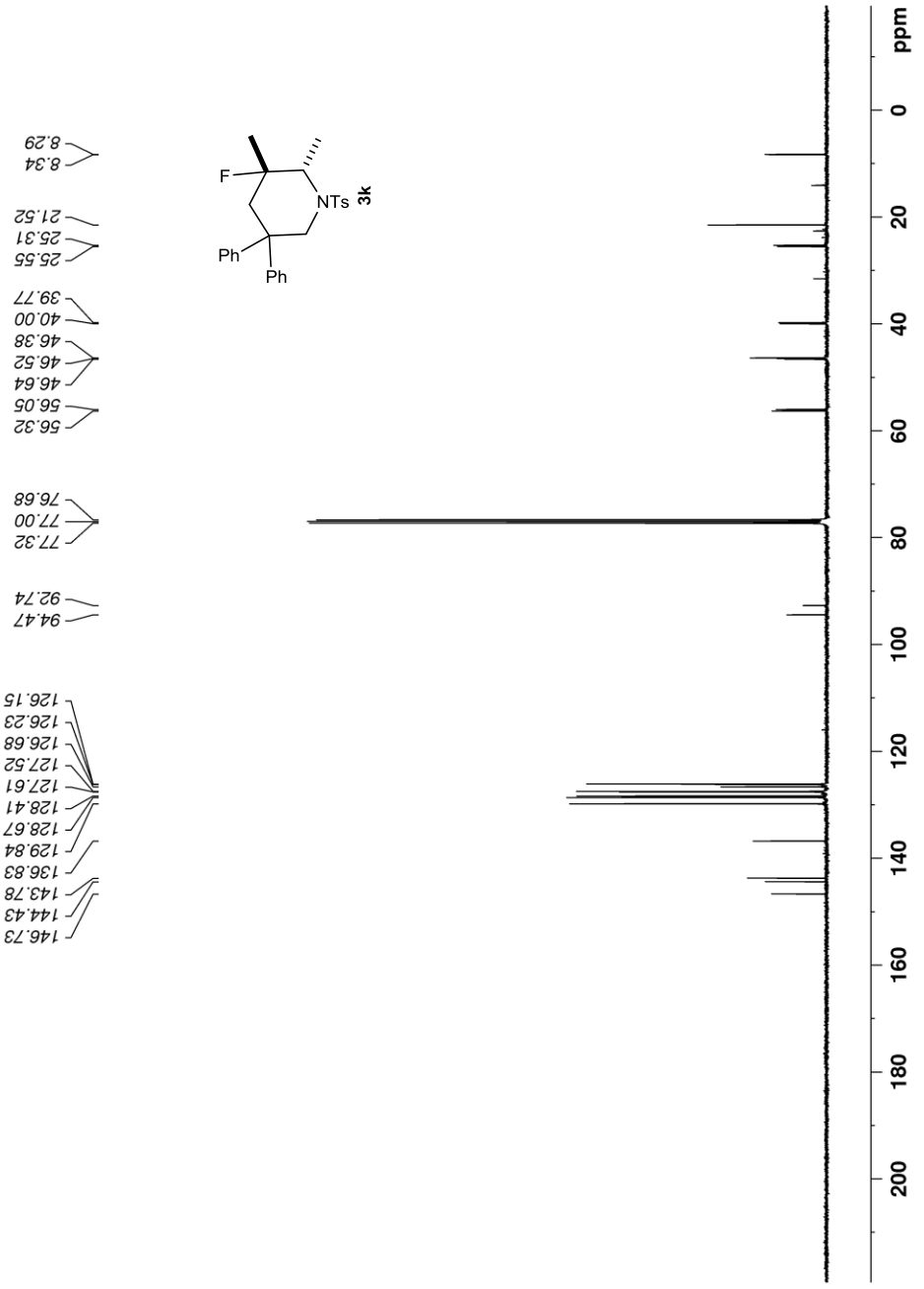
-180
-200
ppm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46.38
46.52
46.64
56.05
56.32
76.68
77.00
77.32
92.74
94.47
126.15
126.23
126.68
127.52
127.61
128.41
128.67
129.84
136.83
143.78
144.43
146.73
F
NTs
3k
Ph
Ph
ppm
0
20
40
60
80
100
120
140
160
180
200

7.72
7.70
7.33
7.31
7.26
3.59
3.56
3.51
3.45
3.38
3.35
3.30
3.25
2.43
2.13
2.10
2.06
2.04
1.93
1.88
1.84
1.76
1.47
1.42
F
NTs
3l
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
2.99
1.05
1.04
3.03
2.01
2.00
2.01
1.98

