Highly Regioselective Lithiation of Pyridines Bearing an Oxetane Unit by *n*-Buthyllithium

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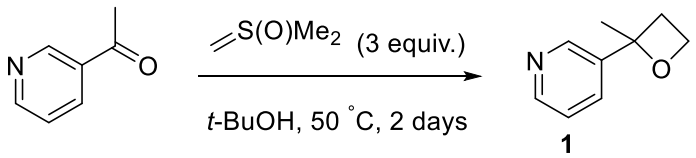
I. Material and Methods.

Unless stated otherwise, reactions were conducted in flame-dried glassware under an atmosphere of argon using anhydrous solvents. All commercially obtained reagents were used as received unless otherwise specified. TMEDA (tetramethylethylenediamine), *n*-BuLi (2.5 M in hexanes), DMF (N,N dimethylformamide, >99.8%), trimethylsilyl chloride (distilled from Mg), phenyl disulfide (CAS: 882-33-7), hexachloroethane (CAS: 67-72-1, recrystallised from EtOH), 1,2-dibromoethane (106-93-4), phenyl isocyanate (103-71-9), ethyl cyanoformate (CAS: 623-49-4), acetaldehyde (CAS: 75-07-0), 4-chlorobenzaldehyde (CAS: 104-88-1), *N*-benzylidenebenzenesulfonamide (CAS: 13909-34-7) were purchased from Sigma Aldrich. diphenylimine (CAS: 538-51-2) was purchased from Alfa Aesar. Analytical thin layer chromatography (TLC) was performed using silica gel 60 F254 precoated glassbacked plates and visualized by ultraviolet radiation (254 nm) and potassium permanganate. Flash column chromatography was performed using silica gel (particle size 40−63 nm) under air pressure. ^{1}H NMR spectra were recorded on Bruker spectrometers (at 400 MHz or 600 MHz) and are reported relative to deuterated solvent signals ($CDCl_3$: 7.26 ppm, s). Data for ^{1}H NMR spectra are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, brs = broad singlet, td = triplet of doublets, dd = doublet of doublets, dt = doublet of triplets, qd = quartet of doublets and m = multiplet), coupling constant (Hz) and integration. ^{13}C NMR spectra were recorded on Bruker Spectrometers (at 100 or 150 MHz). Data for ^{13}C NMR spectra are reported in terms of chemical shift relative to deuterated solvent signals ($^{13}CDCl_3$: 77.16 ppm, t). HRMS was performed using electrospray ionization with time-of-flight mass analysis. HRMS signals are reported to 4 decimal places and are within ±5 ppm of theoretical values. Infrared spectra were recorded neat as thin films and only selected peaks are reported. Gas chromatography-mass spectrometry (GC/MS) were performed using a Shimadzu QP2010-SE fitted with a BPX5 column (10 m, 0.1 mm, 0.1 µm film) for FID analysis and a SHIM-5MS column (30 m, 0.25 mm, 0.25 µm film) for MS analysis.

II. Synthesis of Starting Oxetane Pyridines and Spectroscopic Data for compounds 1, 5, 6 and 7

General Procedure A: Synthesis of 3-(2-methyloxetan-2-yl)pyridine **1**



1

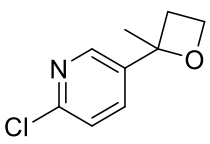
Trimethyloxosulfonium idodide (13.26 g, 60 mmol, 3 equiv.) and *t*-BuOK (6.73 g, 60 mmol, 3 equiv.) were mixed in 100 mL of *t*-BuOH. After 30 min stirring at 50 °C, 3-acetyl pyridine (2.42 g, 20 mmol, 1 equiv.) was added at 50 °C and the mixture was stirred 2 days at 50 °C, then, cooled to room temperature and t-BuOH was removed in vacuo. The resulting mixture was diluted in water (50 mL) and EtOAc (50 mL). Organic layer was separated and aqueous layer extracted with EtOAc (2x20 mL). Combined organic layer were dried over anhydrous $Na_2S_2O_4$ and solvents were removed in vacuo.
Purification: To the resulting crude yellow oil was added 300 mL of n-Hexanes and the mixture was manually shaken until the formation of an orange precipitate (ca. 10 min). The n-hexanes layer containing the product was separated from the orange precipitate by filtration and n-hexanes was removed in vacuo. The crude oxetane pyridine was obtained as a yellow oil and showed a good purity according to NMR analysis. Flash column chromatography on silica gel (*n*-hexanes/Et_2O:20/80) afforded the compound as a yellow liquid (2.38 g, 16 mmol, 80% yield).

Rf 0.28 (Petr. Ether/EtOAc = 20/80). Yellow liquid.
^{1}H NMR (CDCl3, 399.57 MHz) δ 8.63 (d, *J* = 2.0 Hz, 1H), 8.50 (dd, *J* = 4.8, 1.5 Hz, 1H), 7.80 – 7.67 (m, 1H), 7.28 (dd, *J* = 7.9, 4.9 Hz, 1H), 4.64 (dt, *J* = 8.6, 6.3 Hz, 1H), 4.53 (dt, *J* = 8.7, 6.6 Hz, 1H), 2.84 (ddd, *J* = 10.8, 8.7, 7.0 Hz, 1H), 2.73 (ddd, *J* = 10.8, 8.8, 6.6 Hz, 1H), 1.74 (s, 3H).

^{13}C NMR ($CDCl_3$, 150.92 MHz) δ 148.20, 145.89, 143.17, 131.48, 123.09, 85.06, 64.74, 35.47, 30.32.

FTIR (υ_{max} cm^{-1}) 2970, 2883, 1575, 1415, 1289, 1081, 956, 867, 808, 714.
HRMS Calcd for $C_9H_{12}NO$: 150.0913; Found $(M+H)^+$: 150.0912.

2-chloro-5-(2-methyloxetan-2-yl)pyridine (5)



5 was synthesized according to the general procedure A on a 2 mmol scale. The purification procedure was modified: after extraction with EtOAc, the resulting yellow oil was directly

purified by column chromatography over silica gel (Pet. Ether/EtOAc = 70/30). **5** was obtained as a yellow liquid (271 mg, 74% yield).

Rf 0.25 (Pet. Ether/EtOAc = 80/20). Yellow liquid.

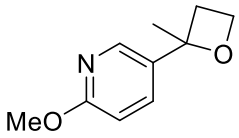
^{1}H NMR (600.13 MHz, $CDCl_3$) δ 8.41 (d, *J* = 2.0 Hz, 1H), 7.72 (dd, *J* = 8.3, 2.6 Hz, 1H), 7.33 (dd, *J* = 8.3, 0.6 Hz, 1H), 4.65 (dt, *J* = 8.7, 6.3 Hz, 1H), 4.54 (ddd, *J* = 8.8, 6.9, 6.2 Hz, 1H), 2.86 (ddd, *J* = 10.9, 8.7, 6.9 Hz, 1H), 2.71 (ddd, *J* = 10.9, 8.8, 6.5 Hz, 1H), 1.75 (s, 3H).

^{13}C NMR (100.61 MHz, $CDCl_3$) δ 149.94, 145.89, 142.31, 134.83, 123.89, 84.82, 64.92, 35.65, 30.42.

FTIR (υ_{max} cm^{-1}) 2970, 2886, 1563, 1457, 1108, 1019, 956, 832.

HRMS Calcd for $C_9H_{11}ClNO$: 184.0538; Found $(M+H)^+$: 184.0543.

2-methoxy-5-(2-methyloxetan-2-yl)pyridine (6)



6 was synthesized according to the general procedure A on a 2 mmol scale. The purification procedure was modified: after extraction with EtOAc, the resulting yellow oil was directly purified by column chromatography over silica gel (Pet. Ether/EtOAc = 70/30). **6** was obtained as a colourless liquid (240 mg, 67% yield).

Rf 0.4 (Pet. Ether/EtOAc = 80/20). Colourless liquid.

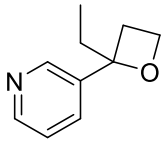
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.20 (dd, *J* = 2.5, 0.4 Hz, 1H), 7.65 (dd, *J* = 8.6, 2.5 Hz, 1H), 6.75 (dd, *J* = 8.6, 0.6 Hz, 1H), 4.63 (dt, *J* = 8.7, 6.3 Hz, 1H), 4.54 (ddd, *J* = 8.8, 6.8, 6.2 Hz, 1H), 3.94 (s, 3H), 2.79 (ddd, *J* = 10.8, 8.7, 6.9 Hz, 1H), 2.72 (ddd, *J* = 10.8, 8.8, 6.6 Hz, 1H), 1.74 (s, 3H).

^{13}C NMR (150.92 MHz, $CDCl_3$) δ 163.35, 142.64, 136.13, 135.19, 110.53, 85.22, 85.18, 64.77, 53.52, 35.69, 30.24.

FTIR (υ_{max} cm^{-1}) 2970, 2882, 1606, 1489, 1375, 1280, 1023, 959.

HRMS Calcd for $C_{10}H_{14}NO_2$: 180.1025; Found $(M+H)^+$: 180.1033.

3-(2-ethyloxetan-2-yl)pyridine (7)



7 was synthesized according to the general procedure A on a 2 mmol scale. **4** was obtained as a yellow liquid (230 mg, 70% yield).

Rf 0.32 (Pet. Ether/EtOAc = 20/80). Yellow liquid.

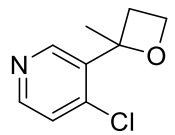
^{1}H NMR (399.78 MHz, $CDCl_3$) δ 8.57 (d, *J* = 1.8 Hz, 1H), 8.52 (dd, *J* = 4.8, 1.5 Hz, 1H), 7.73 – 7.64 (m, 1H), 7.29 (dd, *J* = 7.6, 5.0 Hz, 1H), 4.66 – 4.49 (m, 2H), 2.89 (ddd, *J* = 10.8, 8.8, 7.1 Hz, 1H), 2.68 (ddd, *J* = 10.8, 8.8, 6.4 Hz, 1H), 2.09 (dq, *J* = 14.6, 7.3 Hz, 1H), 1.95 (dq, *J* = 14.7, 7.4 Hz, 1H), 0.86 (t, *J* = 7.4 Hz, 3H).
^{13}C NMR ($CDCl_3$, 150.92 MHz) δ 148.17, 146.43, 142.01, 132.16, 123.07, 87.80, 65.27, 35.81, 33.34, 7.37.
FTIR (υ_{max} cm^{-1}) 2969, 2881, 1575, 1415, 1231, 960, 807.
HRMS Calcd for $C_{10}H_{14}NO$: 164.1075; Found $(M+H)^+$: 164.1082.

III. *O*-lithiation of Oxetane Pyridine Derivatives and Spectroscopic Data for compounds 2a-l, 3a, 4, 8-10

General Procedure B: Synthesis of 4-chloro-3-(2-methyloxetan-2-yl)pyridine **2a**

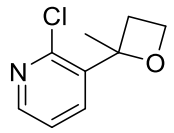
To a solution of 3-(2-methyloxetan-2-yl)pyridine **1** (1 mmol, 149 mg) and TMEDA (0.3 mmol, 35 mg) in diethyl ether (5 mL) under an argon atmosphere, at -78 °C (dry ice/acetone bath), was added *n*-BuLi over 2 min (0.6 mL, 1.5 mmol, 2.5 M in *n*-hexanes). The solution was stirred for 2 h at -78 °C. Then, C_2Cl_6 (2 mmol, 473 mg) was added at -78 °C and the solution was then allowed to reach room temperature and stirred for 1 h 30 at room temperature. The reaction was quenched with H_2O (10 mL), extracted with EtOAc (3x5 mL), dried over Na_2SO_4 and solvents were removed in vacuo. Flash column chromatography on silica gel (Petr. Ether/EtOAc: 70/30) afforded the expected compound as a yellow oil (155 mg**,** 85% *yield*).

4-chloro-3-(2-methyloxetan-2-yl)pyridine (2a)



Rf 0.26 (Petr. Ether/EtOAc = 70/30). Yellow oil.
^{1}H NMR (600.13 MHz, $CDCl_3$) δ 8.85 (s, 1H), 8.44 (d, *J* = 5.3 Hz, 1H), 7.27 (d, *J* = 5.3 Hz, 1H), 4.68 (ddd, *J* = 8.1, 7.2, 6.0 Hz, 1H), 4.51 (ddd, *J* = 8.3, 7.3, 6.0 Hz, 1H), 2.96 – 2.86 (m, 2H), 1.81 (s, 3H).
^{13}C NMR (100.47 MHz, $CDCl_3$) δ 149.20, 147.82, 140.14, 139.52, 124.60, 85.37, 65.45, 34.51, 28.33.
FTIR (υ_{max} cm^{-1}) 2970, 2885, 1552, 1464, 1397, 1072, 963, 823.
HRMS (ESI+) Calculated for $C_9H_{11}ClNO$: 184.0529; Found $(M+H)^+$: 184.0536.

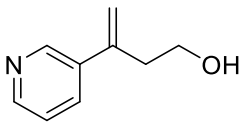
2-chloro-3-(2-methyloxetan-2-yl)pyridine (3a)



Isolated as a by-product (7 mg, 4% yield) by column chromatography over silica gel (Petr. Ether/EtOAc = 80/20) when general procedure B was applied with 1.5 equivalent of TMEDA.

Rf 0.50 (Petr. Ether/EtOAc = 70/30). Yellow oil.
^{1}H NMR (399.57 MHz, $CDCl_3$) δ 8.31 (dd, *J* = 4.7, 1.8 Hz, 1H), 8.00 (dd, *J* = 7.6, 1.8 Hz, 1H), 7.29 (dd, *J* = 7.6, 4.8 Hz, 1H), 4.66 (dd, *J* = 14.4, 6.9 Hz, 1H), 4.44 (dt, *J* = 8.3, 6.9 Hz, 1H), 3.07 – 2.84 (m, 2H), 1.80 (s, 3H).
^{13}C NMR (100.48 MHz, $CDCl_3$) δ 148.27, 146.99, 141.76, 135.27, 122.76, 85.44, 65.38, 34.50, 28.24.
FTIR (υ_{max} cm^{-1}) 2964, 2885, 1563, 1395, 1048, 966, 807.
HRMS (ESI+) Calculated for $C_9H_{11}ClNO$: 184.0529; Found $(M+H)^+$: 184.0531.

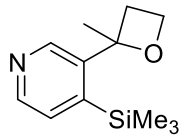
3-(pyridin-3-yl)but-3-en-1-ol (4)



Isolated as a degradation product (22 mg, 15% yield) by column chromatography over silica gel (EtOAc) when general procedure B was applied at 0 °C without TMEDA.

Rf 0.14 (EtOAc). Yellow oil.
^{1}H NMR (600.13 MHz, $CDCl_3$) δ 8.66 (d, *J* = 1.9 Hz, 1H), 8.50 (dd, *J* = 4.7, 1.3 Hz, 1H), 7.73 – 7.69 (m, 1H), 7.29 – 7.23 (m, 1H), 5.45 (s, 1H), 5.27 (d, *J* = 1.0 Hz, 1H), 3.75 (t, *J* = 6.5 Hz, 2H), 2.79 (td, *J* = 6.5, 1.0 Hz, 2H).
^{13}C NMR (150.92 MHz, $CDCl_3$) δ 148.86, 147.63, 142.22, 136.27, 133.58, 123.38, 116.35, 60.88, 38.35.
FTIR (υ_{max} cm^{-1}) 3265, 2873, 1627, 1414, 1042, 1022, 904, 815, 716.
HRMS (ESI+) Calculated for $C_9H_{12}NO$: 150.0919; Found $(M+H)^+$: 150.0921.

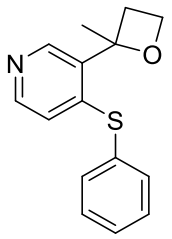
3-(2-methyloxetan-2-yl)-4-(trimethylsilyl)pyridine (2b)



Synthesized according to the general procedure B with trimethylsilyl chloride (3 mmol, 326 mg, 3 eq.) as electrophile and isolated as a yellow oil (172 mg, 78% yield) by column chromatography over silica gel (Petr. ether/EtOAc = 70/30).

Rf 0.30 (Petr. ether/EtOAc = 70/30). Yellow oil.
^{1}H NMR ($CDCl_3$, 600.13 MHz) δ 8.41 (d, *J* = 4.9 Hz, 1H), 8.30 (s, 1H), 7.46 (d, *J* = 4.9 Hz, 1H), 4.52 (ddd, *J* = 9.4, 8.7, 5.9 Hz, 1H), 4.43 (td, *J* = 8.1, 6.3 Hz, 1H), 3.01 (ddd, *J* = 16.6, 9.8, 8.5 Hz, 1H), 2.77 (ddd, *J* = 10.7, 8.6, 5.8 Hz, 1H), 1.72 (s, 3H), 0.30 (s, 9H).
^{13}C NMR ($CDCl_3$, 150.90 MHz) δ 149.17, 146.79, 146.18, 145.09, 129.58, 87.24, 64.64, 37.01, 32.56, 1.17.
FTIR (υ_{max} cm^{-1}) 2954, 2885, 1578, 1243, 967, 830, 742.
HRMS (ESI+) Calculated for $C_{12}H_{20}NOSi$: 222.1341; Found $(M+H)^+$: 222.1332.

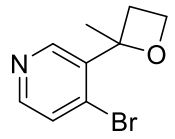
3-(2-methyloxetan-2-yl)-4-(phenylthio)pyridine (2c)



Synthesized according to the general procedure B with phenyl disulfide (2 mmol, 436 mg, 2 eq.) as electrophile and isolated as a yellow oil (234 mg, 91% yield) by column chromatography over silica gel (Petr. ether/EtOAc = 60/40).

Rf 0.26 (Petr. ether/EtOAc = 60/40). Yellow oil.
^{1}H NMR ($CDCl_3$, 399.57 MHz) δ 8.66 (s, 1H), 8.19 (d, *J* = 5.4 Hz, 1H), 7.55 – 7.33 (m, 5H), 6.63 (d, *J* = 5.4 Hz, 1H), 4.69 (dt, *J* = 8.3, 6.5 Hz, 1H), 4.52 (dt, *J* = 8.9, 6.3 Hz, 1H), 3.06 (ddd, *J* = 11.0, 9.0, 6.9 Hz, 1H), 2.90 (ddd, *J* = 11.1, 8.4, 6.7 Hz, 1H), 1.88 (s, 3H).
^{13}C NMR ($CDCl_3$, 100.61 MHz) δ 148.16, 145.82, 145.15, 138.98, 135.15, 130.07, 129.97, 129.60, 121.08, 86.12, 65.63, 34.69, 28.63.
FTIR (υ_{max} cm^{-1}) 2967, 2882, 1568, 1439, 1077, 963, 745.
HRMS (ESI+) Calculated for $C_{15}H_{16}NOS$: 258.0947; Found $(M+H)^+$: 258.0958.

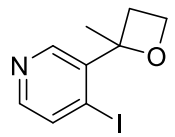
4-bromo-3-(2-methyloxetan-2-yl)pyridine (2d)



Synthesized according to the general procedure B with 1,2 dibromoethane (3 mmol, 563 mg, 3 eq.) as electrophile and isolated as a yellow oil (187 mg, 82% yield) by column chromatography over silica gel (Petr. ether/EtOAc = 70/30).

Rf 0.29 (Petr. ether/EtOAc = 70/30). Colorless oil.
^{1}H NMR (CDCl3, 399.57 MHz) δ 8.80 (s, 1H), 8.28 (d, *J* = 5.2 Hz, 1H), 7.43 (d, *J* = 5.2 Hz, 1H), 4.64 (dd, *J* = 14.2, 7.0 Hz, 1H), 4.45 (dt, *J* = 13.3, 6.7 Hz, 1H), 2.94 – 2.81 (m, 2H), 1.79 (s, 3H).
^{13}C NMR (CDCl3, 100.61 MHz) δ 148.99, 147.81, 141.86, 129.20, 128.10, 86.57, 65.55, 34.89, 28.57.
FTIR (υ_{max} cm^{-1}) 2969, 2885, 1544, 1393, 1257, 1061, 962, 821.
HRMS (ESI+) Calculated for $C_9H_{11}BrNO$: 228.0032; Found $(M+H)^+$: 228.0022.

4-iodo-3-(2-methyloxetan-2-yl)pyridine (2e)

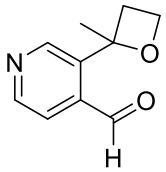


Synthesized according to the general procedure B with iodine (2 mmol, 507 mg, 2 eq.) as electrophile and isolated as beige solid (217 mg, 79% yield) by column chromatography over silica gel (Petr. ether/EtOAc = 70/30).
On a gram-scale: Synthesized according to the general procedure B with 1 (16.7 mmol, 2.5 g, 1 eq.), n-BuLi (23.4 mmol, 9.35 mL, 1.4 eq.), TMEDA (5.01 mmol, 582 mg, 0.3 eq.), iodine (41.7 mmol, 5.3 g, 2.5 eq.) in methyl *tert*-butyl ether (90 mL). *N*-BuLi was added over 20 min to avoid high variation in temperature. Column chromatography over silica gel (Petr. ether/EtOAc = 70/30) followed by a recrystallization from petroleum ether/ethyl acetate (99/1) afforded **2e** as a beige powder (3.10 g, 67% yield).

Rf 0.28 (Petr. ether/EtOAc = 70/30). Beige solid. ***Mp***: 85 °C (Petr. Ether/EtOAc : 99/1).
^{1}H NMR (CDCl3, 600.13 MHz) δ 8.78 (s, 1H), 8.08 (d, *J* = 5.1 Hz, 1H), 7.77 (d, *J* = 5.1 Hz, 1H), 4.66 (dt, *J* = 8.1, 6.7 Hz, 1H), 4.46 (dt, *J* = 8.8, 6.4 Hz, 1H), 3.03 – 2.90 (m, 2H), 1.84 (s, 3H).

^{13}C NMR (150.92 MHz, $CDCl_3$) δ 148.52, 147.11, 145.04, 135.10, 103.32, 88.05, 65.47, 35.13, 28.70.
FTIR (υ_{max} cm^{-1}) 2962, 2888, 1560, 1392, 1226, 1052, 960, 818, 661.
HRMS (ESI+) Calculated for $C_9H_{10}INO$: 275.9807; Found $(M+H)^+$: 275.9900.

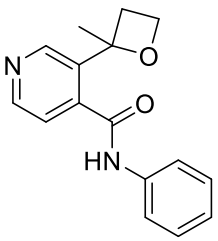
3-(2-methyloxetan-2-yl)isonicotinaldehyde (2f)



Synthesized according to the general procedure B with dimethylformamide (3 mmol, 219 mg, 3 eq.) as electrophile and isolated as a yellow oil (138 mg, 78% yield) by column chromatography over silica gel (Petr. ether/EtOAc = 50/50).

Rf 0.26 (Petr. Ether/EtOAc = 50/50). Yellow oil, becoming solid after 1 night in the fridge.
^{1}H NMR ($CDCl_3$, 600.13 MHz) δ 10.31 (s, 1H), 8.87 (s, 1H), 8.76 (d, *J* = 4.9 Hz, 1H), 7.63 (dd, *J* = 4.9, 0.5 Hz, 1H), 4.77 – 4.56 (m, 1H), 4.54 – 4.39 (m, 1H), 3.04 (ddd, *J* = 11.3, 8.6, 6.7 Hz, 1H), 2.88 (ddd, *J* = 11.3, 9.0, 6.8 Hz, 1H), 1.84 (s, 3H).
^{13}C NMR (CDCl3, 150.92 MHz) δ 191.97, 149.81, 148.09, 142.44, 137.84, 122.72, 86.24, 65.64, 36.30, 31.66.
FTIR (υ_{max} cm^{-1}) 2969, 2887, 1703, 1402, 1265, 964, 874, 837.
HRMS (ESI+) Calculated for $C_{10}H_{12}NO_2$: 178.0789; Found $(M+H)^+$: 178.0875.

3-(2-methyloxetan-2-yl)-N-phenylisonicotinamide (2g)

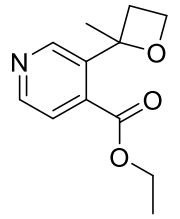


Synthesized according to the general procedure B with phenyl isocyanate (2 mmol, 238 mg, 3 eq.) as electrophile and isolated as a white solid (214 mg, 80% yield) by column chromatography over silica gel (EtOAc).

Rf 0.26 (EtOAc). White solid. ***Mp*** : 192 °C (EtOAc)

^{1}H NMR (600.13 MHz, CD_2Cl_2) δ 8.74 (s, 1H), 8.57 (d, *J* = 4.4 Hz, 1H), 8.48 (s, 1H), 7.60 (d, *J* = 8.0 Hz, 2H), 7.43 (d, *J* = 4.8 Hz, 1H), 7.37 (t, *J* = 7.8 Hz, 2H), 7.17 (t, *J* = 7.4 Hz, 1H), 4.70 – 4.55 (m, 1H), 4.46 (dt, *J* = 9.0, 6.2 Hz, 1H), 3.05 (ddd, *J* = 11.0, 9.0, 7.3 Hz, 1H), 2.84 (ddd, *J* = 11.3, 8.4, 6.5 Hz, 1H), 1.84 (s, 3H).
^{13}C NMR (CD_2Cl_2, 150.92 MHz) δ 166.31, 149.37, 148.10, 141.16, 140.06, 138.40, 129.52, 125.25, 122.02, 120.60, 86.57, 66.02, 35.63, 30.79.
FTIR (υ_{max} cm^{-1}) 3285, 1657, 1556, 1442, 1335, 1181, 955, 860, 752.
HRMS (ESI+) Calculated for $C_{16}H_{17}N_2O_2$: 269.1159; Found $(M+H)^+$: 269.1160.