-140.89
F
NTs
3l
ppm
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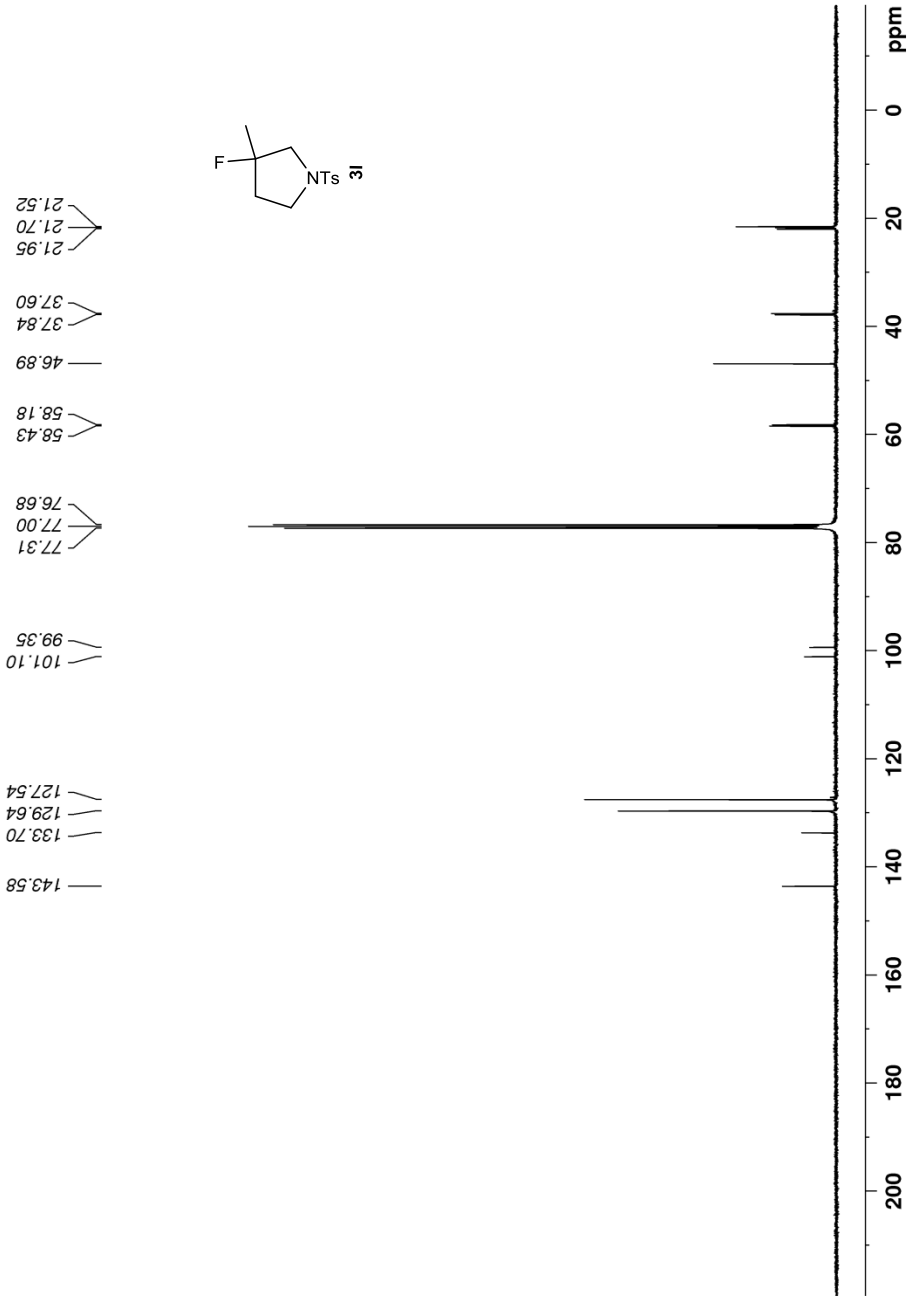
-60
-80
-100
-120
-140
-160


F
NTs
3l
21.52
21.70
21.95
37.60
37.84
46.89
58.18
58.43
76.68
77.00
77.31
99.35
101.10
127.54
129.64
133.70
143.58
ppm
0
20
40
60
80
100
120
140
160
180
200

7.66
7.64
7.33
7.31
7.26
3.38
3.33
3.27
2.79
2.76
2.73
2.70
2.43
1.90
1.84
1.76
1.62
1.59
1.59
1.48
1.40
1.35
F
NTs
3m
ppm
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
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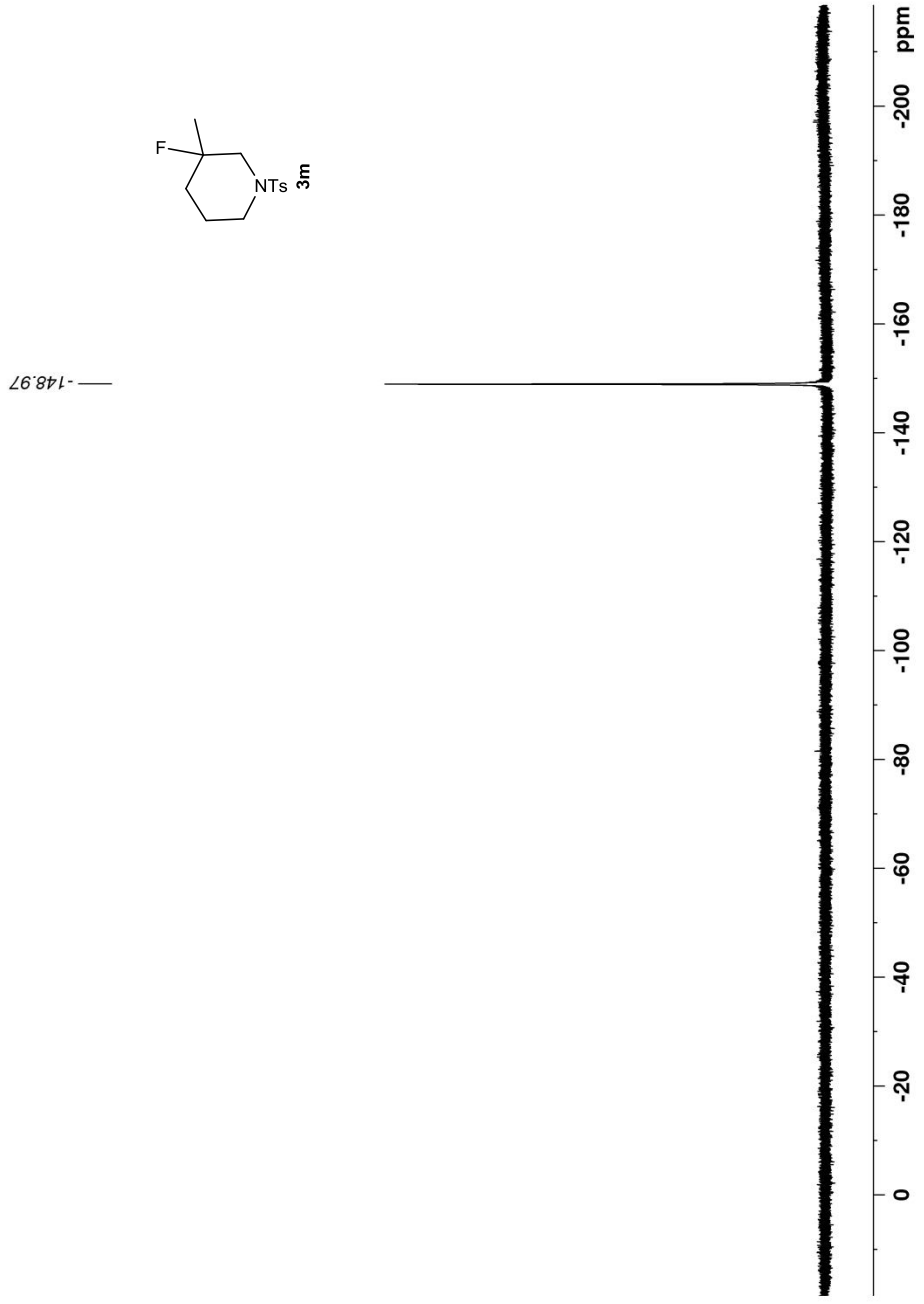
1.0
0.5
2.01
1.99
4.09
1.29
2.07
3.04
2.00
2.00


F
NTs
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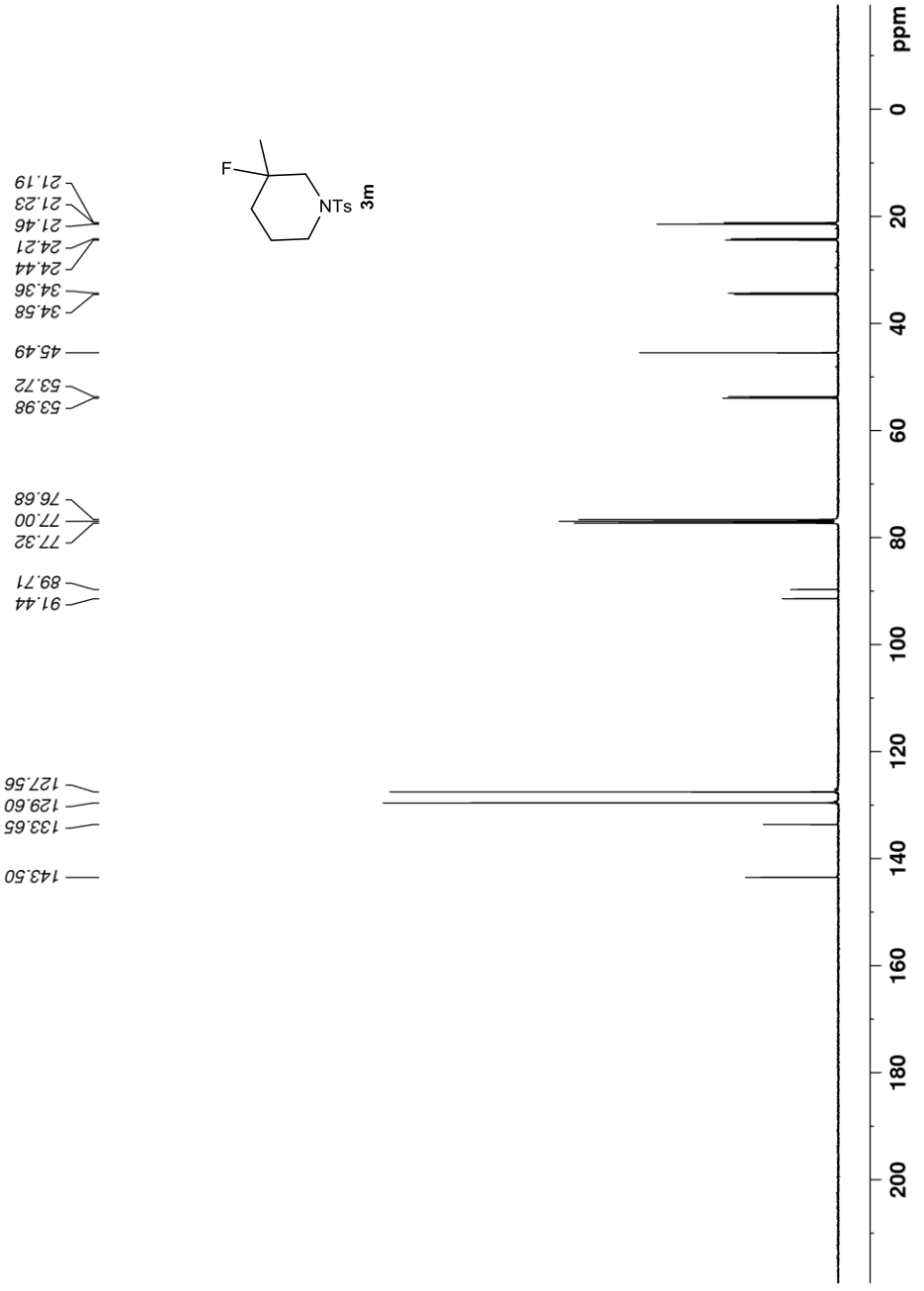
-80
-100
-120
-140
-160
-180
-200
ppm