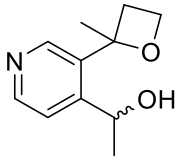
Ethyl 3-(2-methyloxetan-2-yl)isonicotinate (2h)



Synthesized according to the general procedure B with ethyl cyanoformate (2 mmol, 198 mg, 2 eq.) as electrophile and isolated as a yellow oil (170 mg, 77% yield) by column chromatography over silica gel (Petr. ether/EtOAc = 50/50).

Rf 0.3 (Petr. ether/EtOAc = 50/50). Yellow oil.
^{1}H NMR (600.13 MHz, $CDCl_3$) δ 8.93 (s, 1H), 8.63 (d, *J* = 5.0 Hz, 1H), 7.56 (d, *J* = 5.0 Hz, 1H), 4.61 (dt, *J* = 8.6, 6.3 Hz, 1H), 4.44 (ddd, *J* = 8.9, 6.8, 6.1 Hz, 1H), 4.36 (q, *J* = 7.2 Hz, 2H), 2.96 (ddd, *J* = 11.5, 8.6, 6.9 Hz, 1H), 2.79 (ddd, *J* = 11.5, 8.9, 6.6 Hz, 1H), 1.84 (s, 3H), 1.39 (t, *J* = 7.2 Hz, 3H).
^{13}C NMR ($CDCl_3$, 150.92 MHz) δ 166.75, 148.84, 148.32, 141.96, 135.07, 122.66, 86.33, 65.31, 61.97, 36.38, 30.54, 14.24.
FTIR (υ_{max} cm^{-1}) 2976, 2885, 1726, 1366, 1258, 1082, 965, 866, 718.
HRMS (ESI+) Calculated for $C_{12}H_{16}NO_3$: 222.1130; Found $(M+H)^+$: 222.1132.

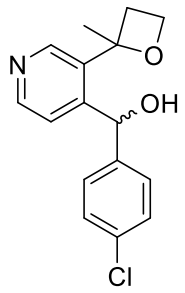
1-(3-(2-methyloxetan-2-yl)pyridin-4-yl)ethan-1-ol (2i)



Synthesized according to the general procedure B with acetaldehyde (3 mmol, 220 mg, 5 eq.) as electrophile and isolated as a yellow oil (135 mg, 70% yield) by column chromatography over silica gel (Petr. ether/EtOAc = 60/40) as a 1/1 mixture of diastereomers.

Rf 0.14 (EtOAc). Yellow oil.
^{1}H NMR ($CDCl_3$, 600.13 MHz) δ 8.32 (d, *J* = 3.5 Hz, 1H), 8.32 (d, *J* = 2.2 Hz, 1H), 8.31 (s, 1H), 8.23 (s, 1H), 7.46 (d, *J* = 5.2 Hz, 1H), 7.34 (d, *J* = 5.2 Hz, 1H`), 4.93 – 4.89 (m, 2H), 4.65 (ddd, *J* = 8.3, 7.3, 6.1 Hz, 1H), 4.57 (dt, *J* = 8.5, 6.5 Hz, 1H), 4.46 (dt, *J* = 9.2, 6.0 Hz, 1H), 4.38 (dt, *J* = 9.0, 6.2 Hz, 1H), 2.97 (ddd, *J* = 10.7, 9.3, 7.4 Hz, 1H), 2.90 (ddd, *J* = 10.7, 9.0, 6.9 Hz, 1H), 2.85 – 2.79 (m, 1H), 1.73 (s, 3H), 1.67 (s, 3H), 1.45 (d, *J* = 6.5 Hz, 3H), 1.42 (d, *J* = 6.3 Hz, 3H).
^{13}C NMR ($CDCl_3$, 150.92 MHz) δ 151.80, 150.56, 148.55, 148.40, 146.19, 145.88, 139.83, 139.16, 121.35, 121.08, 87.17, 86.15, 65.74, 65.31, 65.08, 64.80, 35.95, 35.77, 31.67, 31.48, 24.95, 22.52.
FTIR (υ_{max} cm^{-1}) 3232, 2973, 1597, 1407, 1373, 1084, 964, 907, 840, 727.
HRMS (ESI+) Calculated for $C_{11}H_{15}NO_2$: 194.1208; Found $(M+H)^+$: 194.1216.

(4-chlorophenyl)(3-(2-methyloxetan-2-yl)pyridin-4-yl)methanol (2j)

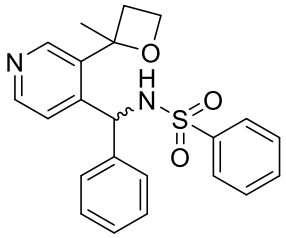


Synthesized according to the general procedure B with 4-chlorobenzaldehyde (2 mmol, 281 mg, 2 eq.) as electrophile and isolated as a white solid (261 mg, 90% yield) by column chromatography over silica gel (Petr. ether/EtOAc = 20/80) as a 1/1 mixture of diastereomers.

Rf 0.37 (Petr. ether/EtOAc = 20/80). White solid. ***Mp*** : 145 °C (EtOH).
^{1}H NMR (CD_2Cl_2, 600.13 MHz) δ 8.36 (d, *J* = 5.1 Hz, 1H), 8.32 (s, 1H), 8.30 (d, *J* = 5.1 Hz, 1H), 8.27 (s, 1H), 7.36 – 7.26 (m, 8H), 7.25 (d, *J* = 5.1 Hz, 1H), 6.87 (d, *J* = 5.1 Hz, 1H), 6.02 (s, 1H), 6.01 (s, 1H), 4.71 (ddd, *J* = 8.3, 7.5, 6.1 Hz, 1H), 4.65 – 4.58 (m, 1H), 4.48 (dt, *J* = 9.4, 6.0 Hz, 1H), 4.44 (dt, *J* = 9.2, 6.1 Hz, 1H), 3.16 – 3.01 (m, 2H), 2.89 (tdd, *J* = 11.0, 8.5, 6.1 Hz, 2H), 1.85 (s, 3H), 1.58 (s, 3H).

^{13}C NMR (CD_2Cl_2, 150.92 MHz) δ 149.34, 149.28, 149.02, 148.81, 147.39, 147.16, 142.49, 141.28, 140.83, 140.72, 133.44, 133.33, 128.80, 128.78, 128.67, 128.60, 123.95, 123.50, 88.20, 87.22, 71.39, 70.78, 66.63, 66.00, 36.44, 36.43, 31.91, 31.90.
FTIR (υ_{max} cm^{-1}) 3150, 2966, 1597, 1486, 1087, 1043, 967, 798.
HRMS (ESI+) Calculated for $C_{16}H_{17}ClNO_2$: 290.0961; Found $(M+H)^+$: 290.0955.

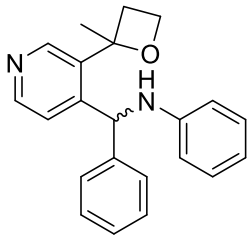
N-((3-(2-methyloxetan-2-yl)pyridin-4-yl)(phenyl)methyl)benzenesulfonamide (2k)



Synthesized according to the general procedure B by using *N*-benzylidenebenzenesulfonamide (3 mmol, 735 mg, 3 eq.) as electrophile and isolated as a white (197 mg, 50% yield) by column chromatography over silica gel (EtOAc) as a mixture of diastereomers (dr 1:0.6).

Rf 0.47 (EtOAc). White solid. ***Mp*** : 200 °C(EtOAc).
^{1}H NMR ($CDCl_3$, 600.13 MHz) δ 8.39 (d, *J* = 5.1 Hz, 1H), 8.33 (d, *J* = 4.4 Hz, 1H), 8.27 (s, 1H), 7.76 (d, *J* = 7.8 Hz, 2H), 7.70 (d, *J* = 7.8 Hz, 1H), 7.54 – 7.45 (m, 1H), 7.43 – 7.33 (m, 4H), 7.20 (dd, *J* = 8.8, 5.0 Hz, 5H), 7.01 (dd, *J* = 6.6, 2.6 Hz, 1H), 6.91 (dd, *J* = 6.6, 2.7 Hz, 2H), 6.21 (d, *J* = 7.4 Hz, 1H), 6.16 (d, *J* = 5.8 Hz, 0.5H), 5.94 (d, *J* = 7.4 Hz, 0.5H), 5.84 (d, *J* = 7.3 Hz, 1H), 4.55 (dd, *J* = 14.7, 6.7 Hz, 0.5H), 4.46 (dd, *J* = 14.4, 6.9 Hz, 1H), 4.30 (dt, *J* = 9.1, 6.1 Hz, 0.5H), 4.07 (dt, *J* = 8.9, 6.2 Hz, 1H), 3.06 – 2.93 (m, 0.5H), 2.85 (ddd, *J* = 10.8, 8.4, 6.3 Hz, 0.5H), 2.78 – 2.65 (m, 2H), 1.47 (s, 3H), 1.39 (s, 1.5H).
^{13}C NMR (CD_2Cl_2, 150.92 MHz) δ 148.40, 148.17, 148.10, 147.79, 144.97, 144.73, 140.80, 140.63, 140.37, 140.23, 140.11, 139.51, 132.80, 132.76, 129.03, 129.00, 128.81, 128.66, 128.02, 127.98, 127.59, 127.55, 127.22, 127.05, 123.96, 123.70, 87.10, 86.90, 65.49, 65.18, 57.97, 36.57, 36.43, 31.10, 30.96.
FTIR (υ_{max} cm^{-1}) 3054, 1592, 1330, 1156, 1050, 960, 859.
HRMS (ESI+) Calculated for $C_{22}H_{23}N_2O_3S$: 395.1451; Found $(M+H)^+$: 395.1453.

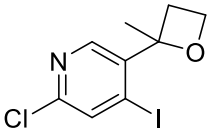
N-((3-(2-methyloxetan-2-yl)pyridin-4-yl)(phenyl)methyl)aniline (2l)



Synthesized according to the general procedure B by using diphenyl imine (2 mmol, 362 mg, 3 eq.) as electrophile and isolated as a white solid (254 mg, 77% yield) by column chromatography over silica gel (Petr. ether/EtOAc = 40/60) as a mixture of diastereomers (dr 1:0.2).

Rf 0.3 (Petr. ether/EtOAc = 40/60). White solid. ***Mp*** : 178 °C (EtOAc/Pet. Ether : 95/5).
^{1}H NMR (CD_2Cl_2, 600.13 MHz) δ 8.57 (s, 1H), 8.54 (s, 0.17H), 8.43 (d, *J* = 5.2 Hz, 0.17H), 8.42 (d, *J* = 5.1 Hz, 1H), 7.39 (d, *J* = 7.4 Hz, 2H), 7.37 (d, *J* = 5.2 Hz, 0.17H), 7.37 – 7.31 (m, 2H), 7.31 – 7.24 (m, 1.5H), 7.15 (d, *J* = 5.1 Hz, 1H), 7.14 – 7.05 (m, 2.18H), 6.68 (t, *J* = 7.3 Hz, 1H), 6.60 (d, *J* = 0.8 Hz, 0.36H), 6.53 (d, *J* = 7.7 Hz, 2H), 6.09 (d, *J* = 4.6 Hz, 0.18H), 5.97 (d, *J* = 5.5 Hz, 1H), 4.70 – 4.61 (m, 1.18H), 4.53 (dt, *J* = 9.2, 6.0 Hz, 0.18H), 4.42 (dt, *J* = 9.2, 6.0 Hz, 1H), 4.36 (d, *J* = 4.8 Hz, 0.18H), 4.30 (d, *J* = 5.4 Hz, 1H), 3.18 (ddd, *J* = 10.8, 9.2, 7.2 Hz, 1H), 3.08 (ddd, *J* = 10.5, 9.2, 7.2 Hz, 0.18H), 2.93 (ddd, *J* = 10.7, 8.4, 6.2 Hz, 0.18H), 2.84 (ddd, *J* = 10.9, 8.4, 6.1 Hz, 1H), 1.80 (s, 3H), 1.53 (s, 0.54H).
^{13}C NMR (CD_2Cl_2, 150.92 MHz) δ 149.56, 149.20, 147.99, 147.85, 147.68, 147.34, 147.23, 142.34, 142.17, 141.02, 140.76, 129.73, 129.68, 129.23, 129.14, 128.62, 128.22, 127.90, 127.88, 123.47, 123.02, 118.34, 118.25, 113.79, 113.72, 87.37, 86.74, 65.86, 65.70, 57.54, 57.09, 37.03, 35.65, 31.89, 31.00.
FTIR (υ_{max} cm^{-1}) 3315, 1602, 1588, 1498, 1318, 947, 747.
HRMS (ESI+) Calculated for $C_{22}H_{23}N_2O_2$: 331.1802; Found $(M+H)^+$: 331.1816.

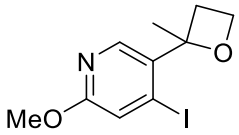
2-chloro-4-iodo-5-(2-methyloxetan-2-yl)pyridine (8)



Synthesized according to the general procedure B with iodine (2 mmol, 507 mg, 2 eq.) and isolated as a yellow solid (188 mg, 61% yield) by column chromatography over silica gel (Petr. ether/EtOAc = 90/10).

Rf 0.35 (Petr. ether/EtOAc = 90/10). Yellow solid.
^{1}H NMR ($CDCl_3$, 600.13 MHz) δ 8.54 (s, 1H), 7.81 (s, 1H), 4.64 (dt, *J* = 8.4, 6.4 Hz, 1H), 4.44 (dt, *J* = 9.0, 6.4 Hz, 1H), 2.96 (ddd, *J* = 11.4, 8.4, 6.7 Hz, 1H), 2.90 (ddd, *J* = 11.4, 9.0, 6.8 Hz, 1H), 1.81 (s, 3H).
^{13}C NMR ($CDCl_3$, 150.90 MHz) δ 149.88, 146.63, 143.90, 134.55, 104.67, 87.63, 65.43, 34.96, 28.61.
FTIR (υ_{max} cm^{-1}) 2971, 2886, 1526, 1439, 1263, 1111, 1089, 958, 872, 761.
HRMS (ESI+) Calculated for $C_9H_{10}ClINO$: 309.9512; Found $(M+H)^+$: 309.9521.

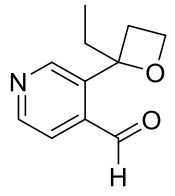
4-iodo-2-methoxy-5-(2-methyloxetan-2-yl)pyridine (9)



Synthesized according to the general procedure B with iodine (2 mmol, 507 mg, 2 eq.) and Isolated as a yellow solid (234 mg, 77% yield) by column chromatography over silica gel (Petr. ether/EtOAc = 90/10).

Rf 0.4 (Petr. ether/EtOAc = 90/10). Yellow oil.
^{1}H NMR ($CDCl_3$, 600.13 MHz) δ 8.32 (s, 1H), 7.27 (s, 1H), 4.61 (ddd, *J* = 8.4, 6.7, 6.0 Hz, 1H), 4.43 (ddd, *J* = 8.9, 6.7, 6.0 Hz, 1H), 3.90 (s, 3H), 2.93 – 2.83 (m, 2H), 1.80 (s, 3H).
^{13}C NMR ($CDCl_3$, 150.90 MHz) δ 163.11, 143.50, 137.22, 121.95, 104.47, 87.89, 65.38, 53.84, 35.17, 29.35.
FTIR (υ_{max} cm^{-1}) 2968, 2882, 1573, 1470, 1345, 1278, 1213, 1017, 966, 852.
HRMS (ESI+) Calculated for $C_{10}H_{13}INO_2$: 305.9980; Found $(M+H)^+$: 305.9968.

3-(2-ethyloxetan-2-yl)isonicotinaldehyde (10)



Synthesized according to the general procedure B with dimethylformamide (3 mmol, 219 mg, 3 eq.) as electrophile and isolated as a yellow oil (134 mg, 70% yield) by column chromatography over silica gel (Petr. ether/EtOAc = 40/60).

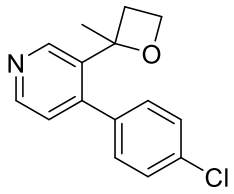
Rf 0.42 (Petr. ether/EtOAc = 40/60). Yellow oil.

^{1}H NMR ($CDCl_3$, 600.13 MHz) δ 10.33 (s, 1H), 8.76 (s, 1H), 8.74 (d, *J* = 4.9 Hz, 1H), 7.64 (dd, *J* = 5.0, 0.6 Hz, 1H), 4.61 (dt, *J* = 8.7, 6.4 Hz, 1H), 4.55 – 4.50 (m, 1H), 3.07 (ddd, *J* = 11.3, 8.7, 6.7 Hz, 1H), 2.84 (ddd, *J* = 11.3, 9.1, 6.7 Hz, 1H), 2.17 (dq, *J* = 14.7, 7.4 Hz, 1H), 2.01 (dq, *J* = 14.8, 7.4 Hz, 1H), 0.94 (t, *J* = 7.4 Hz, 3H).
^{13}C NMR ($CDCl_3$, 150.90 MHz) δ 192.16, 149.65, 148.74, 141.47, 138.56, 122.11, 89.06, 66.06, 36.60, 34.19, 7.77.
FTIR (υ_{max} cm^{-1}) 2969, 2884, 1700, 1461, 1220, 963, 831, 729.
HRMS (ESI+) Calculated for $C_{11}H_{14}NO_2$: 192.1025; Found $(M+H)^+$: 192.1031.

IV. Suzuki coupling of iodide 2e.

General procedure C: To **2e** (0.5 mmol, 137.5 mg, 1eq.), K_2CO_3 (1.5 mmol, 207 mg, 3 eq.), tetrakis(triphenylphosphine)palladium (0.025 mmol, 29 mg, 5 mol %) and arylboronic acid (0.75 mmol, 1.5 eq.) was added toluene (4 mL) and H_2O (2 mL), the mixture was then heated at 120 °C for 1 hour with a vigorous stirring. The mixture was cooled to room temperature and mixed with ethyl acetate (10 mL) and $NaHCO_{3aq}$ (10 mL), the organic layer was separated and the aqueous layer extracted with ethyl acetate (2x10mL). Organic layers were combined, dried over Na_2SO_4 and solvent were removed in vacuo. The resulting crude mixture was purified by column chromatography over silica gel (petroleum ether/ethyl acetate) to afford the expected compound.

4-(4-chlorophenyl)-3-(2-methyloxetan-2-yl)pyridine (11)

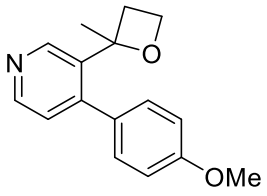


Synthesized according to the general procedure C with 4-chlorophenylboronic acid (0.75, 117 mg, 1.5 eq.) and isolated as a slightly yellow solid (106 mg, 82% yield) by column chromatography over silica gel (Petr. ether/EtOAc = 30/70).

Rf 0.34 (Petr. ether/EtOAc = 30/70). Slightly yellow solid.
^{1}H NMR ($CDCl_3$, 600.13 MHz) δ 8.89 (s, 1H), 8.54 (d, *J* = 4.9 Hz, 1H), 7.39 (d, *J* = 8.5 Hz, 2H), 7.17 (d, *J* = 8.5 Hz, 2H), 7.04 (d, *J* = 4.9 Hz, 1H), 4.57 (td, *J* = 7.7, 6.1 Hz, 1H), 4.38 (dt, *J* = 9.3, 5.9 Hz, 1H), 2.74 (ddd, *J* = 10.9, 9.2, 7.6 Hz, 1H), 2.34 (ddd, *J* = 11.0, 8.2, 5.9 Hz, 1H), 1.64 (s, 3H).

^{13}C NMR ($CDCl_3$, 150.92 MHz) δ 148.27, 147.47, 145.20, 140.55, 138.23, 134.33, 130.08, 128.55, 125.22, 86.00, 65.15, 36.05, 31.04.
FTIR (υ_{max} cm^{-1}) 2972, 2883, 1598, 1473, 1403, 1090, 965, 826.
HRMS (ESI+) Calculated for $C_{15}H_{15}ClNO$: 260.0856; Found $(M+H)^+$: 260.0866.

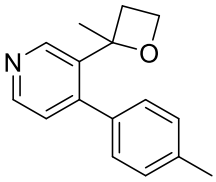
4-(4-methoxyphenyl)-3-(2-methyloxetan-2-yl)pyridine (12)



Synthesized according to the general procedure C with 4-methoxylbenzeneboronic acid (0.75, 114 mg, 1.5 eq.) and isolated as a yellow oil (108 mg, 85% yield) by column chromatography over silica gel (Petr. ether/EtOAc = 20/80).

Rf 0.37 (Petr. ether/EtOAc = 20/80). Colourless oil.
^{1}H NMR ($CDCl_3$, 600.13 MHz) δ 8.89 (s, 1H), 8.51 (d, *J* = 5.0 Hz, 1H), 7.14 (d, *J* = 8.7 Hz, 2H), 7.06 (d, *J* = 5.0 Hz, 1H), 6.93 (d, *J* = 8.7 Hz, 2H), 4.56 (td, *J* = 7.8, 6.1 Hz, 1H), 4.38 (dt, *J* = 9.2, 5.9 Hz, 1H), 3.85 (s, 3H), 2.73 (ddd, *J* = 11.0, 9.2, 7.6 Hz, 1H), 2.31 (ddd, *J* = 11.1, 8.2, 5.8 Hz, 1H), 1.65 (s, 3H).
^{13}C NMR ($CDCl_3$, 150.90 MHz) δ 159.49, 147.99, 147.21, 146.24, 140.80, 131.94, 129.87, 125.62, 113.73, 86.20, 65.13, 55.42, 35.92, 30.88.
FTIR (υ_{max} cm^{-1}) 2968, 2885, 1611, 1515, 1477, 1247, 1177, 1042, 907, 729.
HRMS (ESI+) Calculated for $C_{16}H_{18}NO_2$: 256.1343; Found $(M+H)^+$: 256.1346.

3-(2-methyloxetan-2-yl)-4-(p-tolyl)pyridine (13)



Synthesized according to the general procedure C with 4-methylbenzeneboronic acid (0.75, 102 mg, 1.5 eq.) and isolated as a colorless oil (104 mg, 87% yield) by column chromatography over silica gel (Petr. ether/EtOAc = 30/70).

Rf 0.34 (Petr. ether/EtOAc = 30/70). colorless oil.

[1]H NMR ($CDCl_3$, 399.57 MHz) δ 8.91 (s, 1H), 8.53 (d, *J* = 5.0 Hz, 1H), 7.22 (d, *J* = 7.9 Hz, 2H), 7.11 (d, *J* = 8.0 Hz, 2H), 7.08 (d, *J* = 5.0 Hz, 1H), 4.57 (dt, *J* = 13.9, 7.0 Hz, 1H), 4.39 (dt, *J* = 9.2, 5.9 Hz, 1H), 2.74 (ddd, *J* = 10.9, 9.2, 7.7 Hz, 1H), 2.41 (s, 3H), 2.31 (ddd, *J* = 11.1, 8.2, 5.9 Hz, 1H), 1.64 (s, 3H).

[13]C NMR ($CDCl_3$, 100.47 MHz) δ 147.99, 147.17, 146.45, 140.65, 137.91, 136.77, 128.95, 128.52, 125.44, 86.19, 65.10, 35.95, 30.88, 21.30.

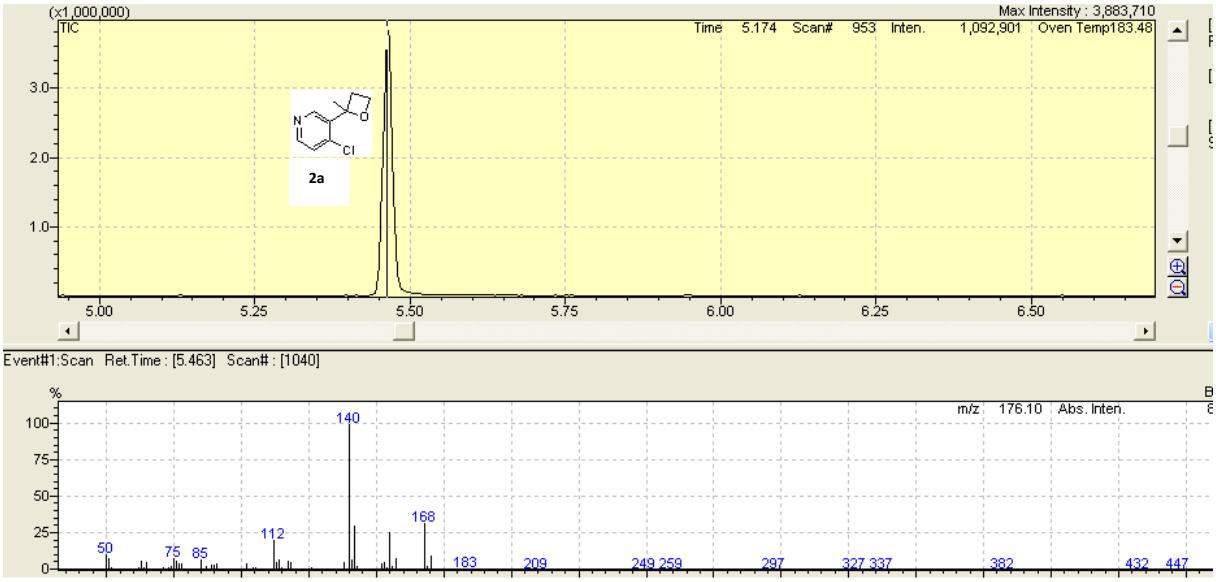
FTIR (υ_{max} cm^{-1}) 2966, 2880, 1586, 1476, 1401, 1253, 1083, 964, 817.

HRMS (ESI+) Calculated for $C_{16}H_{18}NO$: 240.1380; Found $(M+H)^+$: 240.1385.

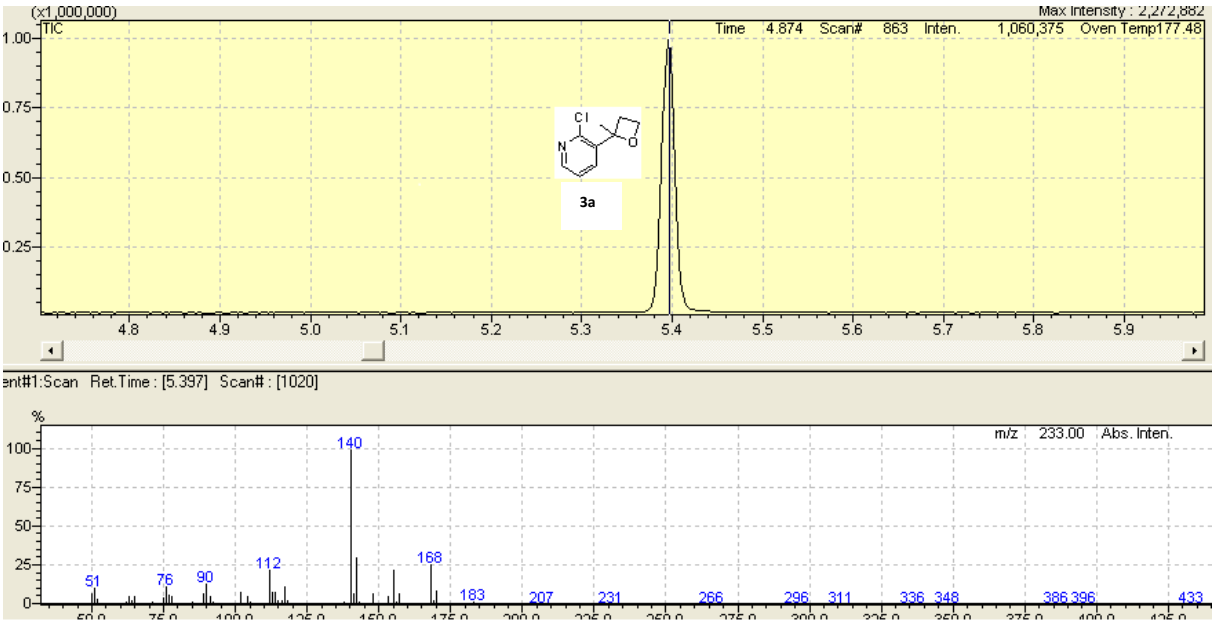
V. GC/MS analysis of the chlorination reaction

GC/MS analysis of the crude mixture of the reaction of **1** according to general procedure B.

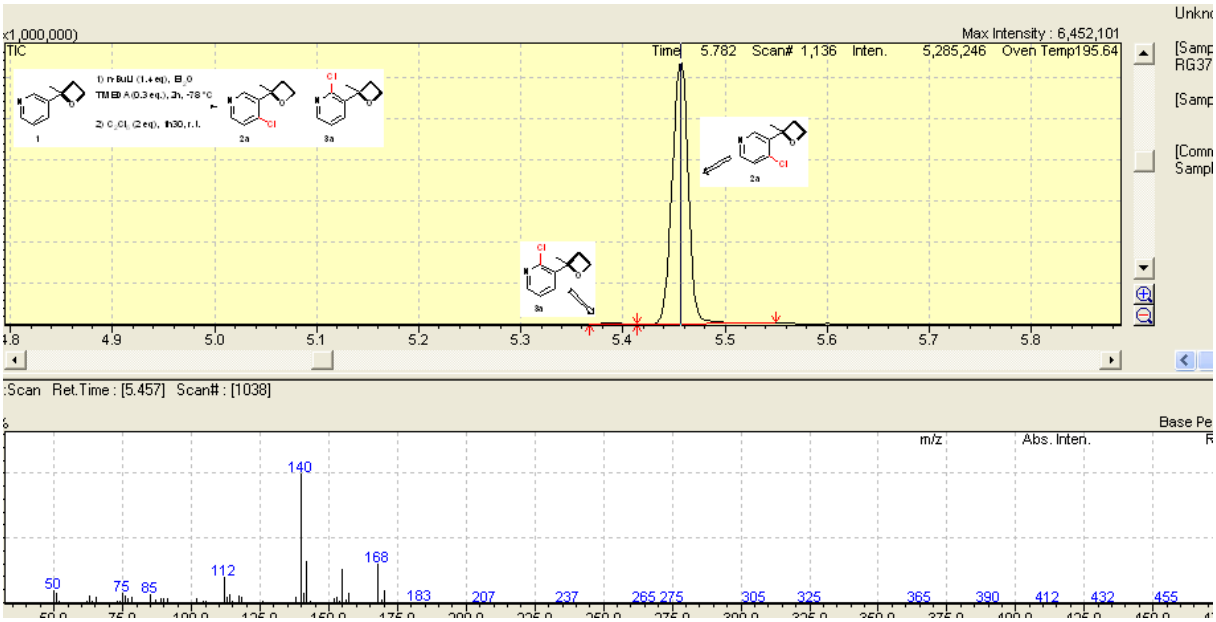
Reference **2a.**



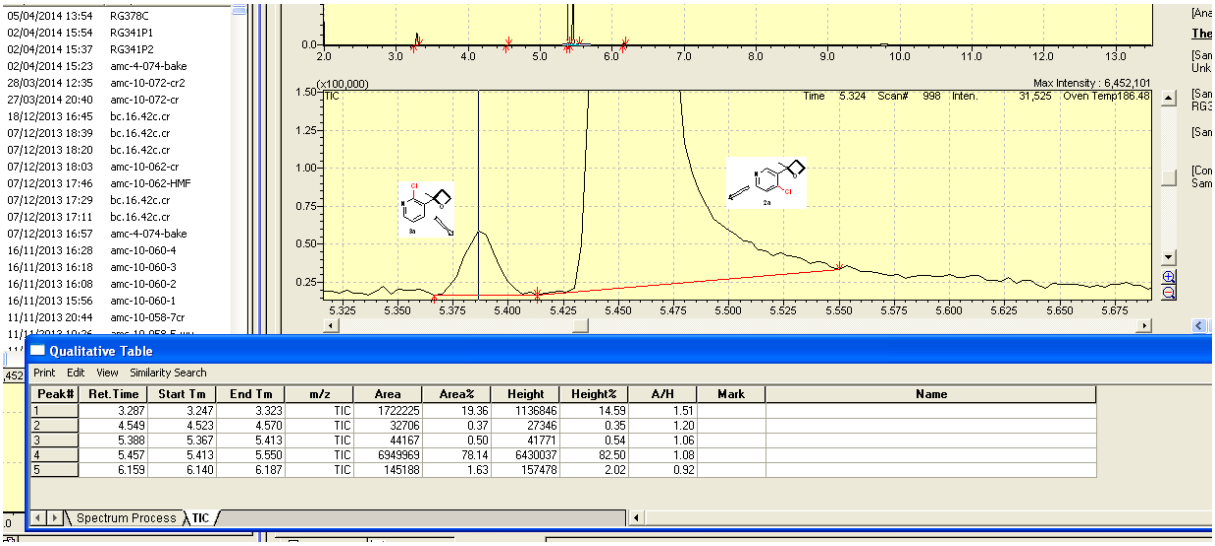
Reference **3a.**



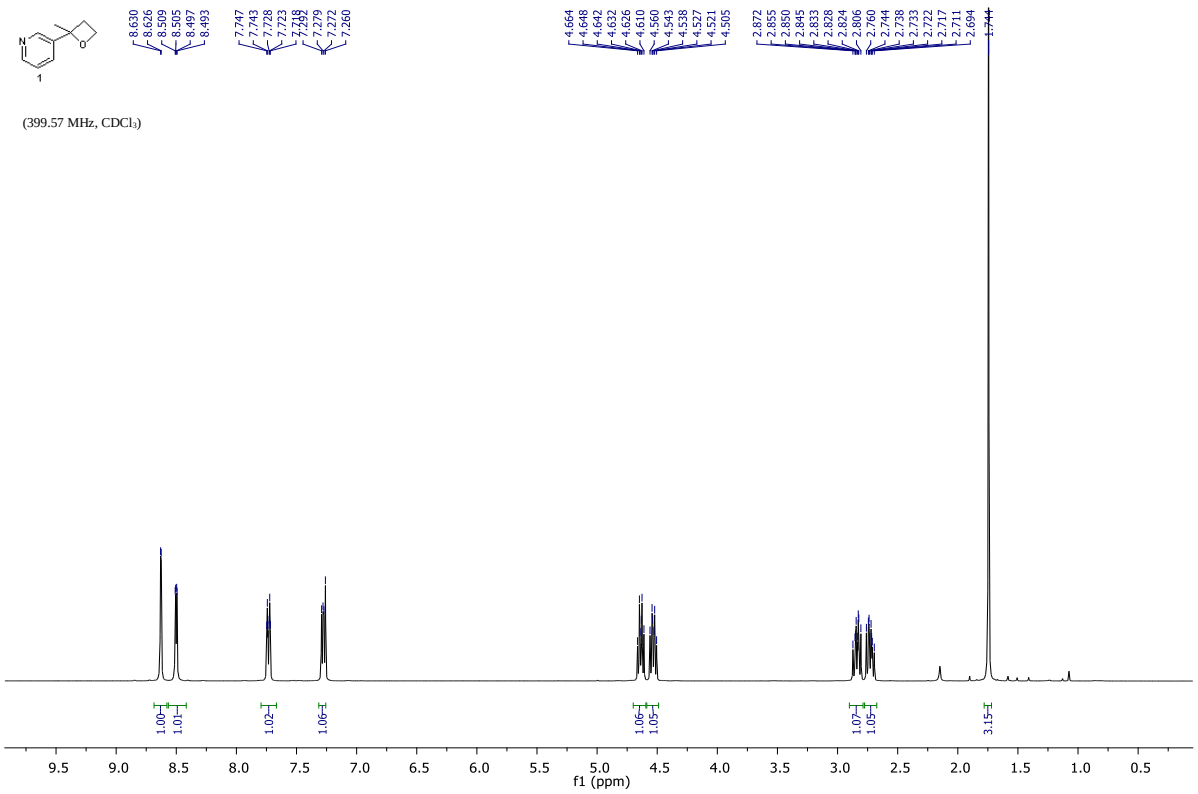
Crude mixture.

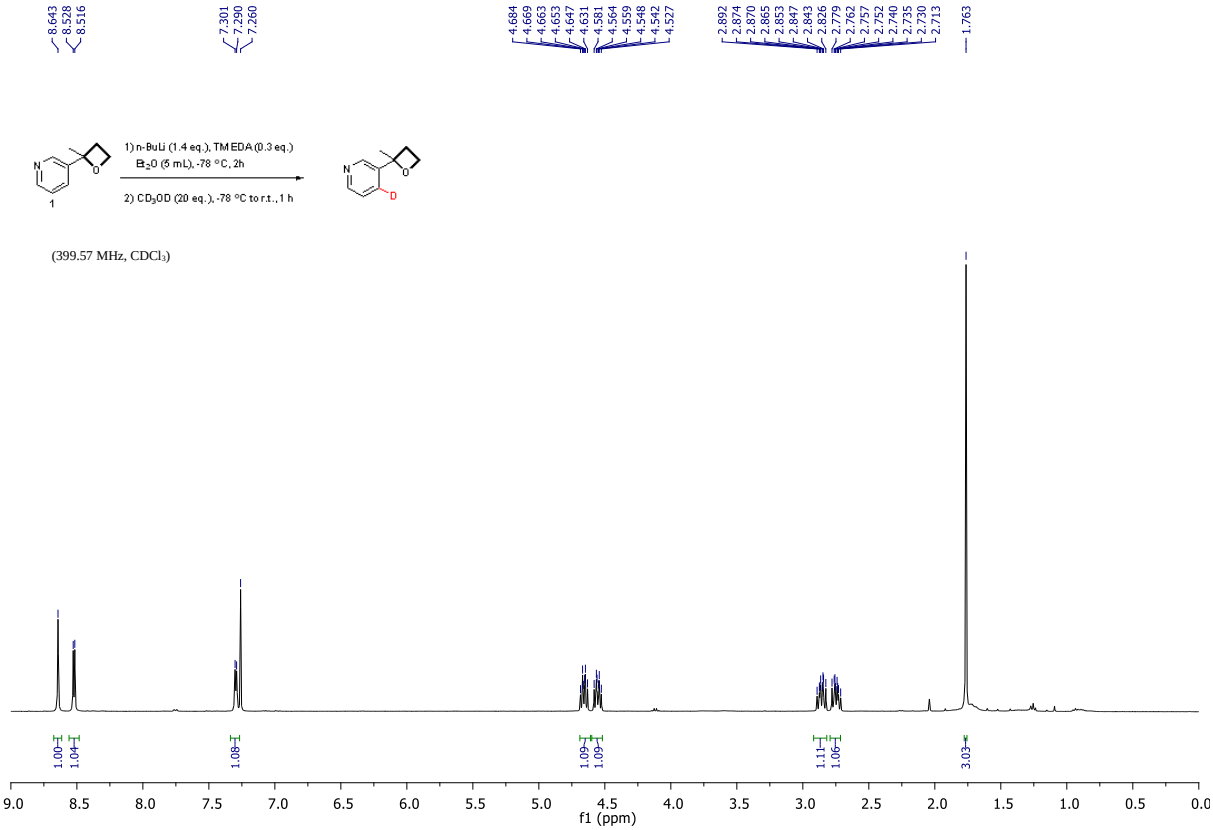


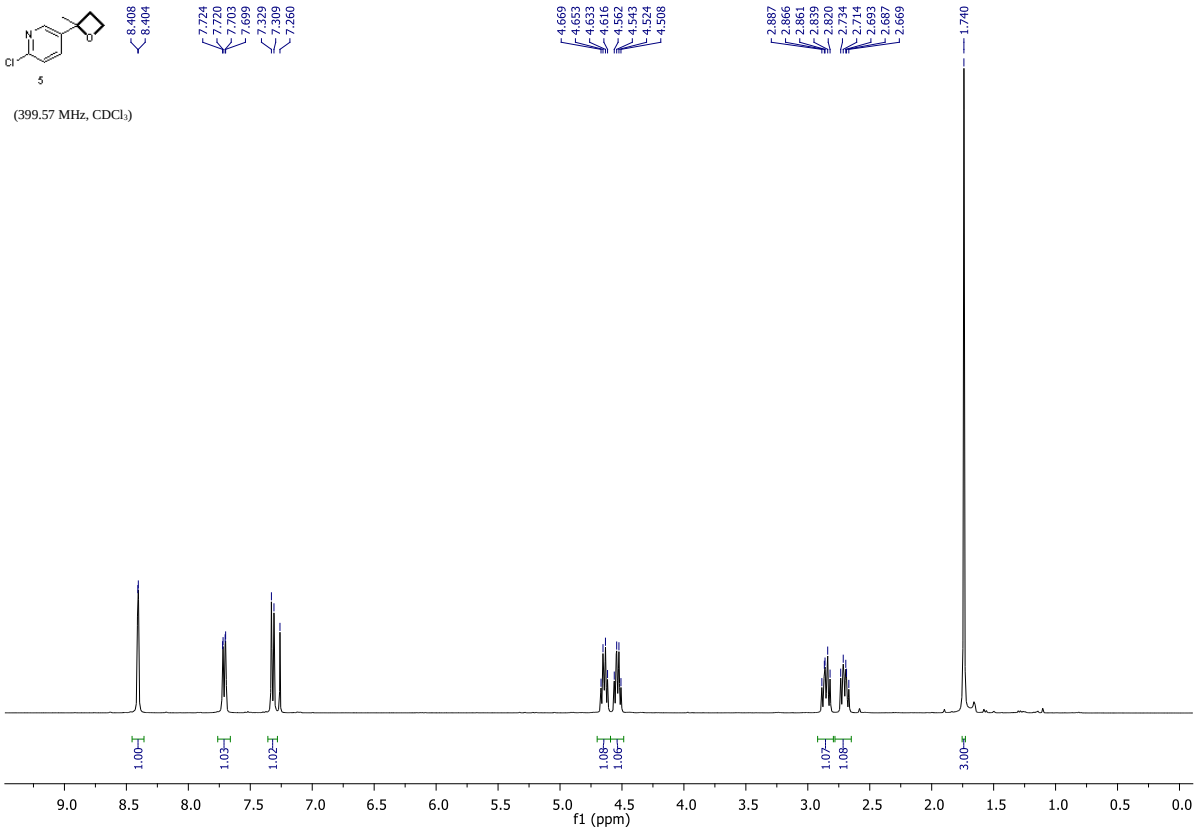
Zoom on the crude mixture.



VI. Deuterium labelled experiments

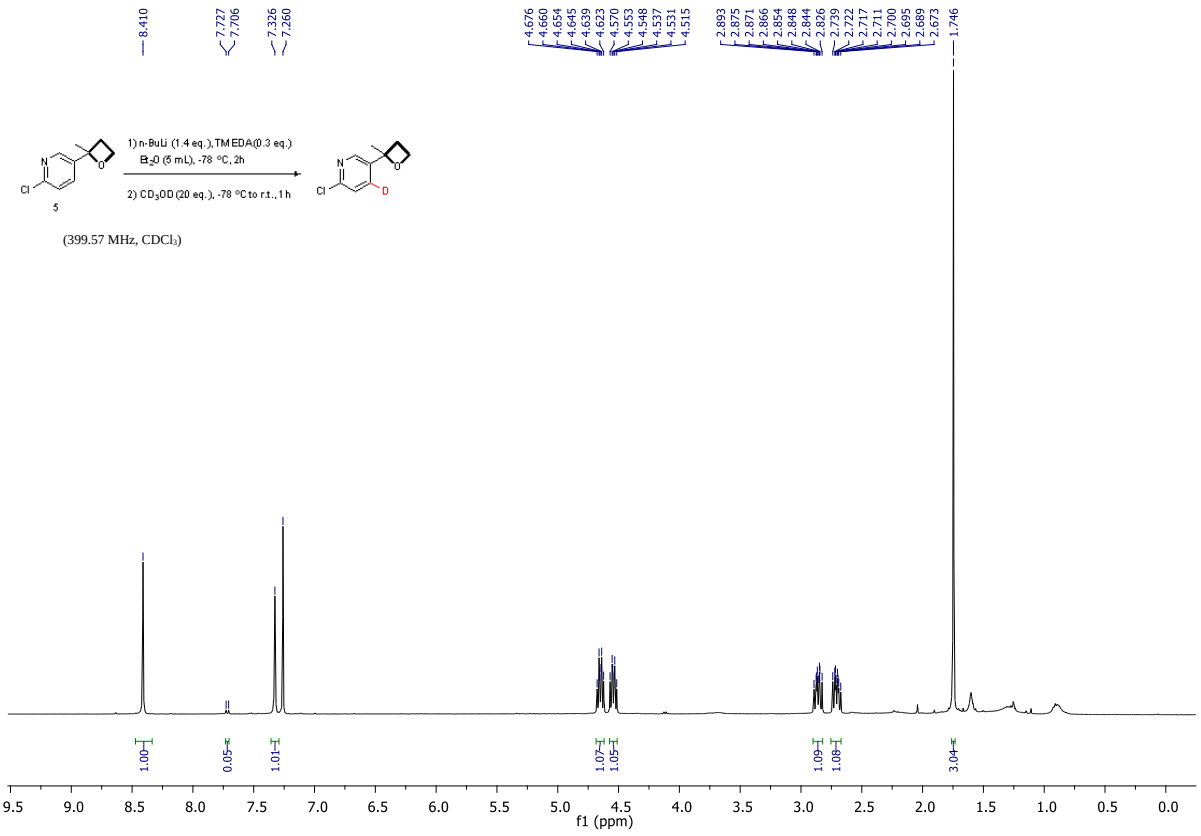






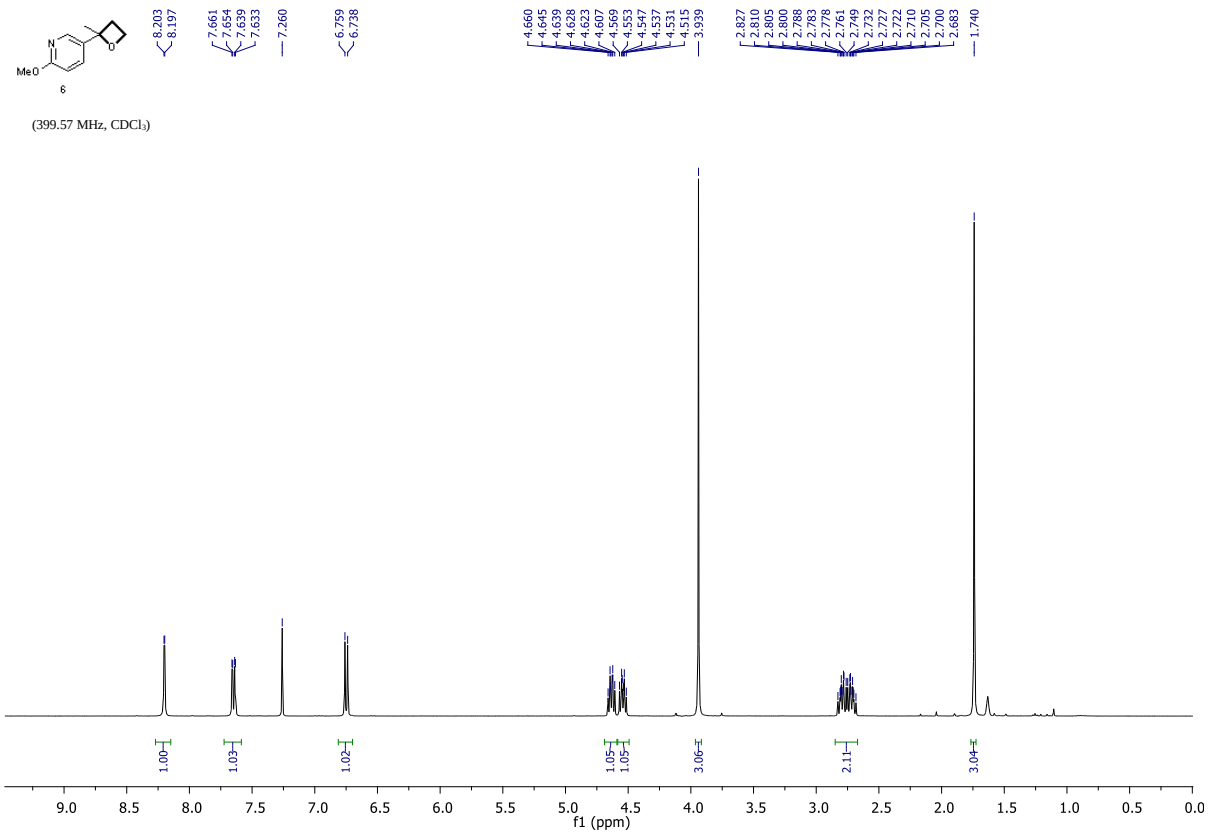
5

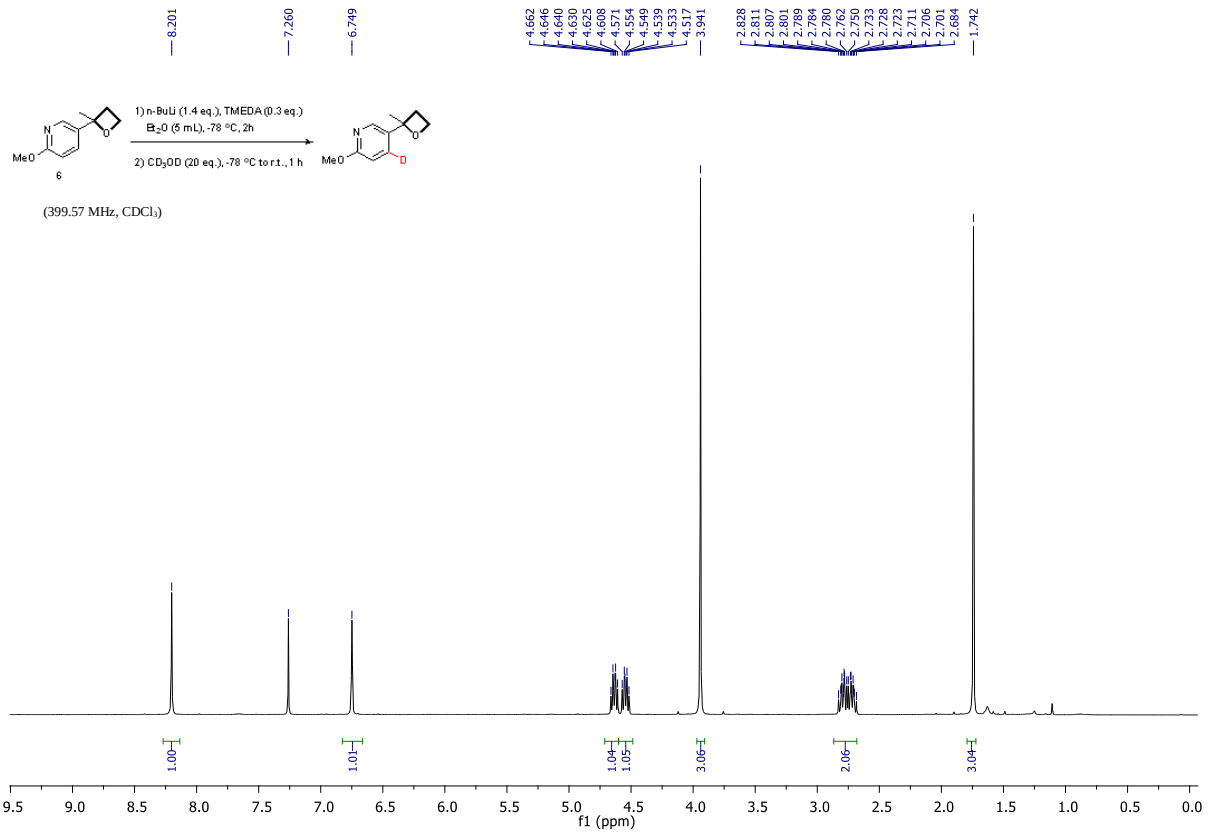
(399.57 MHz, $CDCl_3$)