F
NTs
3m
21.19
21.23
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24.21
24.44
34.36
34.58
45.49
53.72
53.98
76.68
77.00
77.32
89.71
91.44
127.56
129.60
133.65
143.50
ppm
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20
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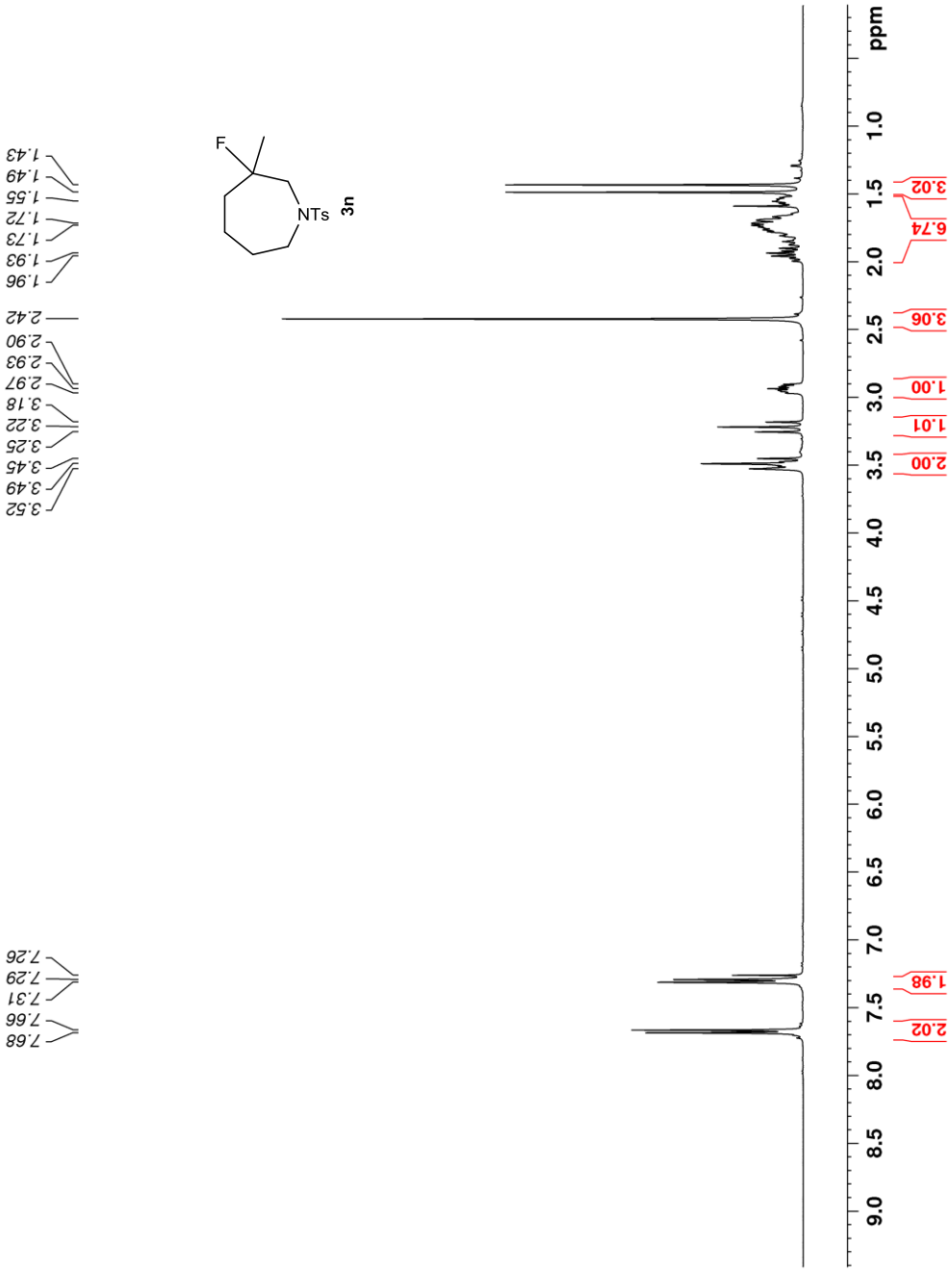
60
80
100
120
140
160
180
200