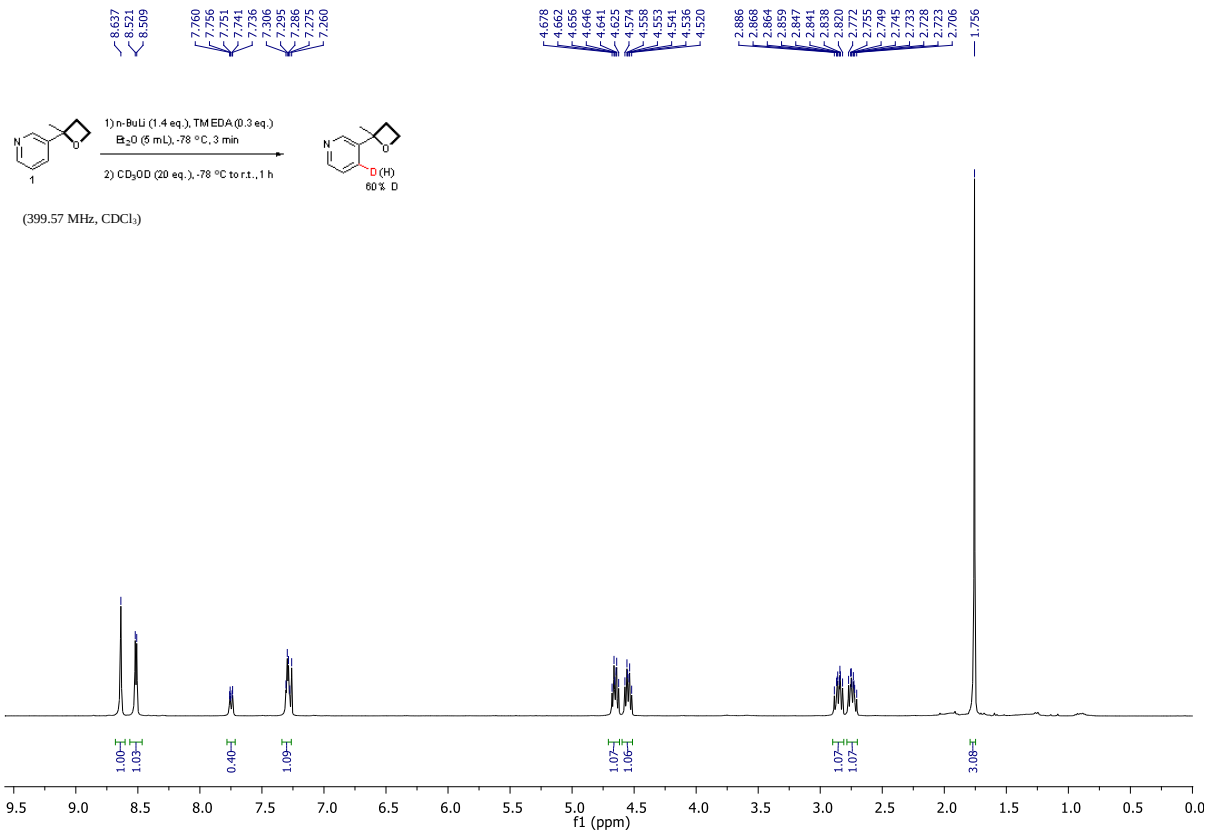


5

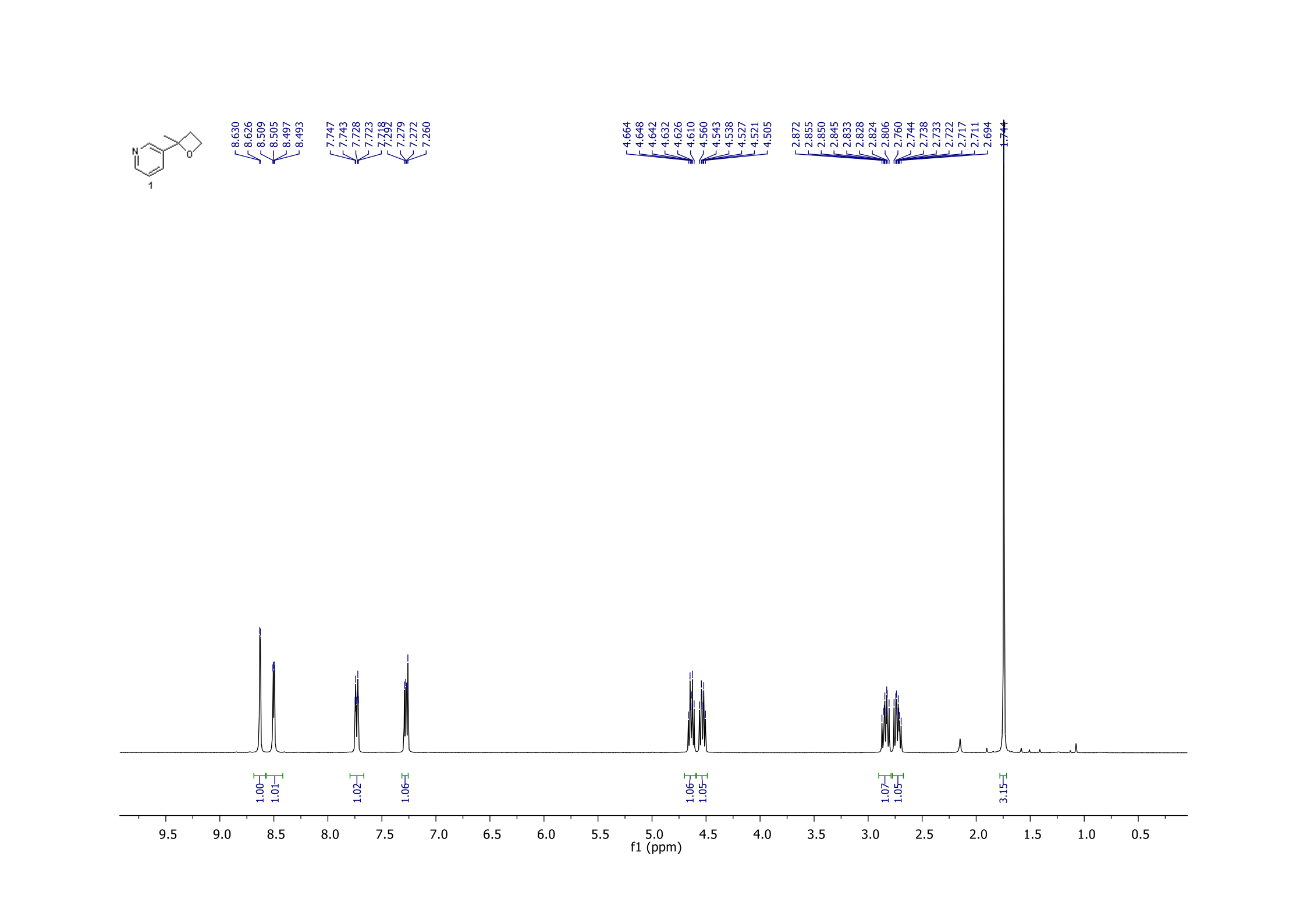
(399.57 MHz, $CDCl_3$)

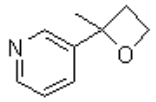

8.203
8.197
7.661
7.654
7.639
7.633
7.260
6.759
6.738
4.660
4.645
4.639
4.628
4.623
4.607
4.569
4.553
4.547
4.537
4.531
4.515
3.939
2.827
2.810
2.805
2.800
2.788
2.783
2.778
2.761
2.749
2.732
2.727
2.722
2.710
2.705
2.700
2.683
1.740
MeO
6
(399.57 MHz, CDCl3)
1.00
1.03
1.02
1.05
1.05
3.06
2.11
3.04
f1 (ppm)


8.201
7.260
6.749
4.662
4.646
4.640
4.630
4.625
4.608
4.571
4.554
4.549
4.539
4.533
4.517
3.941
2.828
2.811
2.807
2.801
2.789
2.784
2.780
2.762
2.750
2.733
2.728
2.723
2.711
2.706
2.701
2.684
1.742
1) n-BuLi (1.4 eq.), TMEDA (0.3 eq.)
Et2O (5 mL), -78 °C, 2h
2) CD3OD (20 eq.), -78 °C to r.t., 1 h
MeO
6
D
(399.57 MHz, CDCl3)
1.00
1.01
1.04
1.05
3.06
2.06
3.04
f1 (ppm)


8.637
8.521
8.509
7.760
7.756
7.751
7.741
7.736
7.306
7.295
7.286
7.275
7.260
4.678
4.662
4.656
4.646
4.641
4.625
4.574
4.558
4.553
4.541
4.536
4.520
2.886
2.868
2.864
2.859
2.847
2.841
2.838
2.820
2.772
2.755
2.749
2.745
2.733
2.728
2.723
2.706
1.756
1) n-BuLi (1.4 eq.), TMEDA (0.3 eq.)
Et2O (5 mL), -78 °C, 3 min
2) CD3OD (20 eq.), -78 °C to r.t., 1 h
1
D (H)
60 % D
(399.57 MHz, CDCl3)
1.00
1.03
0.40
1.09
1.07
1.06
1.07
1.07
3.08
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
0.0
f1 (ppm)

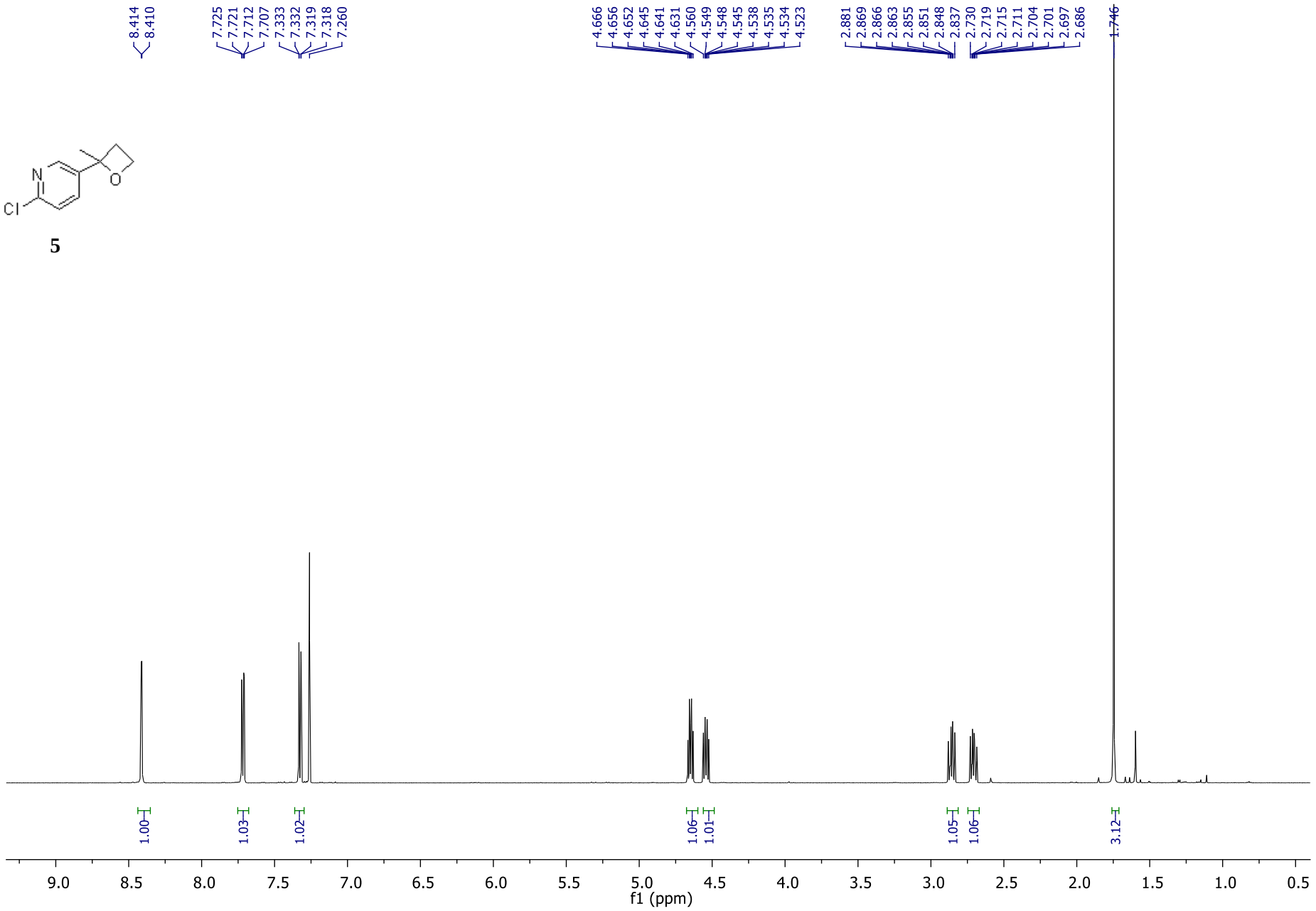
VII. Copies of ^{1}H and ^{13}C NMR Spectra


8.630
8.626
8.509
8.505
8.497
8.493
7.747
7.743
7.728
7.723
7.718
7.292
7.279
7.272
7.260
4.664
4.648
4.642
4.632
4.626
4.610
4.560
4.543
4.538
4.527
4.521
4.505
2.872
2.855
2.850
2.845
2.833
2.828
2.824
2.806
2.760
2.744
2.738
2.733
2.722
2.717
2.711
2.694
1.744
1.00
1.01
1.02
1.06
1.06
1.05
1.07
1.05
3.15
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)
1

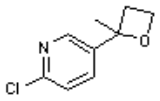

N
O

1

148.195
145.888
143.174
131.484
123.092
85.060
77.372
77.160
76.947
64.738
35.474
30.324
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

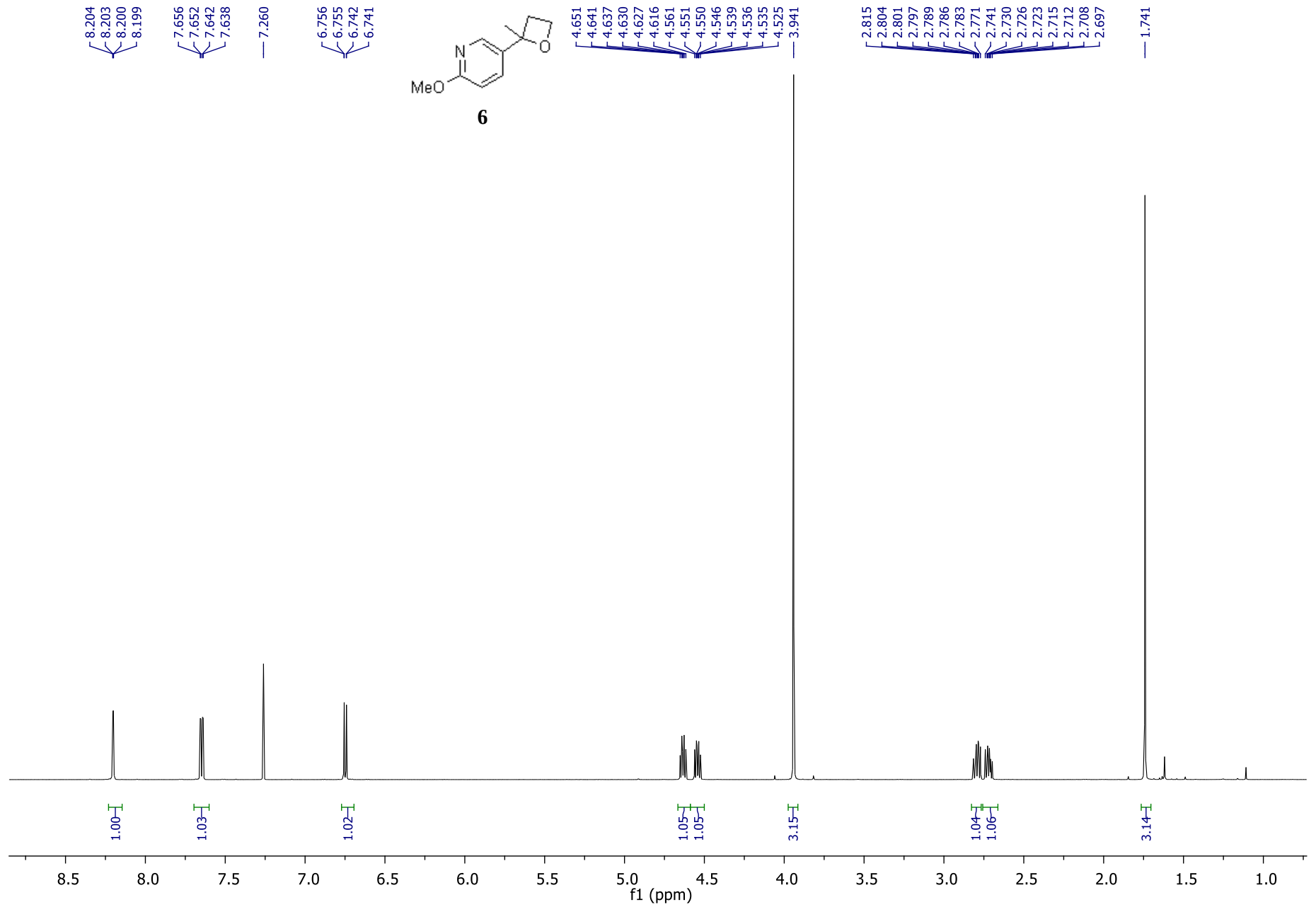

8.414
8.410
7.725
7.721
7.712
7.707
7.333
7.332
7.319
7.318
7.260
4.666
4.656
4.652
4.645
4.641
4.631
4.560
4.549
4.548
4.545
4.538
4.535
4.534
4.523
2.881
2.869
2.866
2.863
2.855
2.851
2.848
2.837
2.730
2.719
2.715
2.711
2.704
2.701
2.697
2.686
1.746
N
O
Cl
1.00
1.03
1.02
1.06
1.01
1.05
1.06
3.12
9.0
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)

5

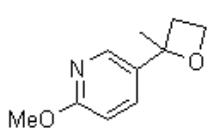

N
O
Cl

5

149.936
145.890
142.309
134.826
123.888
84.815
77.477
77.160
76.842
64.919
35.651
30.415
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

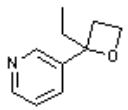

8.204
8.203
8.200
8.199
7.656
7.652
7.642
7.638
7.260
6.756
6.755
6.742
6.741
4.651
4.641
4.637
4.630
4.627
4.616
4.561
4.551
4.550
4.546
4.539
4.536
4.535
4.525
3.941
2.815
2.804
2.801
2.797
2.789
2.786
2.783
2.771
2.741
2.730
2.726
2.723
2.715
2.712
2.708
2.697
1.741
N
O
MeO
1.00
1.03
1.02
1.05
1.05
3.15
1.04
1.06
3.14
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
f1 (ppm)

6


N
O
MeO

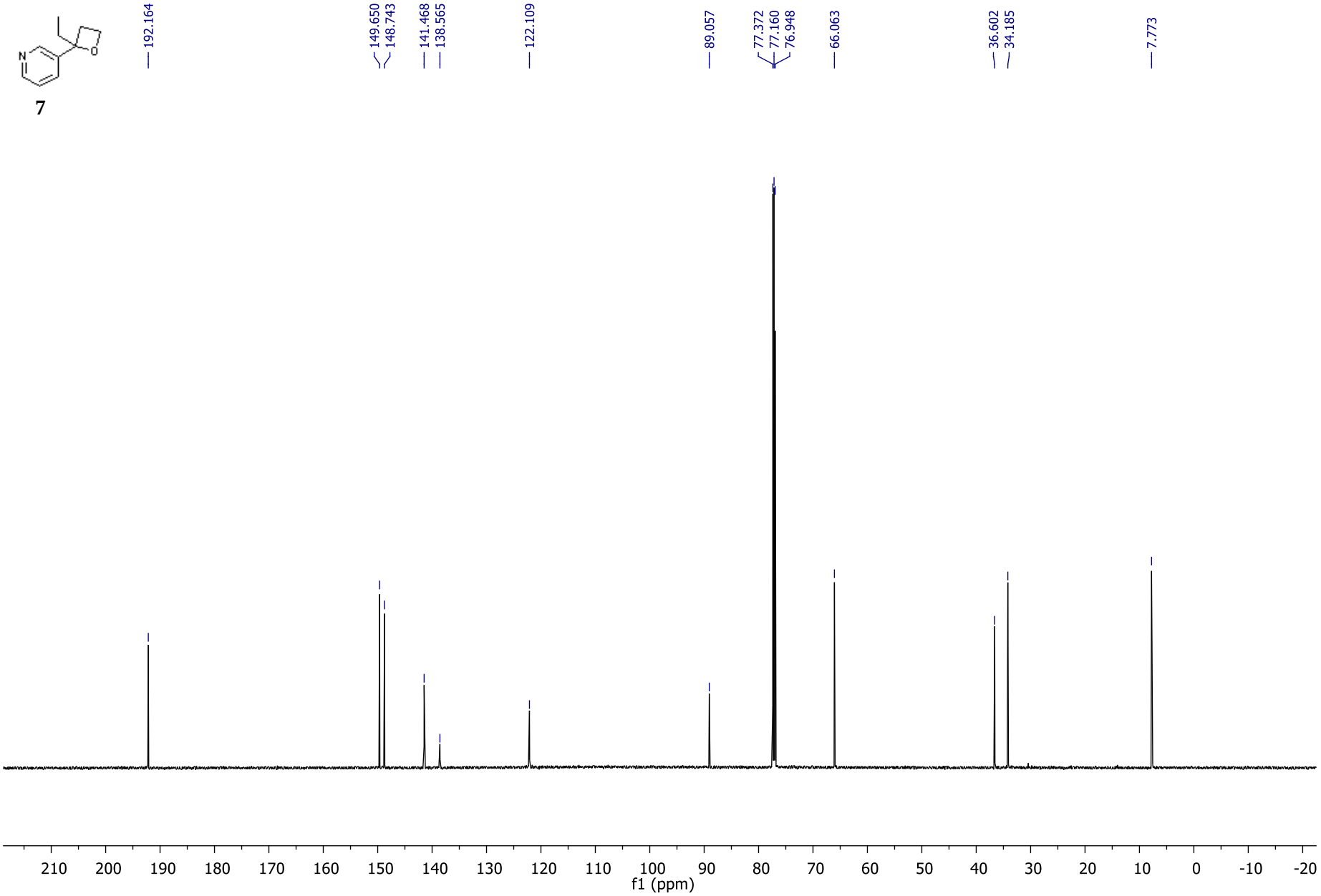
6

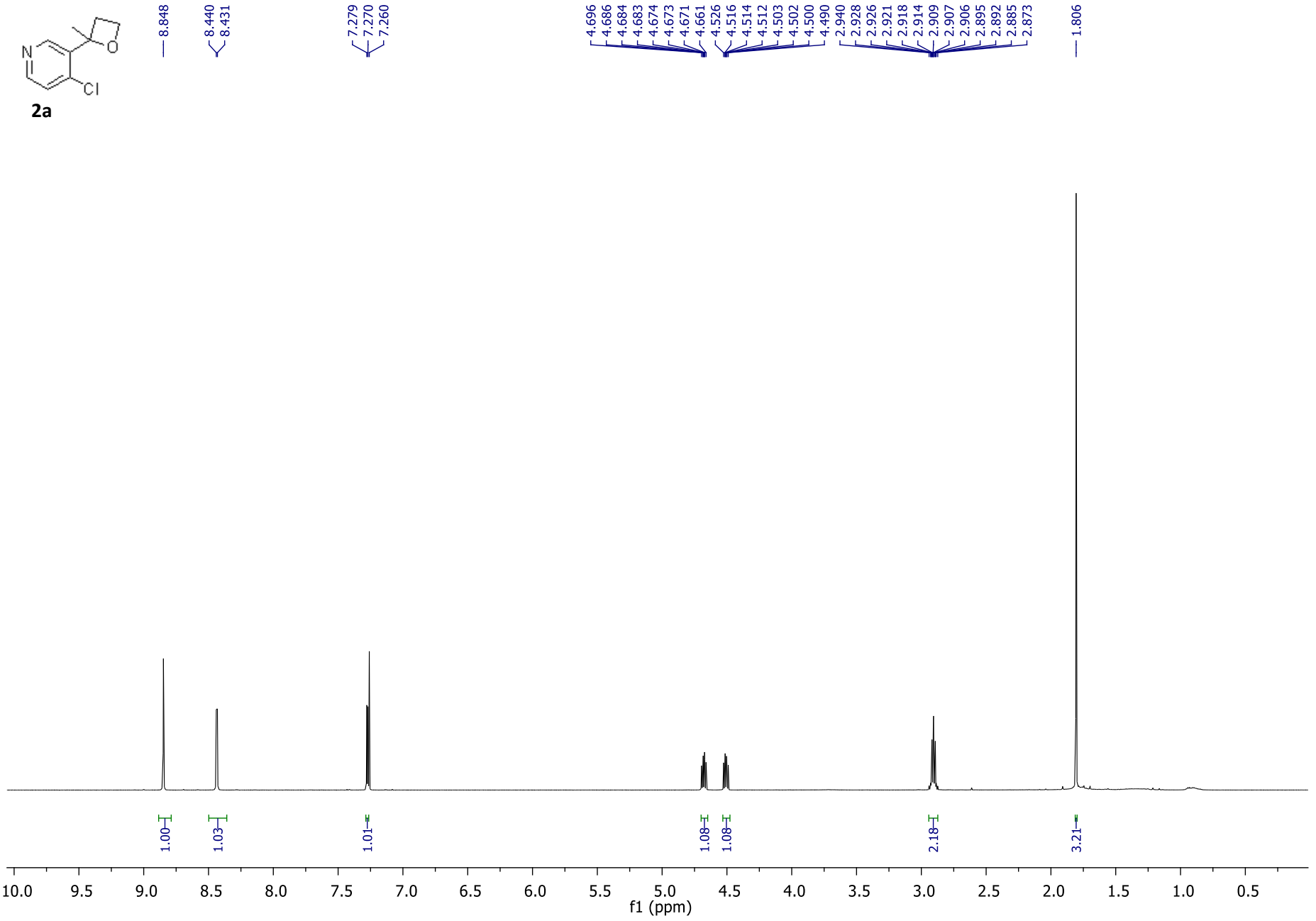
163.356
142.642
136.129
135.205
110.541
85.201
77.372
77.160
76.948
64.785
53.543
35.686
30.246
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



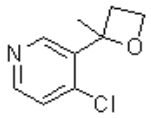
7

10.331
8.764
8.742
8.734
7.646
7.645
7.637
7.636
7.260
4.625
4.615
4.611
4.604
4.601
4.590
4.541
4.530
4.526
4.520
4.515
4.515
4.504
3.083
3.080
3.076
3.065
3.061
3.050
2.849
2.845
2.190
2.177
2.166
2.154
2.141
2.032
2.020
2.008
0.940
0.928
1.00
2.00
1.01
1.10
1.09
1.07
1.08
1.04
1.08
3.15
12.0
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
0.0
f1 (ppm)


192.164
149.650
148.743
141.468
138.565
122.109
89.057
77.372
77.160
76.948
66.063
36.602
34.185
7.773
N
O
7
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

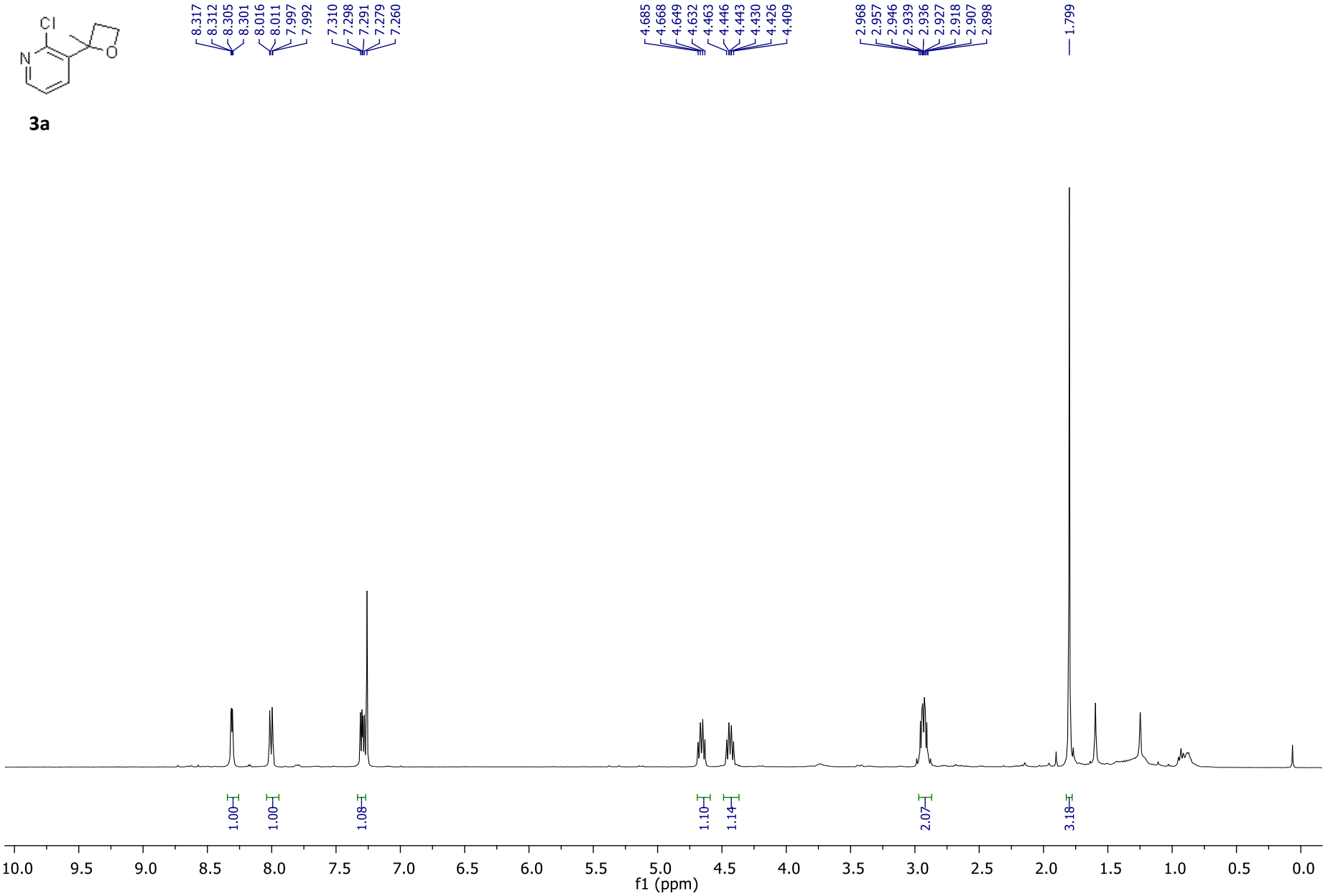

8.848
8.440
8.431
7.279
7.270
7.260
4.696
4.686
4.684
4.683
4.674
4.673
4.671
4.661
4.526
4.516
4.514
4.512
4.503
4.502
4.500
4.490
2.940
2.928
2.926
2.921
2.918
2.914
2.909
2.907
2.906
2.895
2.892
2.885
2.873
1.806
N
O
Cl
1.00
1.03
1.01
1.08
1.08
2.18
3.21
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

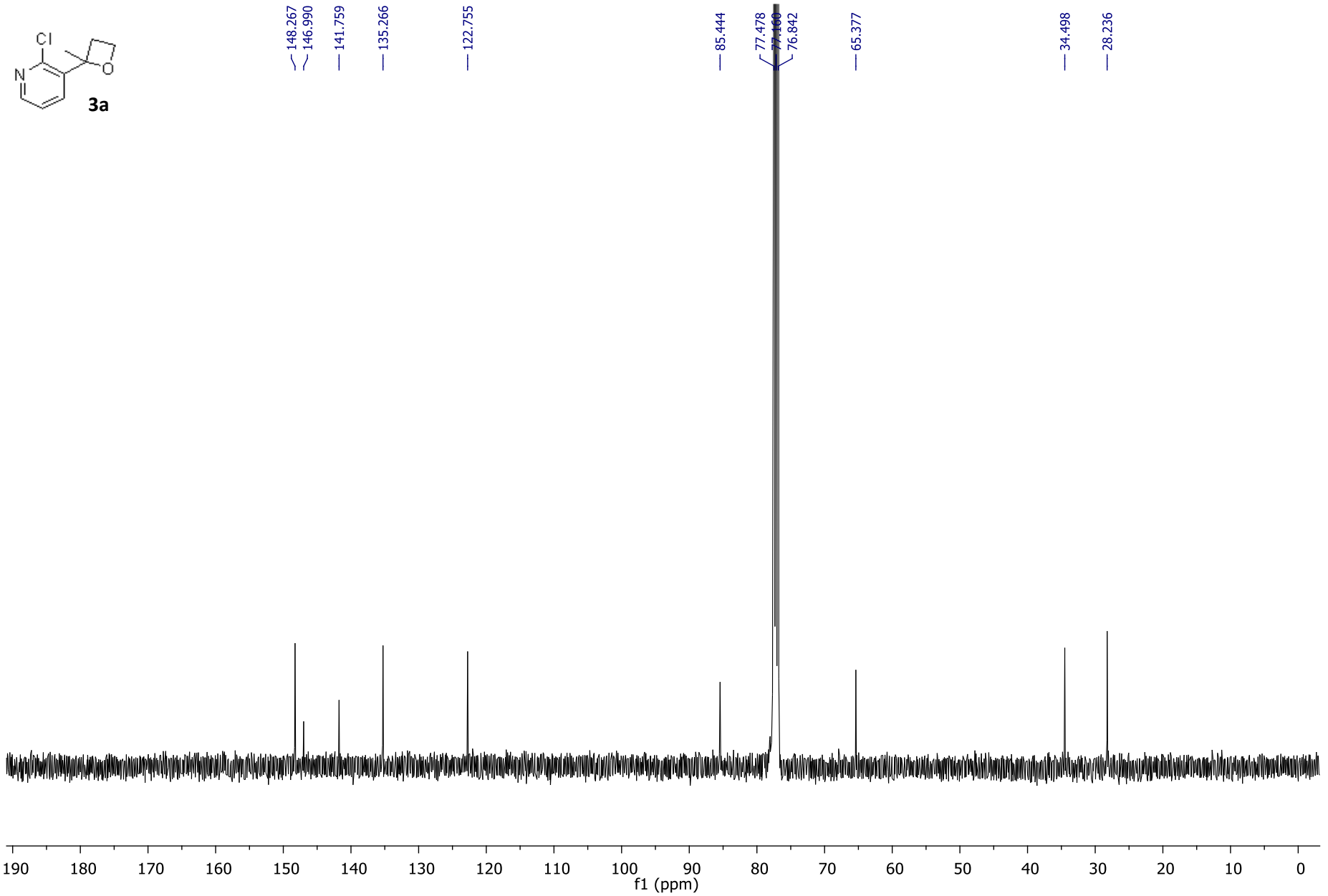
2a

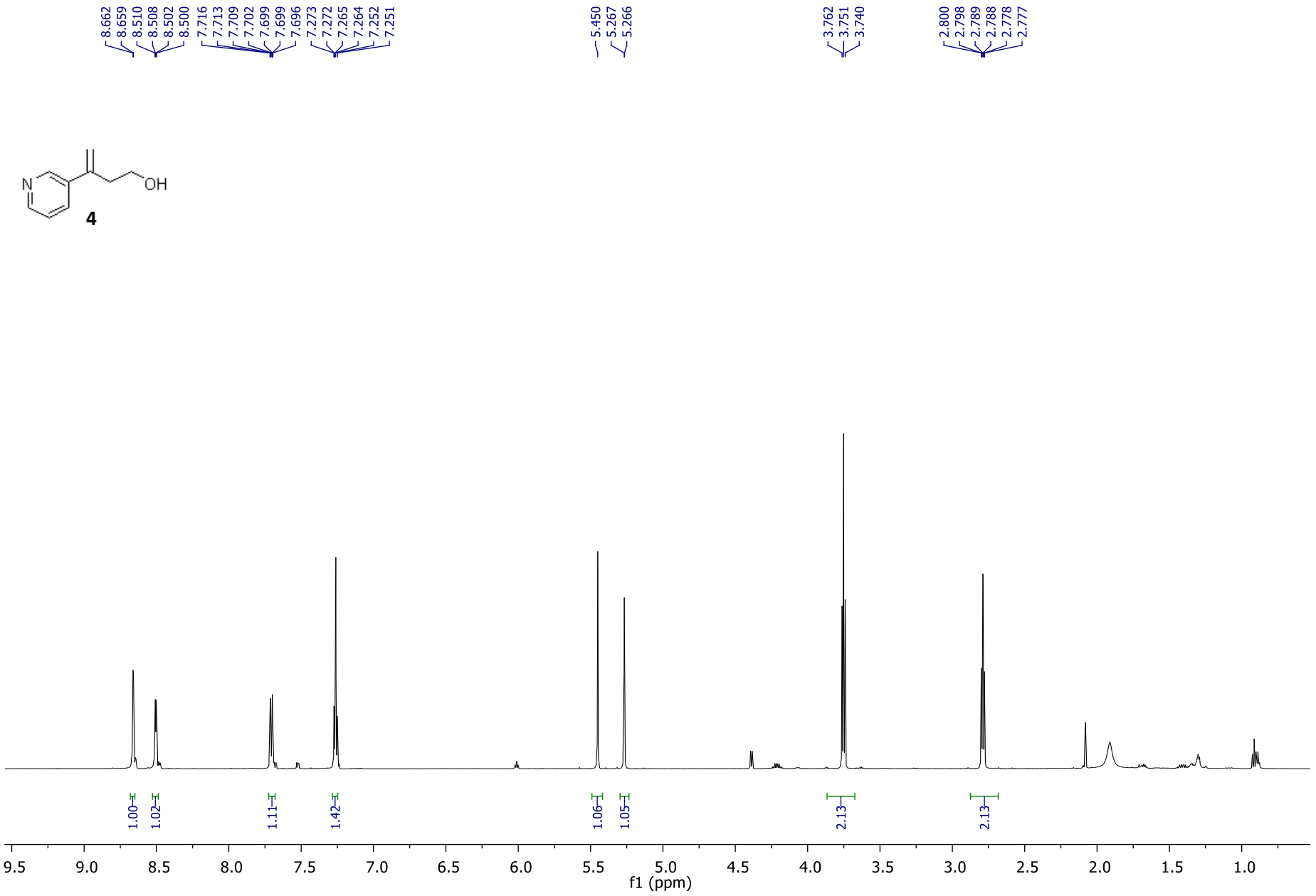

N
O
Cl

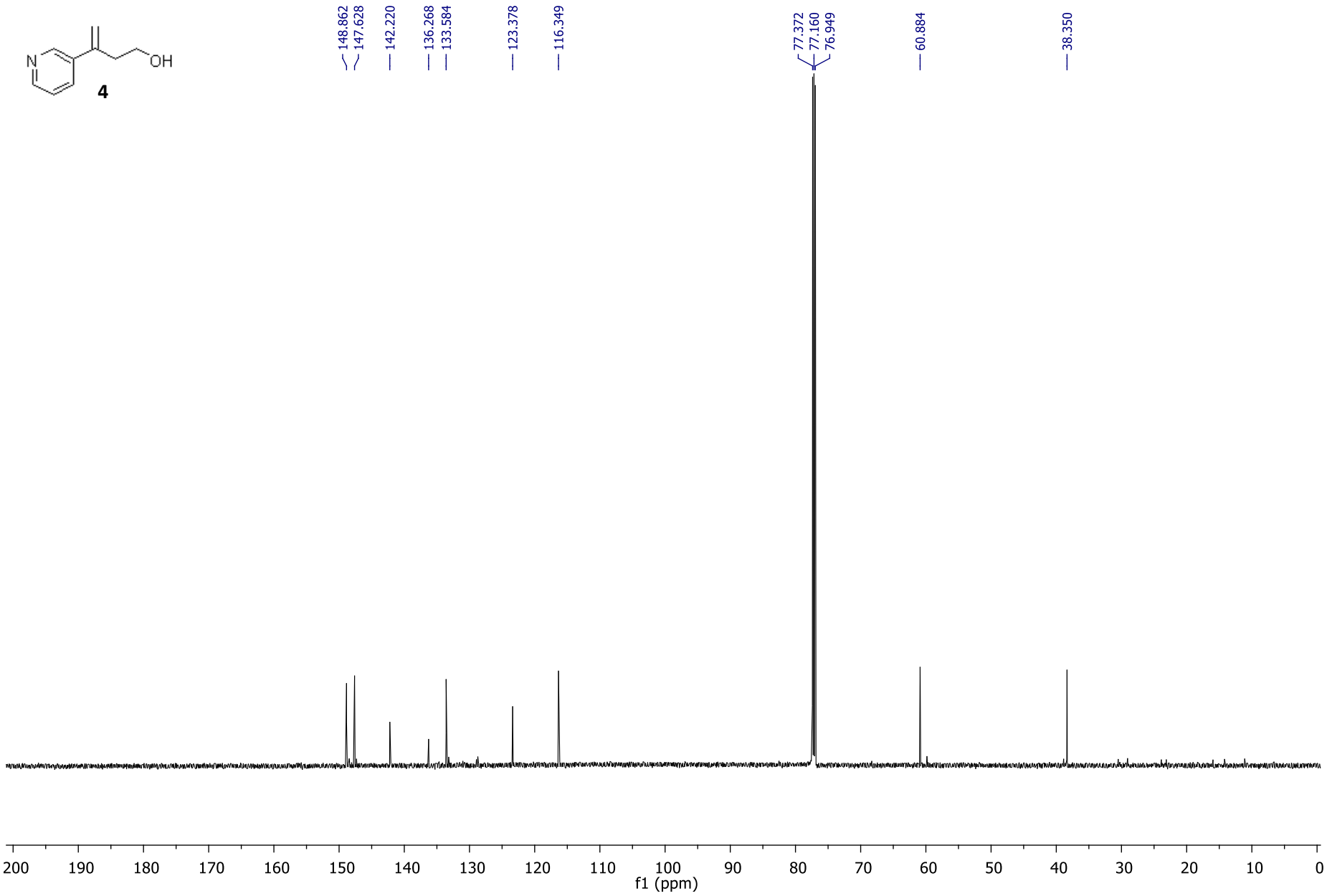
2a

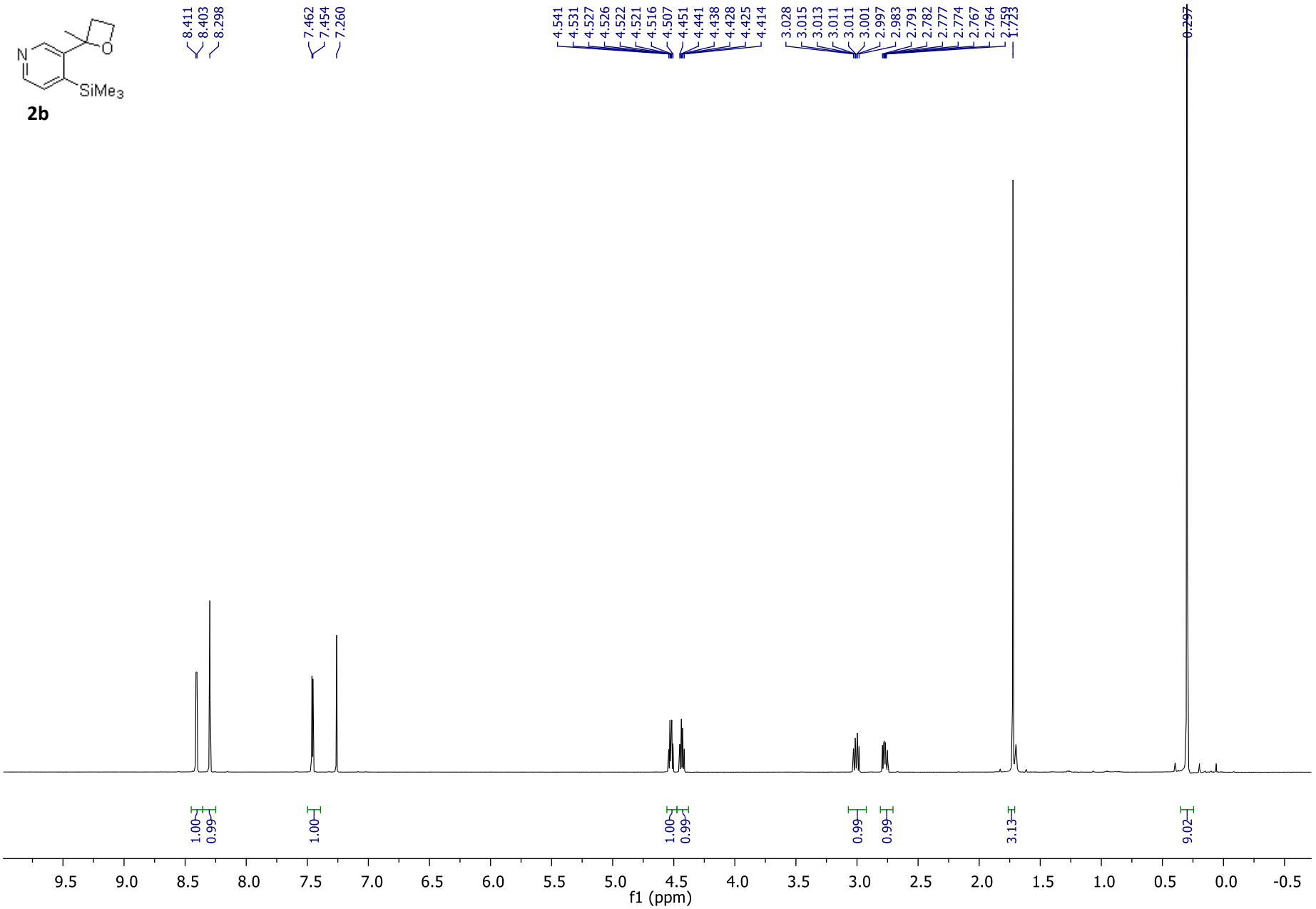
149.20
147.82
140.14
139.52
124.60
85.37
77.48
77.16
76.84
65.45
34.51
28.33
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

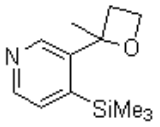

Cl
N
O
3a
8.317
8.312
8.305
8.301
8.016
8.011
7.997
7.992
7.310
7.298
7.291
7.279
7.260
4.685
4.668
4.649
4.632
4.463
4.446
4.443
4.430
4.426
4.409
2.968
2.957
2.946
2.939
2.936
2.927
2.918
2.907
2.898
1.799
1.00
1.00
1.08
1.10
1.14
2.07
3.18
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


Cl
N
O
3a
148.267
146.990
141.759
135.266
122.755
85.444
77.478
77.160
76.842
65.377
34.498
28.236
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.662
8.659
8.510
8.508
8.502
8.500
7.716
7.713
7.709
7.702
7.699
7.699
7.696
7.273
7.272
7.265
7.264
7.252
7.251
5.450
5.267
5.266
3.762
3.751
3.740
2.800
2.798
2.789
2.788
2.778
2.777
N
OH
4
1.00
1.02
1.11
1.42
1.06
1.05
2.13
2.13
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
f1 (ppm)

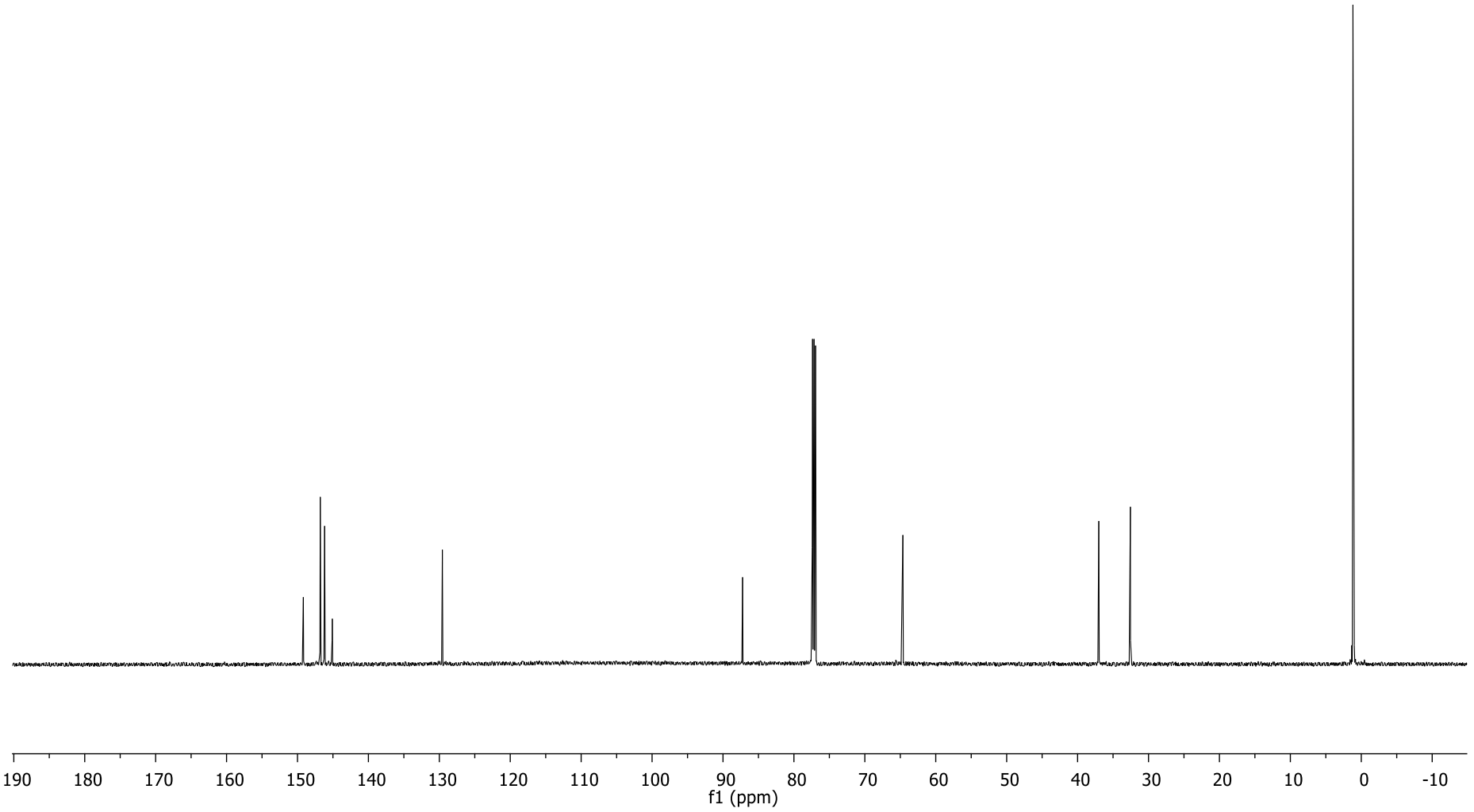

N
OH
4
148.862
147.628
142.220
136.268
133.584
123.378
116.349
77.372
77.160
76.949
60.884
38.350
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