F
NTs
3n
ppm
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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
3.02
6.74
3.06
1.00
1.01
2.00
1.98
2.02
1.43
1.49
1.55
1.72
1.73
1.93
1.96
2.42
2.90
2.93
2.97
3.18
3.22
3.25
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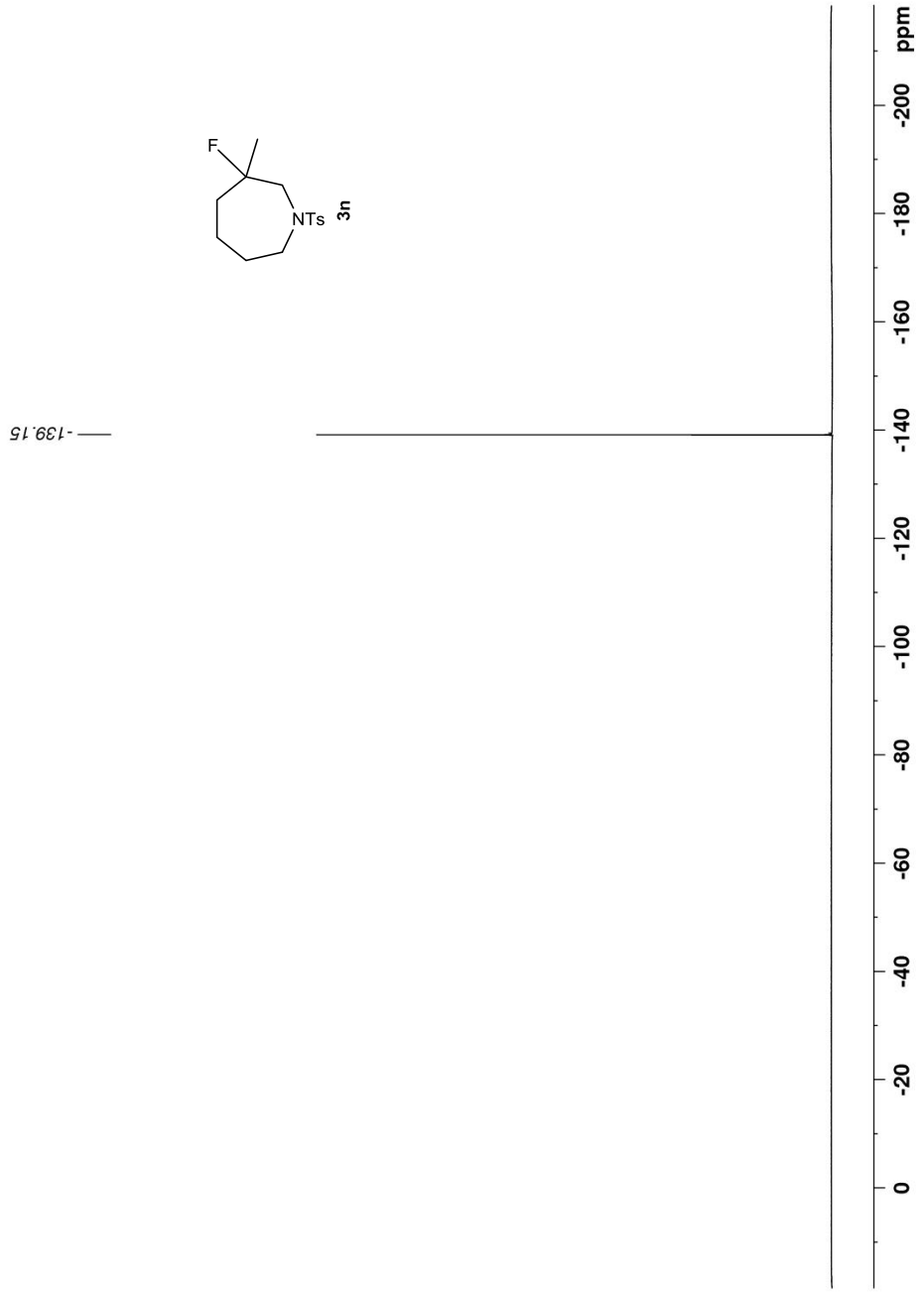
3.52
7.26
7.29
7.31
7.66
7.68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NTs
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-100
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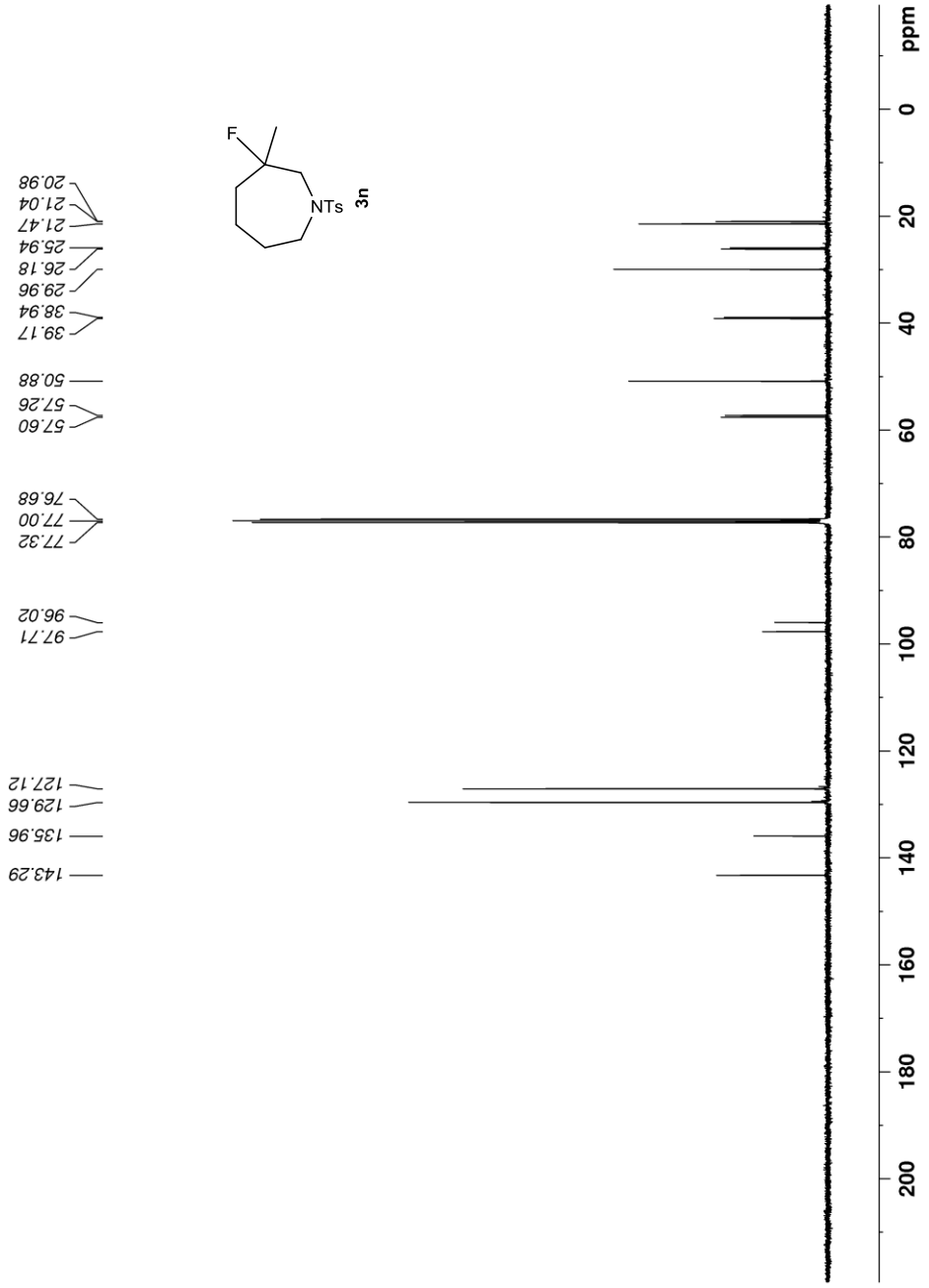
-140
-160
-180
-200
ppm