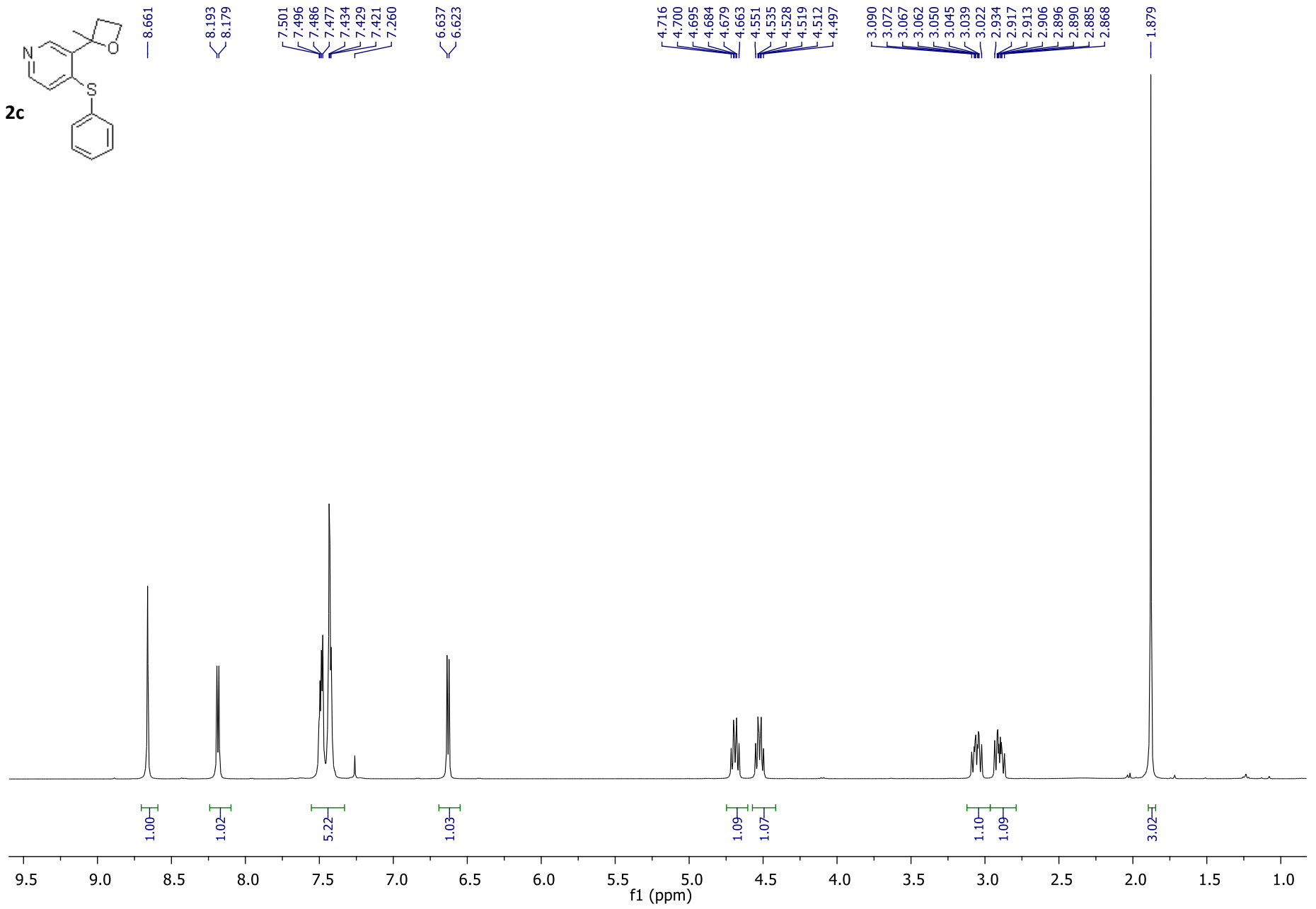

N
O
SiMe3
2b
8.411
8.403
8.298
7.462
7.454
7.260
4.541
4.531
4.527
4.526
4.522
4.521
4.516
4.507
4.451
4.441
4.438
4.428
4.425
4.414
3.028
3.015
3.013
3.011
3.011
3.001
2.997
2.983
2.791
2.782
2.777
2.774
2.767
2.764
2.759
2.753
1.723
0.297
1.00
0.99
1.00
1.00
0.99
0.99
0.99
3.13
9.02
9.5
9.0
8.5
8.0
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
0.0
-0.5
f1 (ppm)

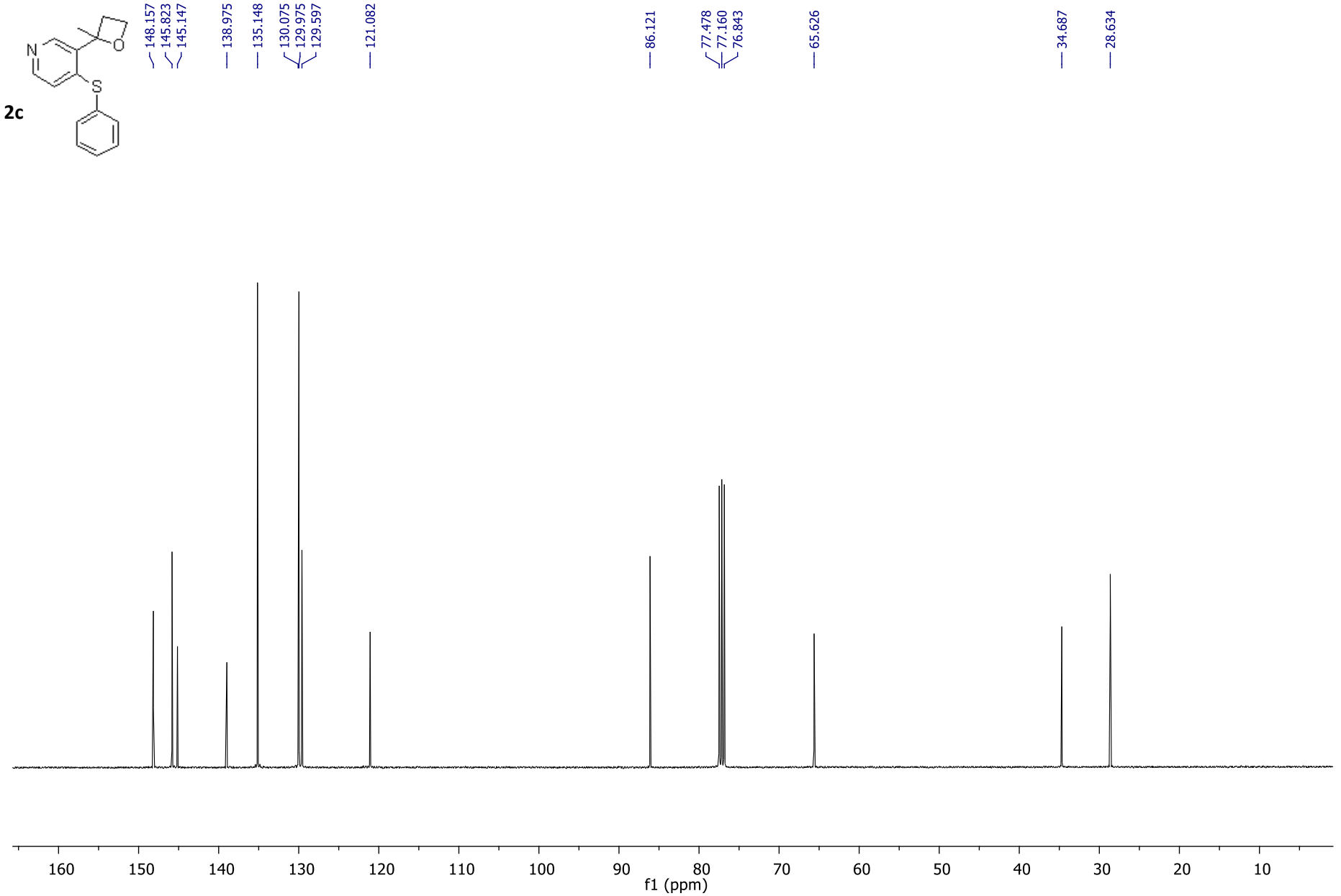


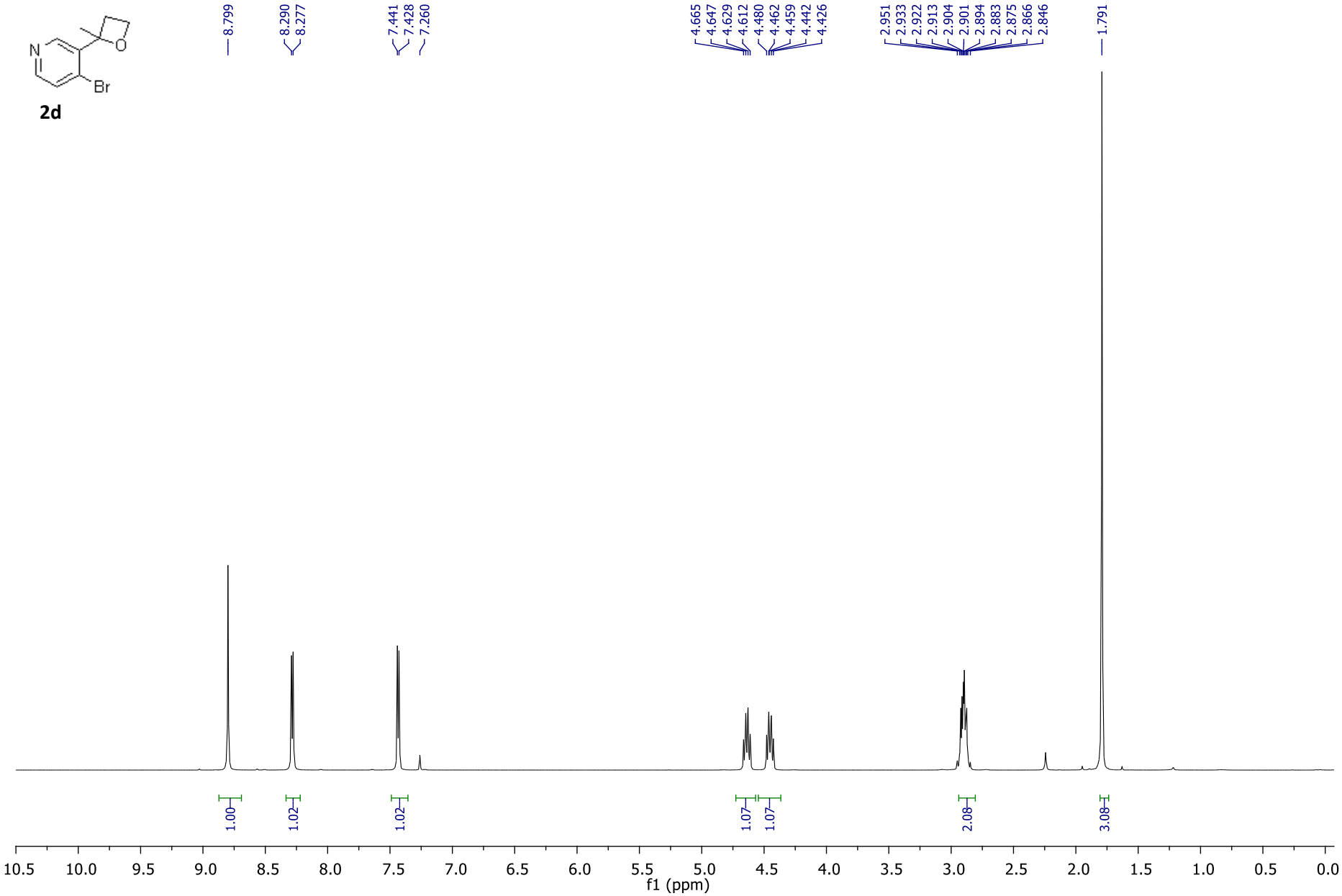
2b

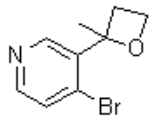
149.172
146.787
146.177
145.088
129.576
87.237
77.372
77.160
76.949
64.637
37.011
32.555
1.171




2c
8.661
8.193
8.179
7.501
7.496
7.486
7.477
7.434
7.429
7.421
7.260
6.637
6.623
4.716
4.700
4.695
4.684
4.679
4.663
4.551
4.535
4.528
4.519
4.512
4.497
3.090
3.072
3.067
3.062
3.050
3.045
3.039
3.022
2.934
2.917
2.913
2.906
2.896
2.890
2.885
2.868
1.879
1.00
1.02
5.22
1.03
1.09
1.07
1.10
1.09
3.02
f1 (ppm)

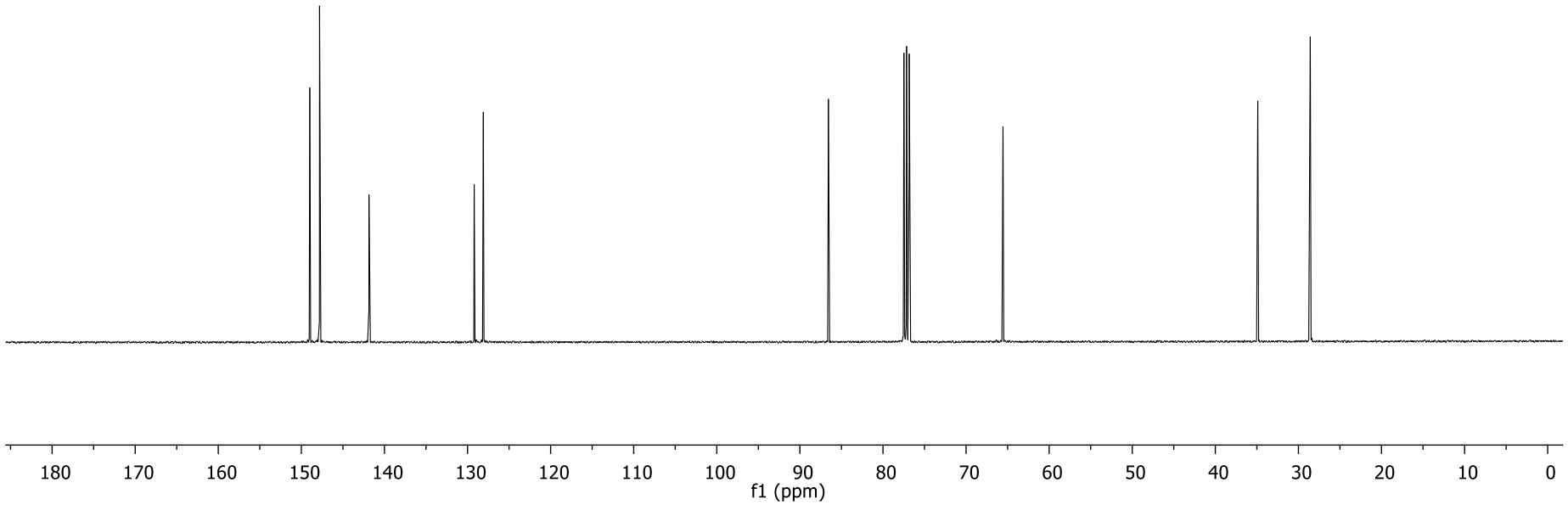

2c
148.157
145.823
145.147
138.975
135.148
130.075
129.975
129.597
121.082
86.121
77.478
77.160
76.843
65.626
34.687
28.634
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

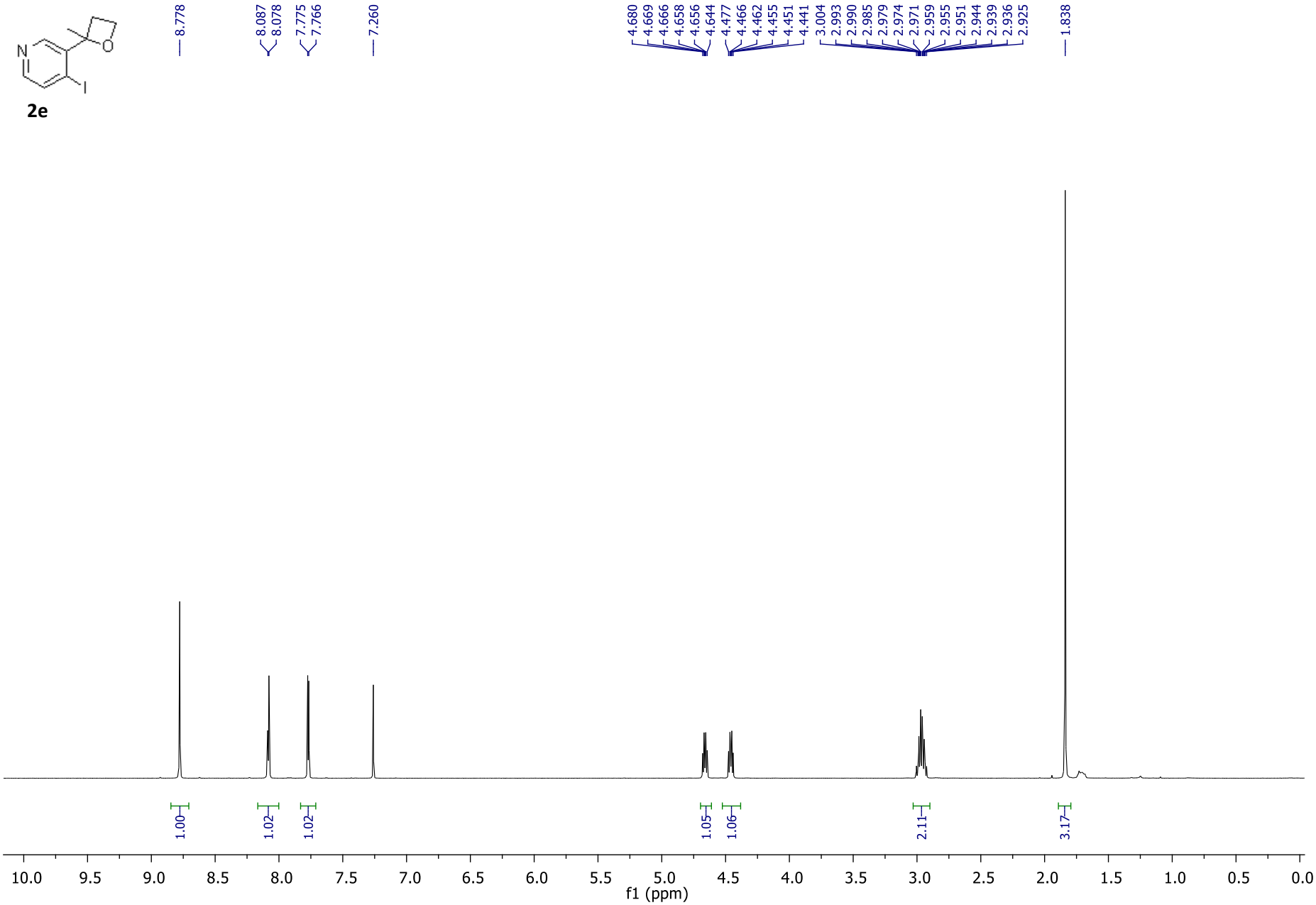

N
O
Br
2d
8.799
8.290
8.277
7.441
7.428
7.260
4.665
4.647
4.629
4.612
4.480
4.462
4.459
4.442
4.426
2.951
2.933
2.922
2.913
2.904
2.901
2.894
2.883
2.875
2.866
2.846
1.791
1.00
1.02
1.02
1.07
1.07
2.08
3.08
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)



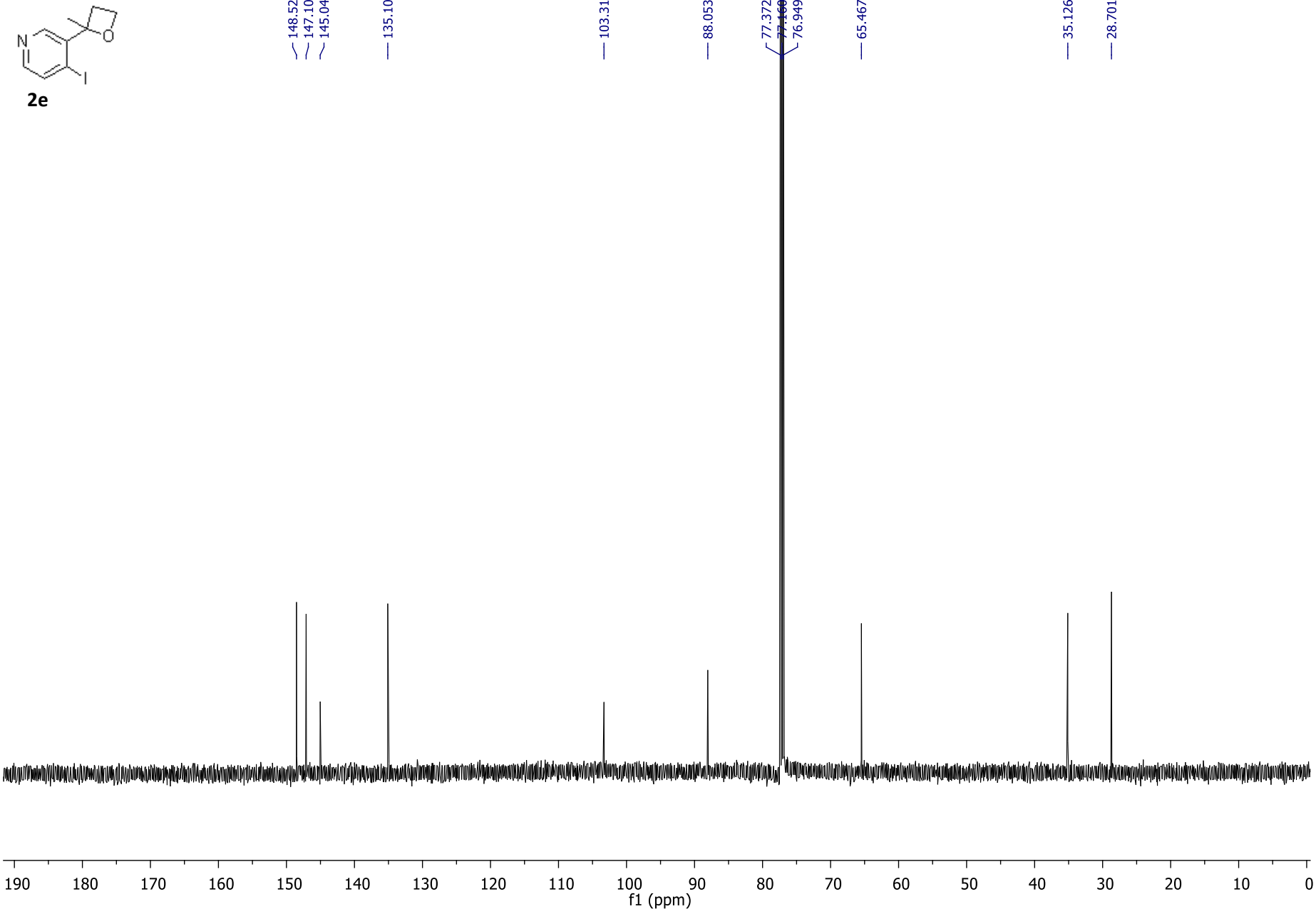
2d

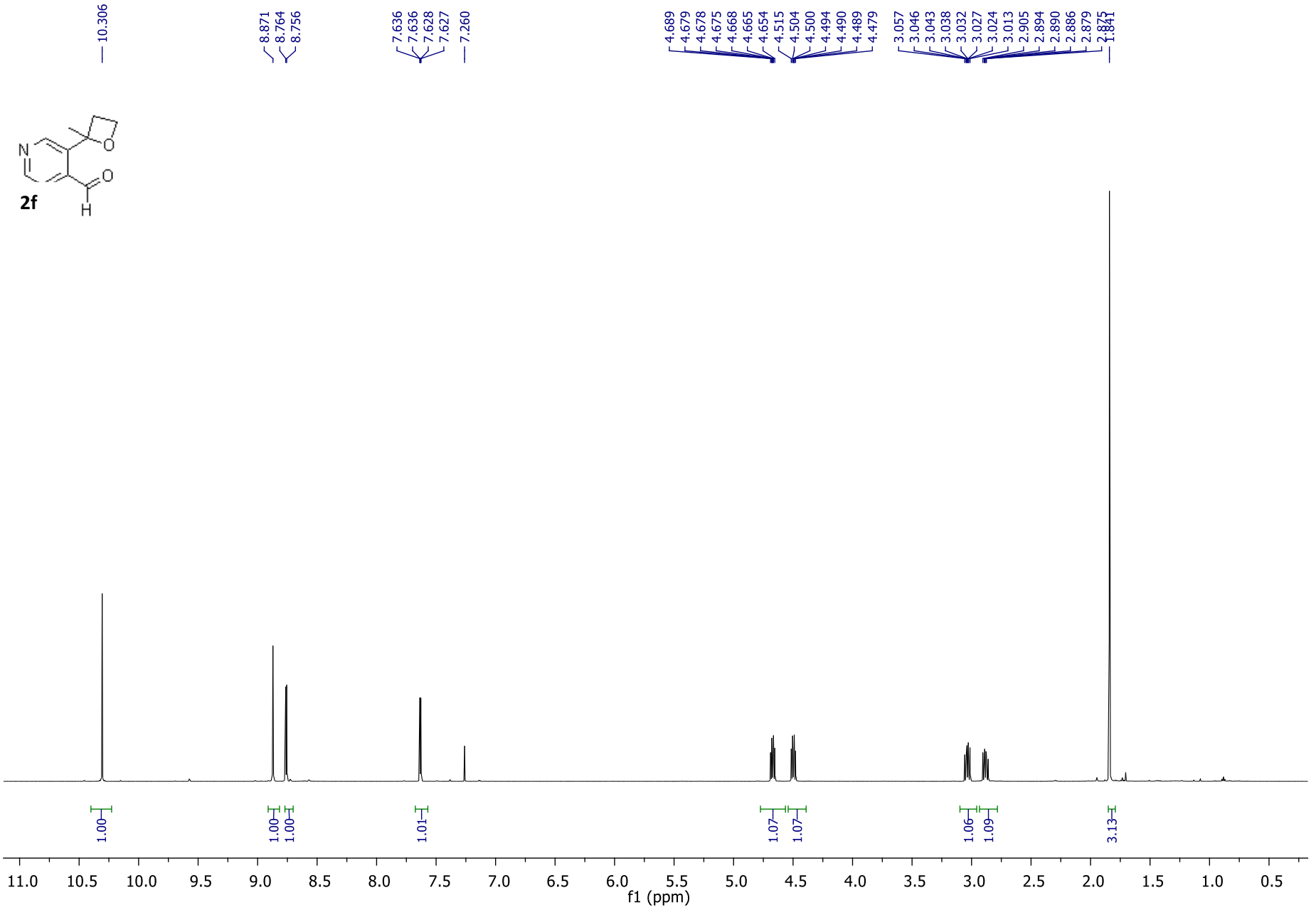
148.986
147.811
141.863
129.200
128.098
86.568
77.477
77.160
76.842
65.553
34.891
28.573

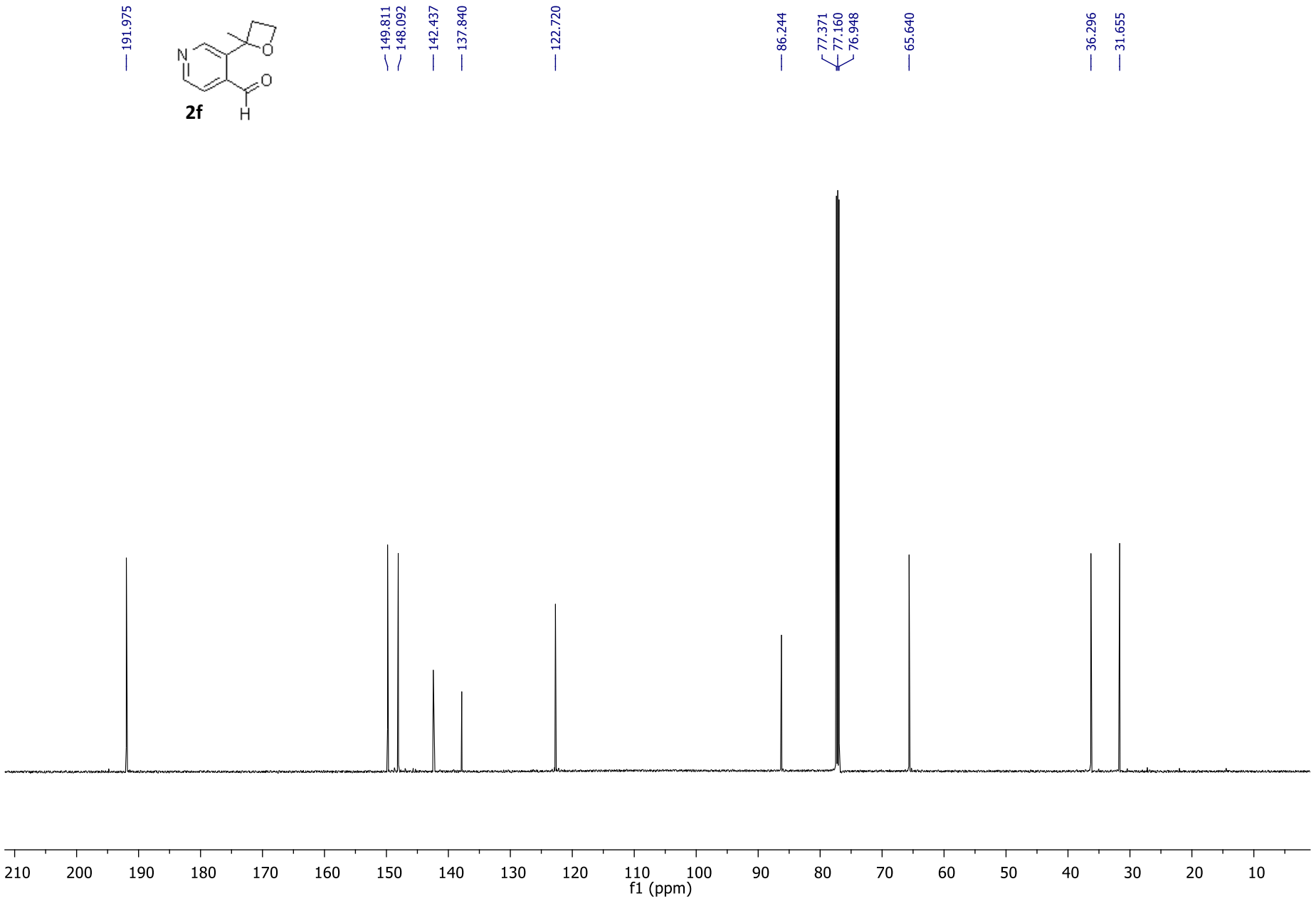


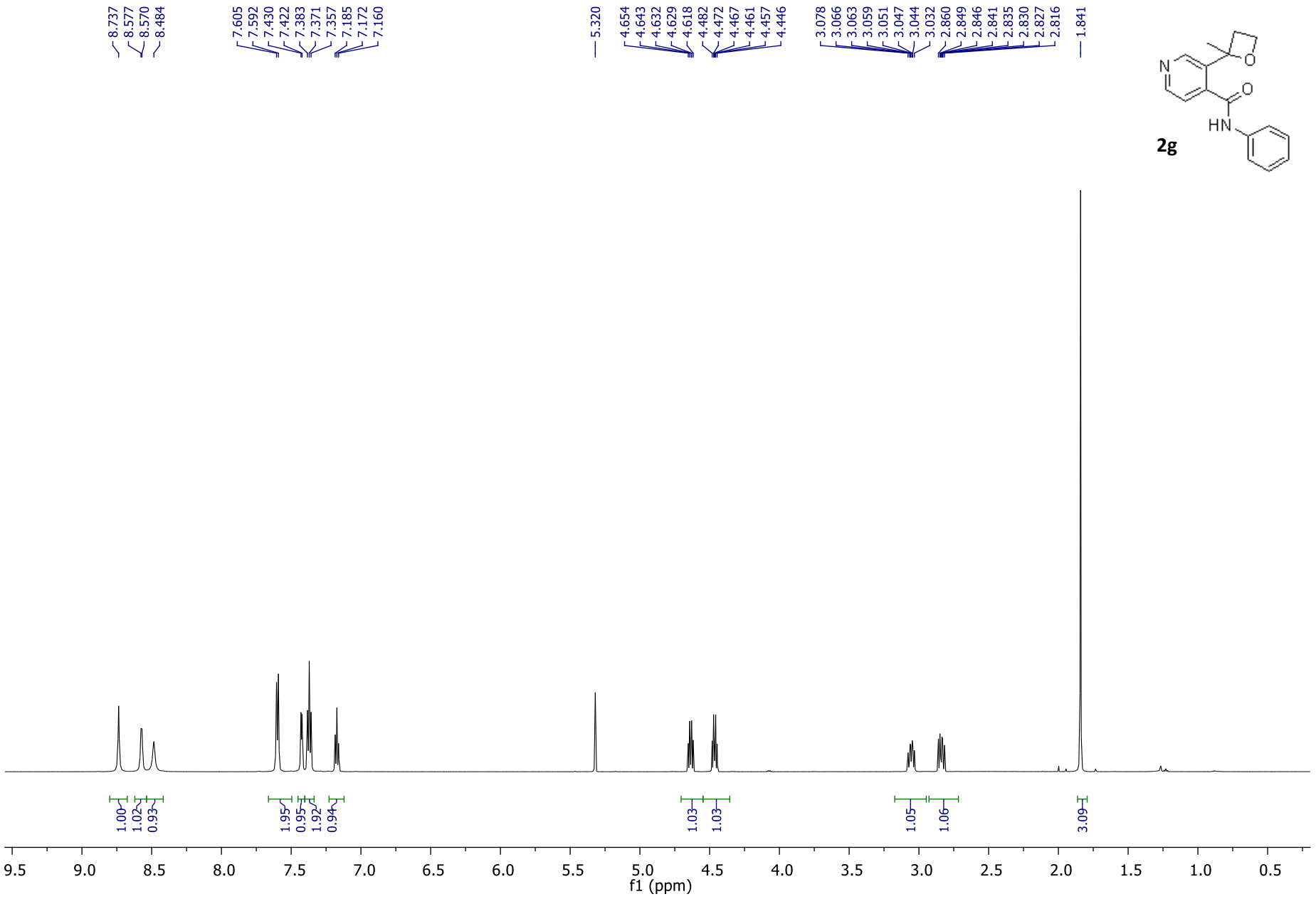

8.778
8.087
8.078
7.775
7.766
7.260
4.680
4.669
4.666
4.658
4.656
4.644
4.477
4.466
4.462
4.455
4.451
4.441
3.004
2.993
2.990
2.985
2.979
2.974
2.971
2.959
2.955
2.951
2.944
2.939
2.936
2.925
1.838
1.00
1.02
1.02
1.05
1.06
2.11
3.17
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

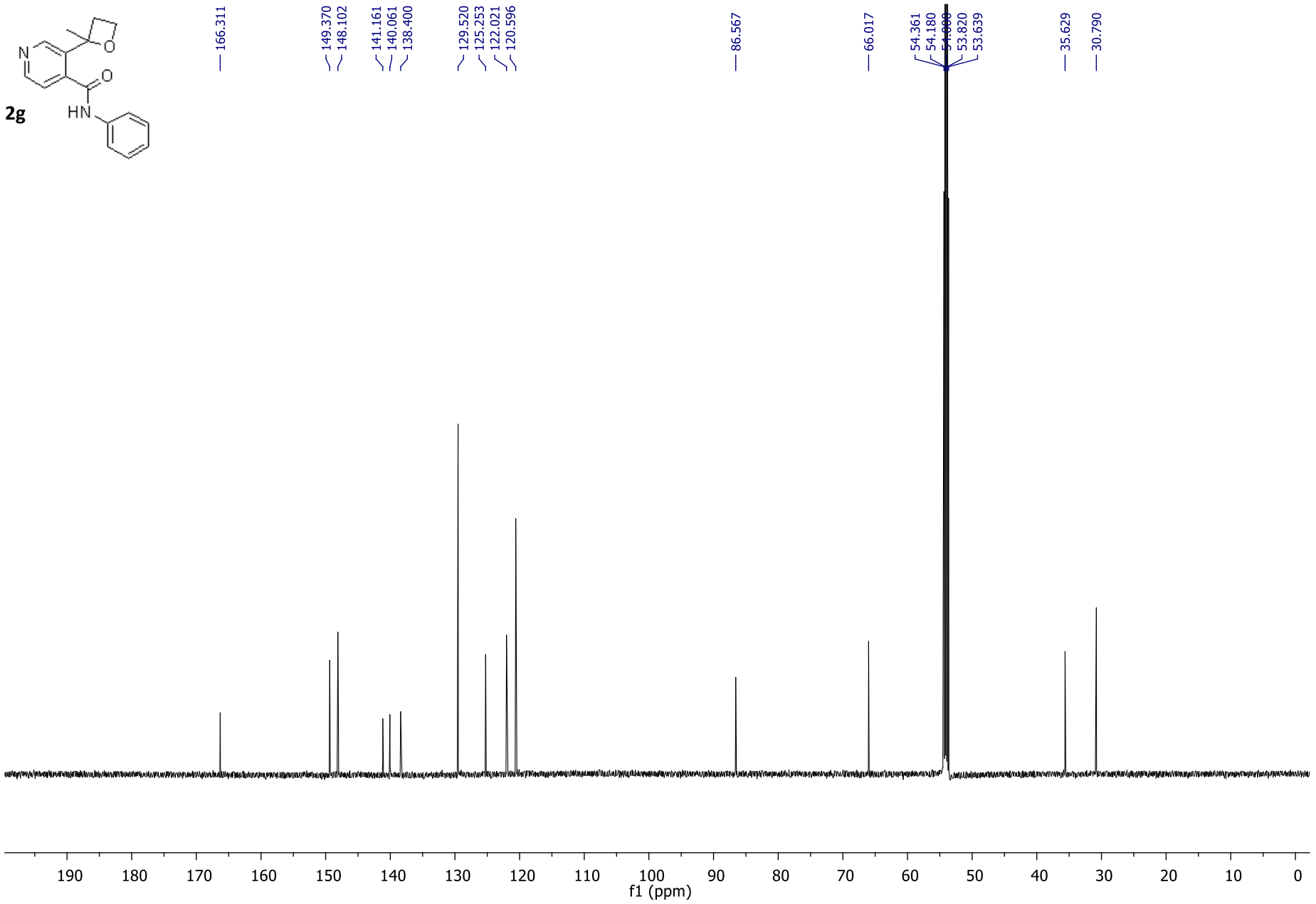
2e

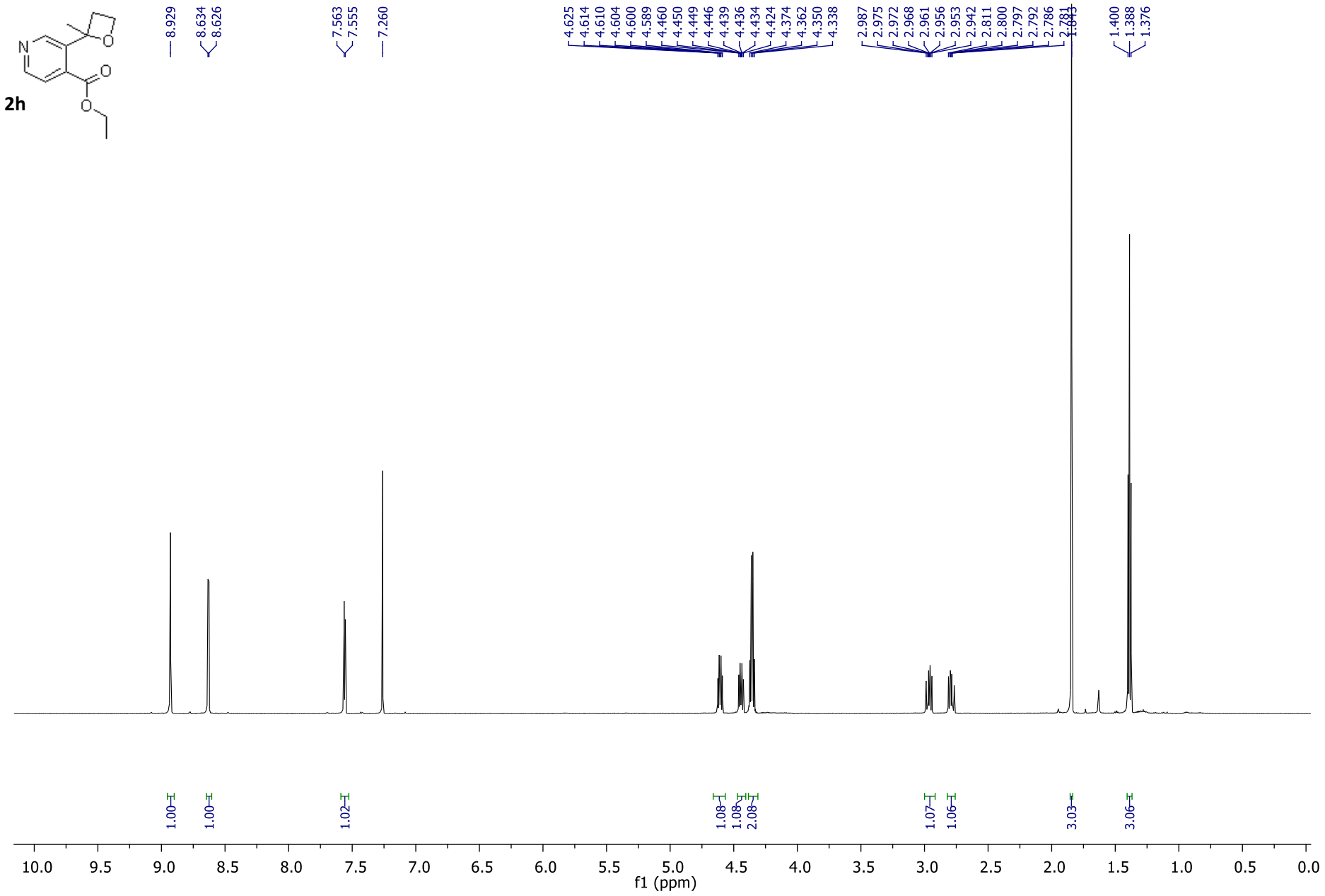

148.522
147.106
145.040
135.105
103.315
88.053
77.372
77.160
76.949
65.467
35.126
28.701
N
O
I
2e
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

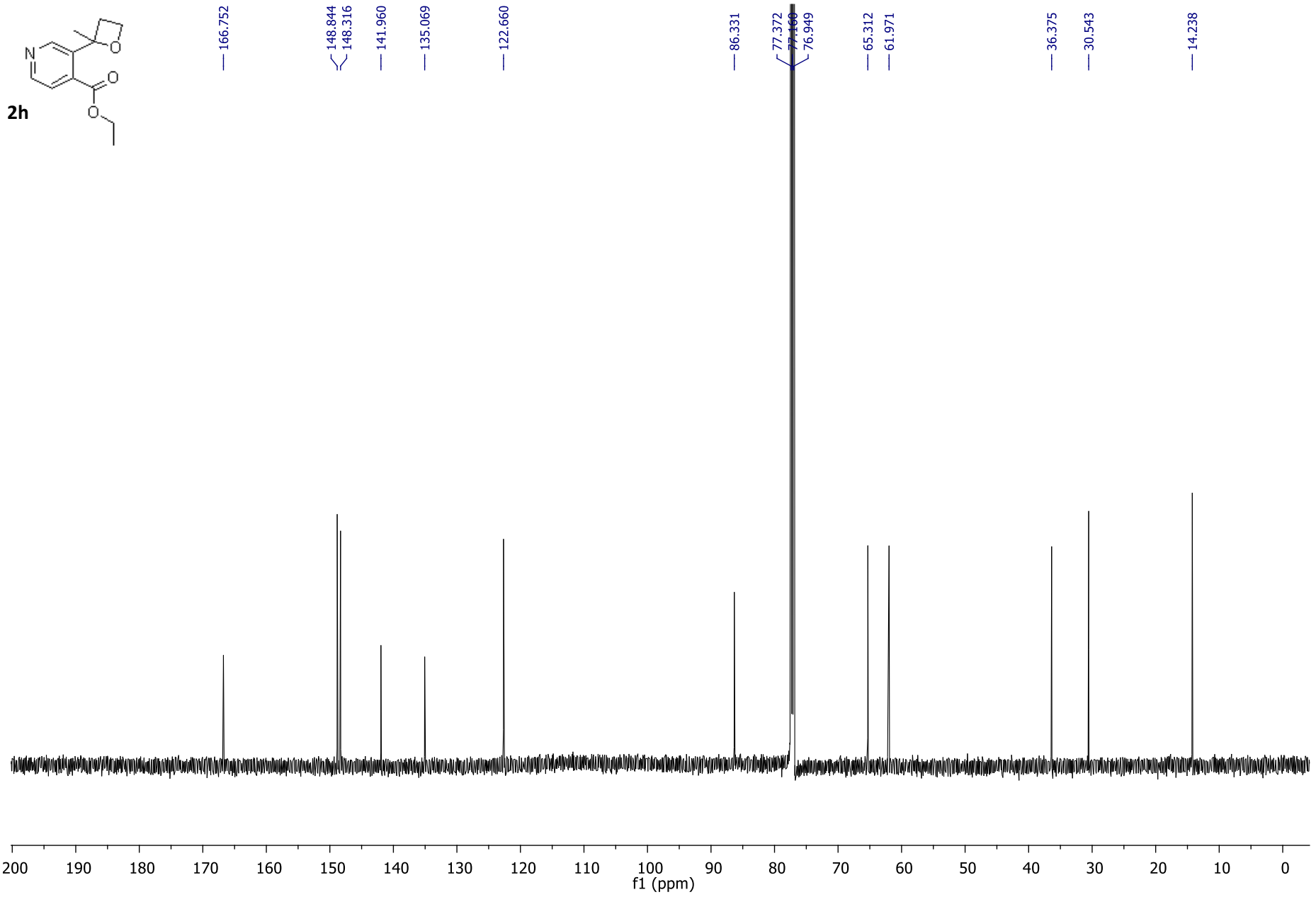

10.306
8.871
8.764
8.756
7.636
7.636
7.628
7.627
7.260
4.689
4.679
4.678
4.675
4.668
4.665
4.654
4.515
4.504
4.500
4.494
4.490
4.489
4.479
3.057
3.046
3.043
3.038
3.032
3.027
3.024
3.013
2.905
2.894
2.890
2.886
2.879
2.875
1.841
N
O
O
H
2f
1.00
1.00
1.00
1.01
1.07
1.07
1.06
1.09
3.13
11.0
10.5
10.0
9.5
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)

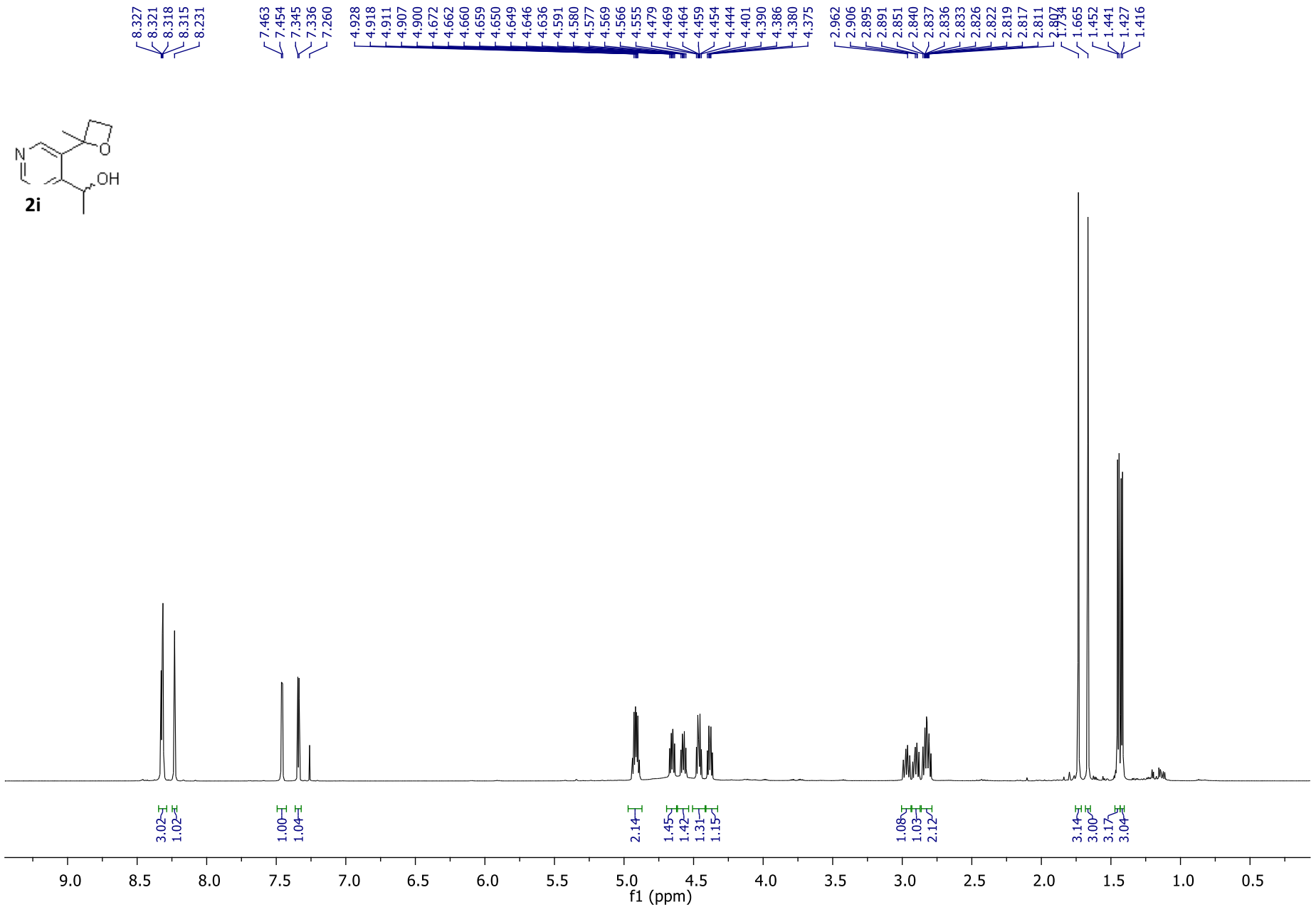

191.975
149.811
148.092
142.437
137.840
122.720
86.244
77.371
77.160
76.948
65.640
36.296
31.655
N
O
O
H
2f
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