F
NTs
3n
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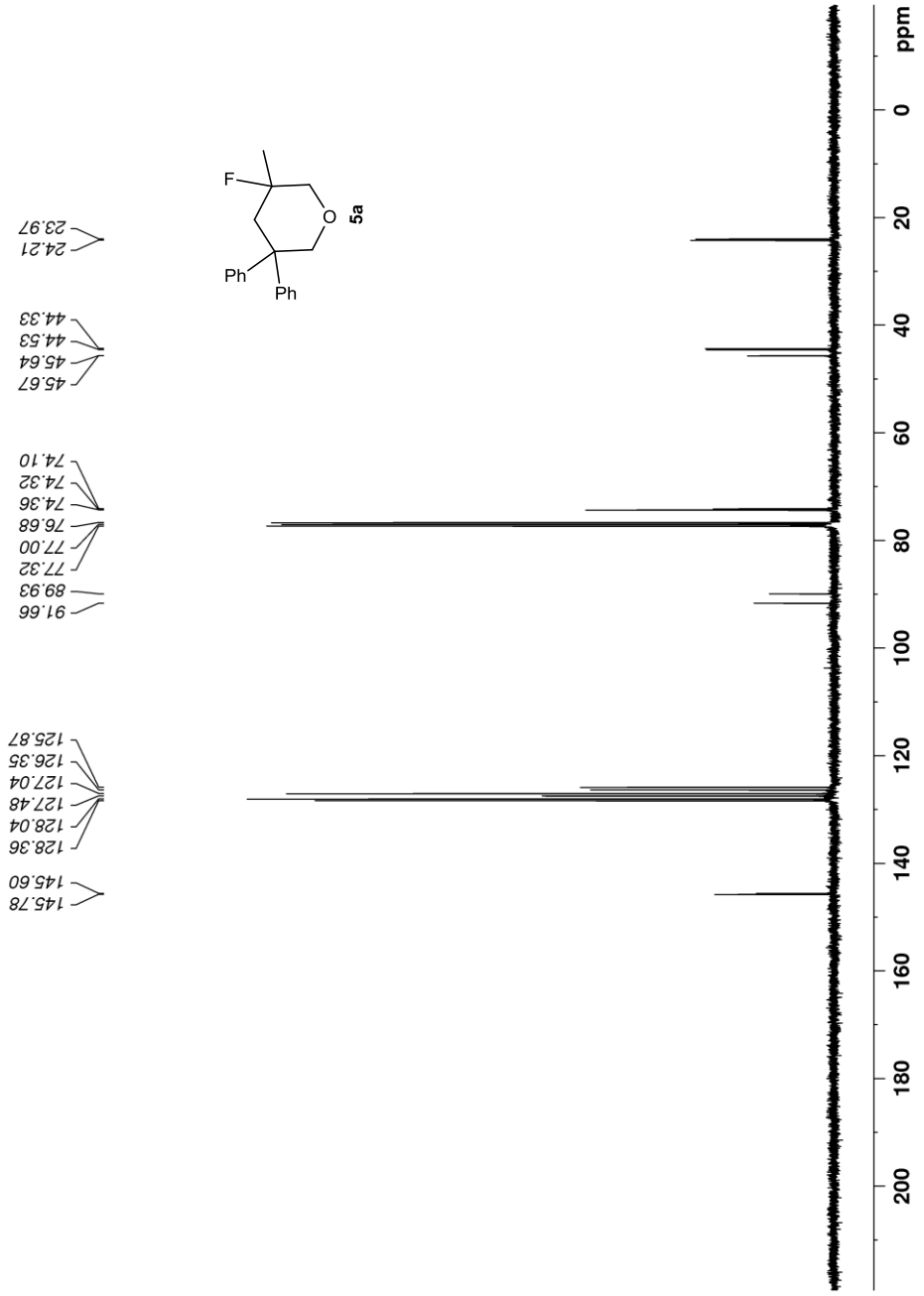
29.96
38.94
39.17
50.88
57.26
57.60
76.68
77.00
77.32
96.02
97.71
127.12
129.66
135.96
143.29
ppm
0
20
40
60
80
100
120
140
160
180
200

1.24
1.19
2.53
2.57
2.60
2.63
2.86
2.89
2.92
3.52
3.55
3.58
3.60
3.83
3.83
3.86
3.89
4.39
4.39
4.42
4.42
7.20
7.26
7.29
7.34
7.40
F
O
Ph
Ph
5a
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
3.10
1.03
1.01
1.04
2.02
1.00
10.45

-145.58
F
O
Ph
Ph
5a
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
ppm


F
O
Ph
Ph
5a
23.97
24.21
44.33
44.53
45.64
45.67
74.10
74.32
74.36
76.68
77.00
77.32
89.93
91.66
125.87
126.35
127.04
127.48
128.04
128.36
145.60
145.78
ppm
0
20
40
60
80
100
120
140
160
180
200

7.26
3.88
3.87
3.80
3.80
3.29
3.26
3.20
3.17
3.00
2.97
2.04
2.01
1.69
1.67
1.48
1.47
1.40
1.25
1.20
1.16
1.14
1.13
F
O
5b
ppm
7.5
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O
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32.82
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76.02
76.68
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77.32
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92.57

7.26
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3.63
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3.50
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3.30
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77.00
76.68
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20
15
ppm

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2.25
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F
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COOMe
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3.02
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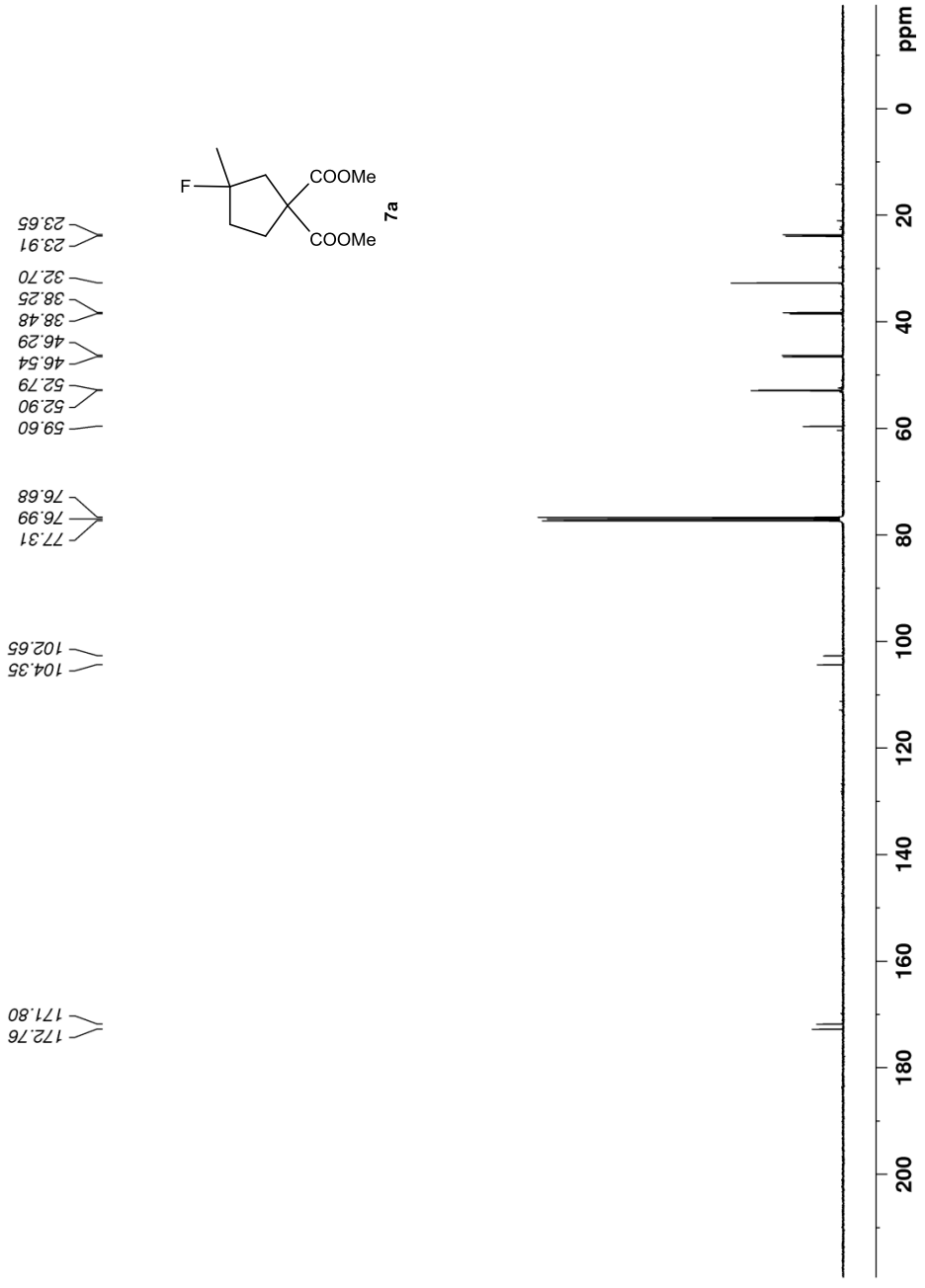
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76.68
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104.35
171.80
172.76
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40
60
80
100
120
140
160
180
200

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4.14
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2.40
2.35
2.32
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2.08
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COOEt
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ppm
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1.0
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3.02
1.35
1.09
1.20
1.15
2.00
4.17

F
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COOEt
7b
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-180
-200
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32.55
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38.51
46.16
46.40
59.71
61.48
61.63
76.68
77.00
77.31
102.67
104.37
171.30
172.32
F
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COOEt
7b
ppm
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60
80
100
120
140
160
180
200

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1.28
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1.51
1.56
2.03
2.07
2.09
2.13
2.17
2.20
2.24
2.28
2.43
2.47
2.47
2.51
2.86
2.90
2.93
2.97
4.17
4.18
4.22
4.24
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COOEt
7c
ppm
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4.0
4.5
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6.0
6.5
7.0
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8.0
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9.01
2.99
2.11
1.04
1.00
4.06

F
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COOEt
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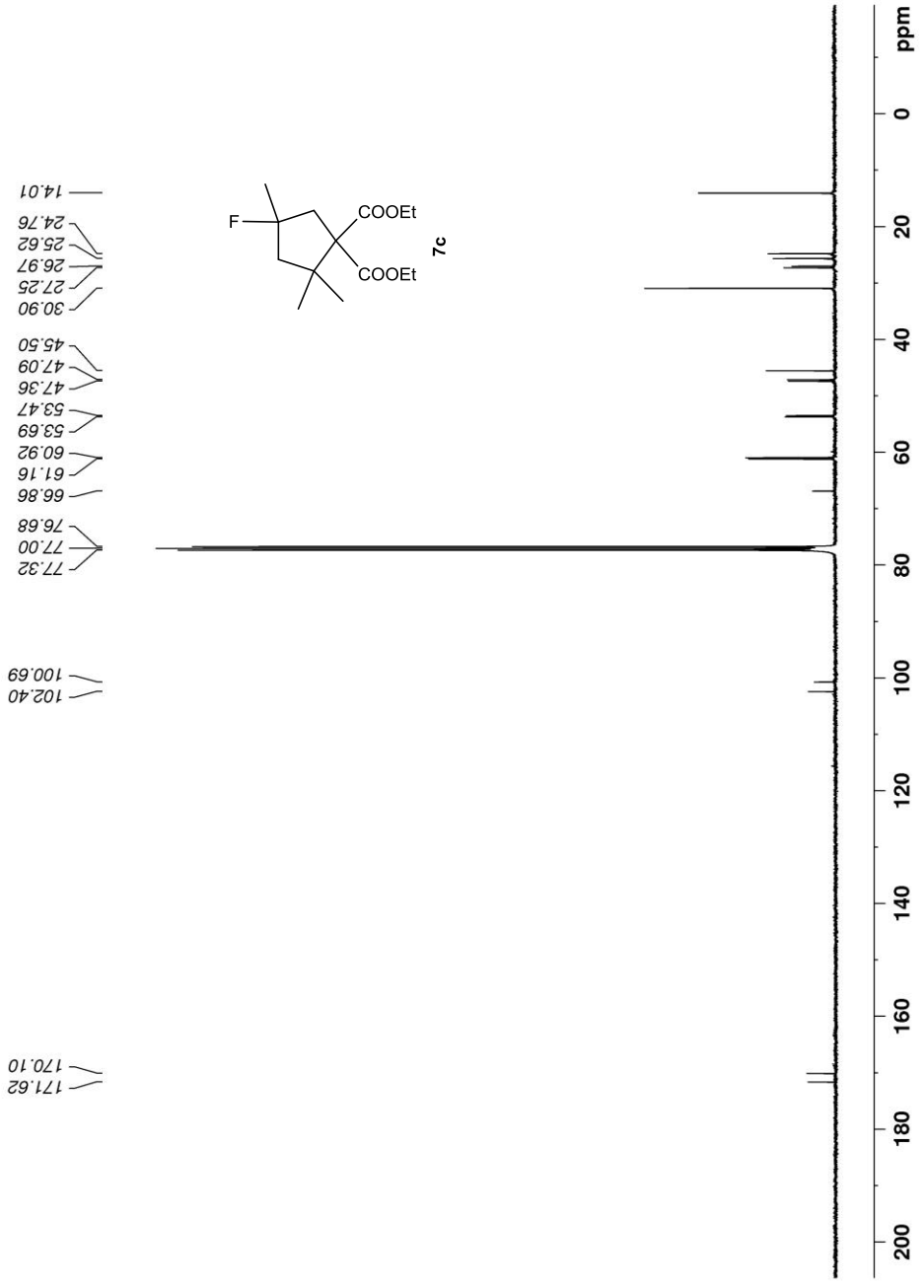
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53.69
60.92
61.16
66.86
76.68
77.00
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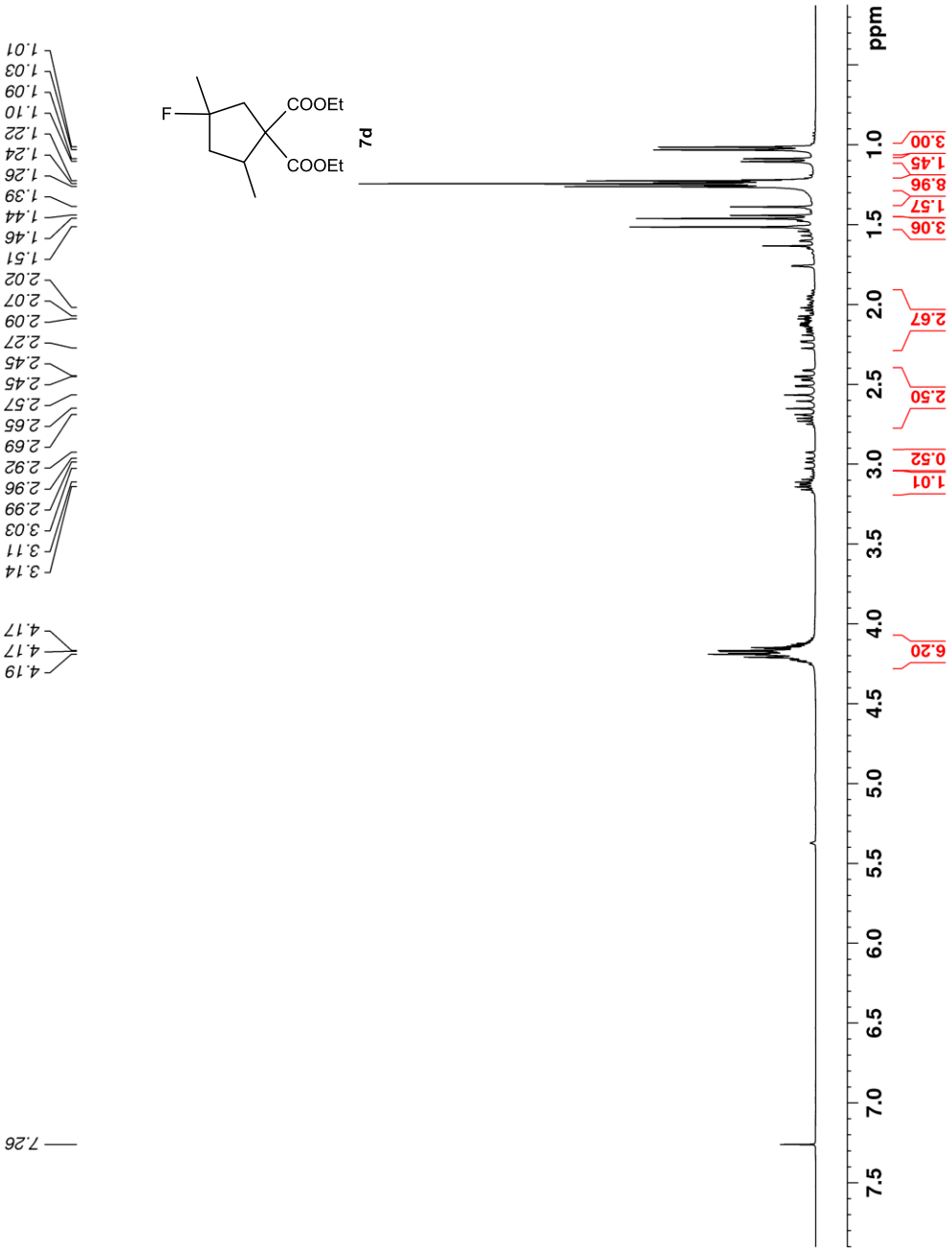
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COOEt
7d
ppm
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5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
6.20
0.52
1.01
2.50
2.67
3.06
1.57
8.96
1.45
3.00
7.26
4.19
4.17
4.17
3.14
3.11
3.03
2.99
2.96
2.92
2.69
2.65
2.57
2.45
2.45
2.27
2.09
2.07
2.02
1.51
1.46
1.44
1.39
1.26
1.24
1.22
1.10
1.09
1.03
1.01

F
COOEt
COOEt
7d
-124.74
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-80
-100
-120
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-160
-180
-200
ppm

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14.06
14.08
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16.11
23.98
24.24
26.81
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46.17
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46.40
46.53
47.03
47.27
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61.27
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77.00
77.32
100.50
101.47
102.25
103.15
170.01
171.28
171.57
171.80
F
COOEt
COOEt
7d
ppm
0
20
40
60
80
100
120
140
160
180
200