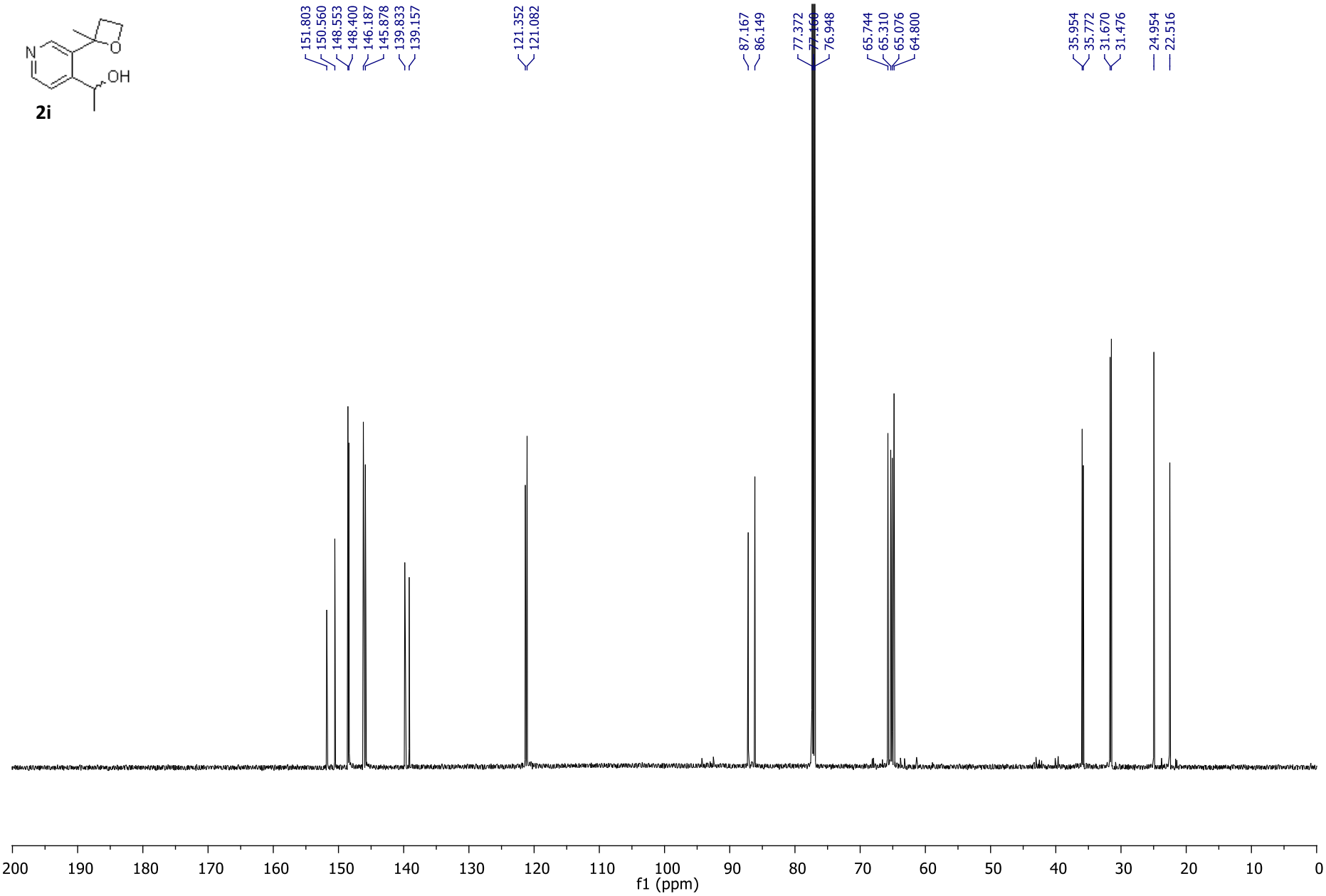

8.737
8.577
8.570
8.484
7.605
7.592
7.430
7.422
7.383
7.371
7.357
7.185
7.172
7.160
5.320
4.654
4.643
4.632
4.629
4.618
4.482
4.472
4.467
4.461
4.457
4.446
3.078
3.066
3.063
3.059
3.051
3.047
3.044
3.032
2.860
2.849
2.846
2.841
2.835
2.830
2.827
2.816
1.841
N
O
O
HN
2g
1.00
1.02
0.93
1.95
0.95
1.92
0.94
1.03
1.03
1.05
1.06
3.09
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)


2g
166.311
149.370
148.102
141.161
140.061
138.400
129.520
125.253
122.021
120.596
86.567
66.017
54.361
54.180
53.820
53.639
35.629
30.790
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

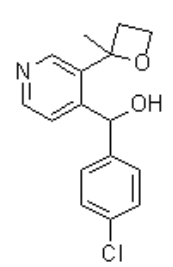

2h
8.929
8.634
8.626
7.563
7.555
7.260
4.625
4.614
4.610
4.604
4.600
4.589
4.460
4.450
4.449
4.446
4.439
4.436
4.434
4.424
4.374
4.362
4.350
4.338
2.987
2.975
2.972
2.968
2.961
2.956
2.953
2.942
2.811
2.800
2.797
2.792
2.786
2.781
1.843
1.400
1.388
1.376
1.00
1.00
1.02
1.08
1.08
2.08
1.07
1.06
3.03
3.06
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
0.0
f1 (ppm)


2h
166.752
148.844
148.316
141.960
135.069
122.660
86.331
77.372
77.160
76.949
65.312
61.971
36.375
30.543
14.238
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.327
8.321
8.318
8.315
8.231
7.463
7.454
7.345
7.336
7.260
4.928
4.918
4.911
4.907
4.900
4.672
4.662
4.660
4.659
4.650
4.649
4.646
4.636
4.591
4.580
4.577
4.569
4.566
4.555
4.479
4.469
4.464
4.459
4.454
4.444
4.401
4.390
4.386
4.380
4.375
2.962
2.906
2.895
2.891
2.851
2.840
2.837
2.836
2.833
2.826
2.822
2.819
2.817
2.811
1.665
1.452
1.441
1.427
1.416
N
O
OH
2i
3.02
1.02
1.00
1.04
2.14
1.45
1.42
1.31
1.15
1.08
1.03
2.12
3.14
3.00
3.17
3.04
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)

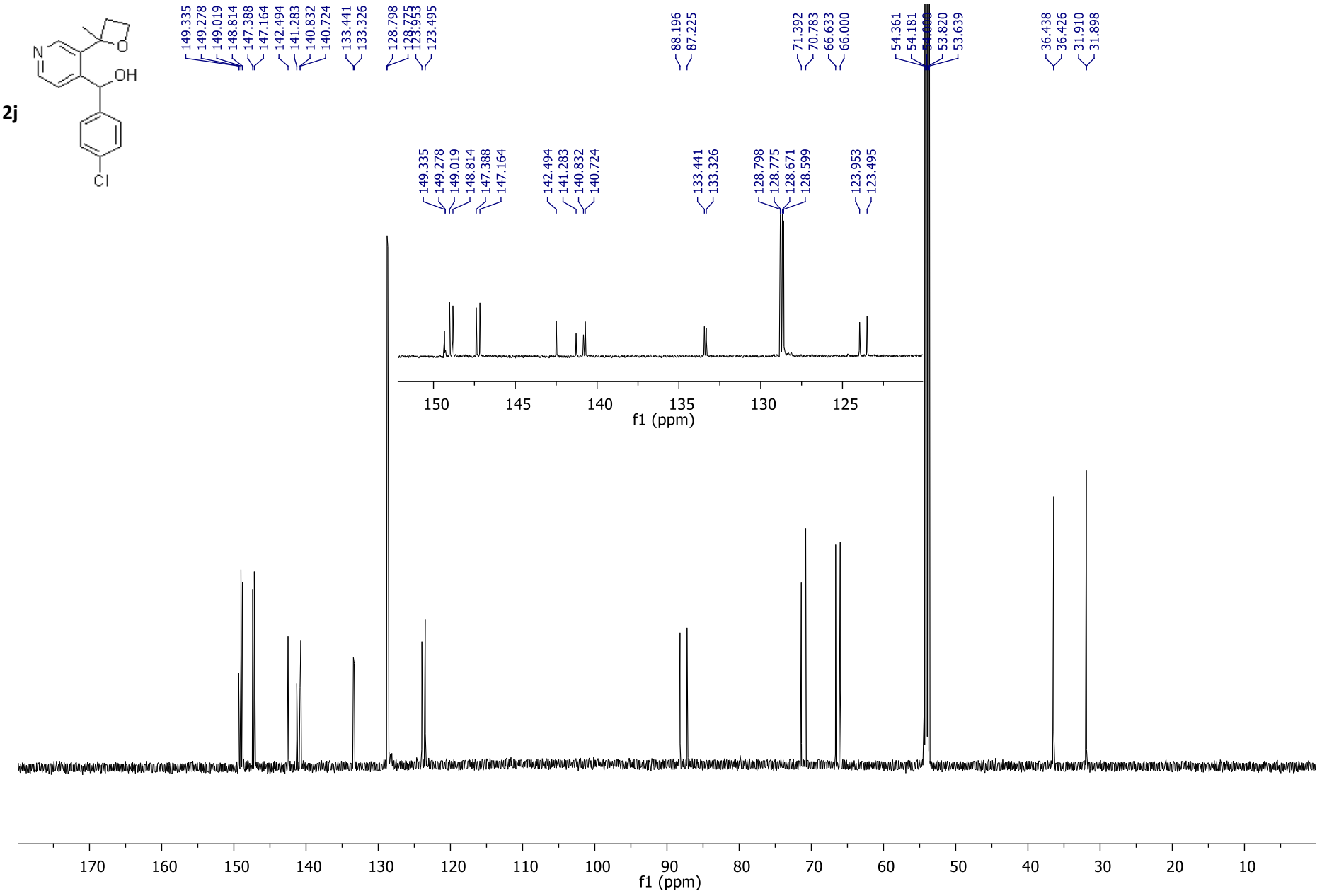

N
O
OH
2i
151.803
150.560
148.553
148.400
146.187
145.878
139.833
139.157
121.352
121.082
87.167
86.149
77.372
77.160
76.948
65.744
65.310
65.076
64.800
35.954
35.772
31.670
31.476
24.954
22.516
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

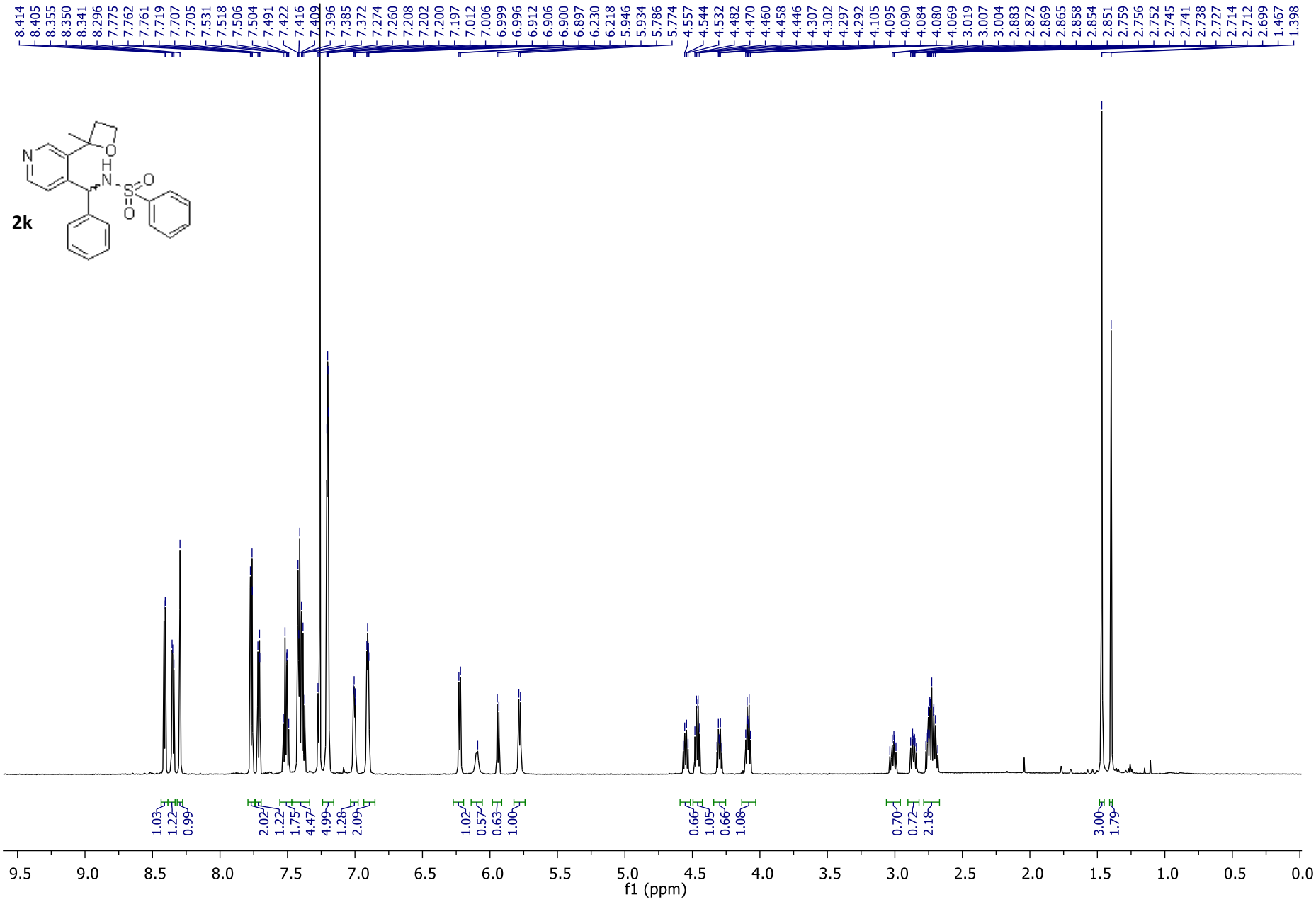
2j

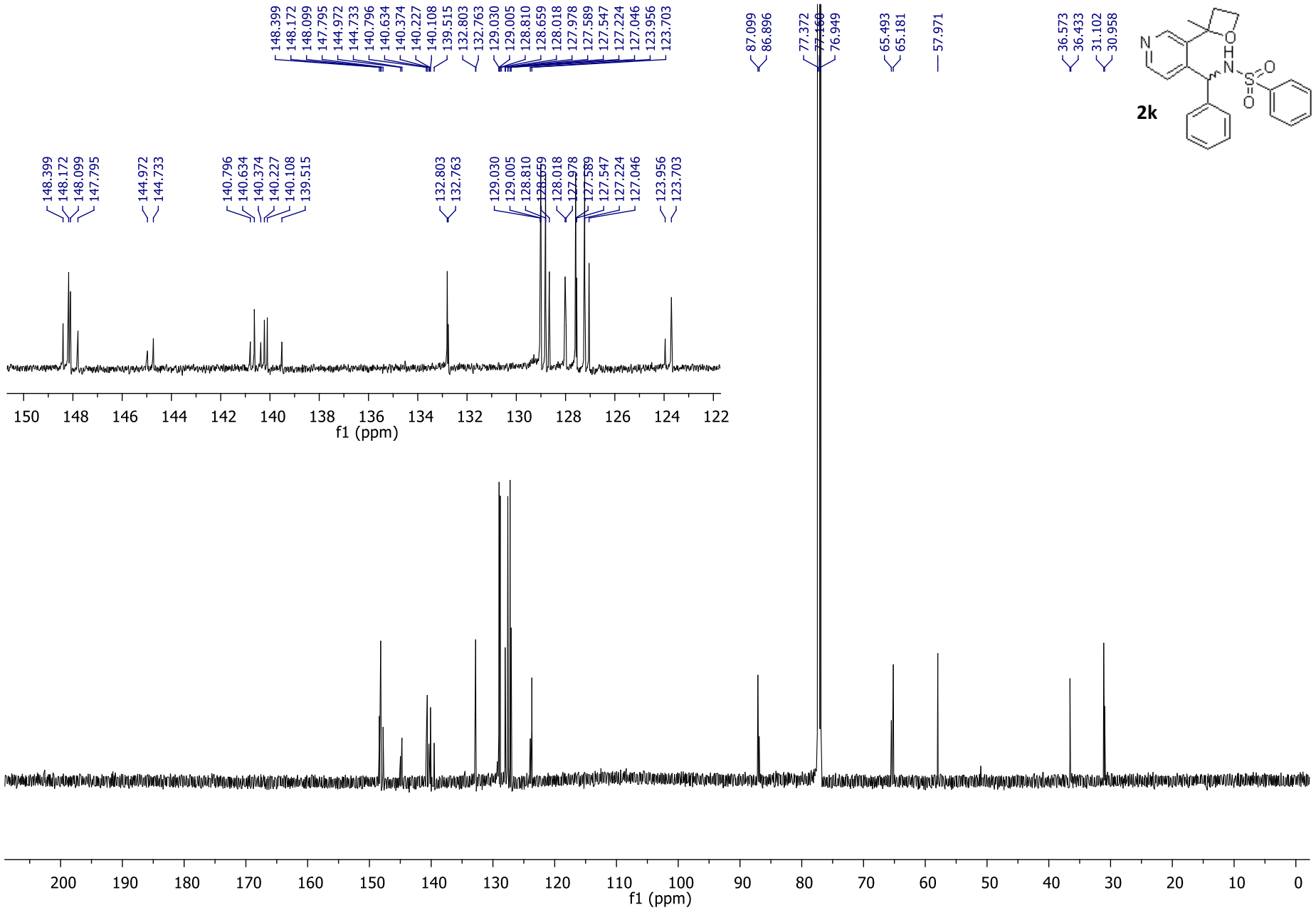

N
O
OH
Cl

8.368
8.360
8.321
8.300
8.292
8.272
7.345
7.331
7.300
7.291
7.286
7.276
7.272
6.863
6.024
6.018
5.320
4.716
4.714
4.705
4.704
4.627
4.626
4.615
4.613
4.490
4.474
4.446
4.431
3.096
3.084
3.080
3.066
3.062
2.921
2.911
2.907
2.903
2.897
2.893
2.889
2.885
2.878
1.846
1.576
1.04
0.95
0.96
0.95
7.50
0.98
0.92
1.94
2.71
1.09
1.03
1.99
2.06
3.00
3.01
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
0.0
f1 (ppm)

2j


N
O
OH
Cl
149.335
149.278
149.019
148.814
147.388
147.164
142.494
141.283
140.832
140.724
133.441
133.326
128.798
128.775
128.671
128.599
123.953
123.495
88.196
87.225
71.392
70.783
66.633
66.000
54.361
54.181
53.820
53.639
36.438
36.426
31.910
31.898
f1 (ppm)
f1 (ppm)


8.414
8.405
8.355
8.350
8.341
8.296
7.775
7.762
7.761
7.719
7.707
7.705
7.531
7.518
7.506
7.504
7.491
7.422
7.416
7.409
7.396
7.385
7.372
7.274
7.260
7.208
7.202
7.200
7.197
7.012
7.006
6.999
6.996
6.912
6.906
6.900
6.897
6.230
6.218
5.946
5.934
5.786
5.774
4.557
4.544
4.532
4.482
4.470
4.460
4.458
4.446
4.307
4.302
4.297
4.292
4.105
4.095
4.090
4.084
4.080
4.069
3.019
3.007
3.004
2.883
2.872
2.869
2.865
2.858
2.854
2.851
2.759
2.756
2.752
2.745
2.741
2.738
2.727
2.714
2.712
2.699
1.467
1.398
2k
1.03
1.22
0.99
2.02
1.22
1.75
4.47
4.99
1.28
2.09
1.02
0.57
0.63
1.00
0.66
1.05
0.66
1.08
0.70
0.72
2.18
3.00
1.79
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
0.0
f1 (ppm)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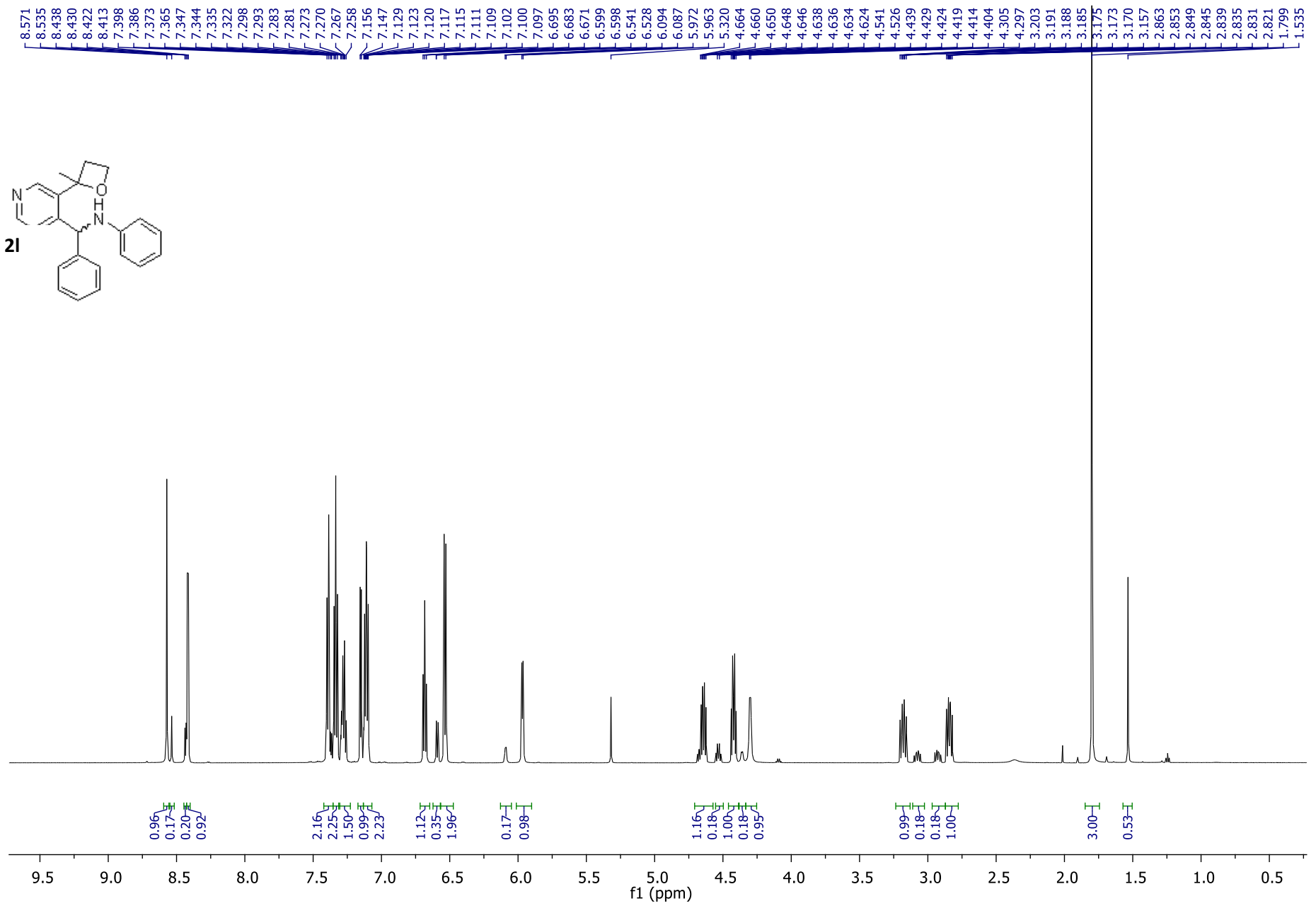
148.172
148.099
147.795
144.972
144.733
140.796
140.634
140.374
140.227
140.108
139.515
132.803
132.763
129.030
129.005
128.810
128.659
128.018
127.978
127.589
127.547
127.224
127.046
123.956
123.703
87.099
86.896
77.372
77.160
76.949
65.493
65.181
57.971
36.573
36.433
31.102
30.958
2k
f1 (ppm)
f1 (ppm)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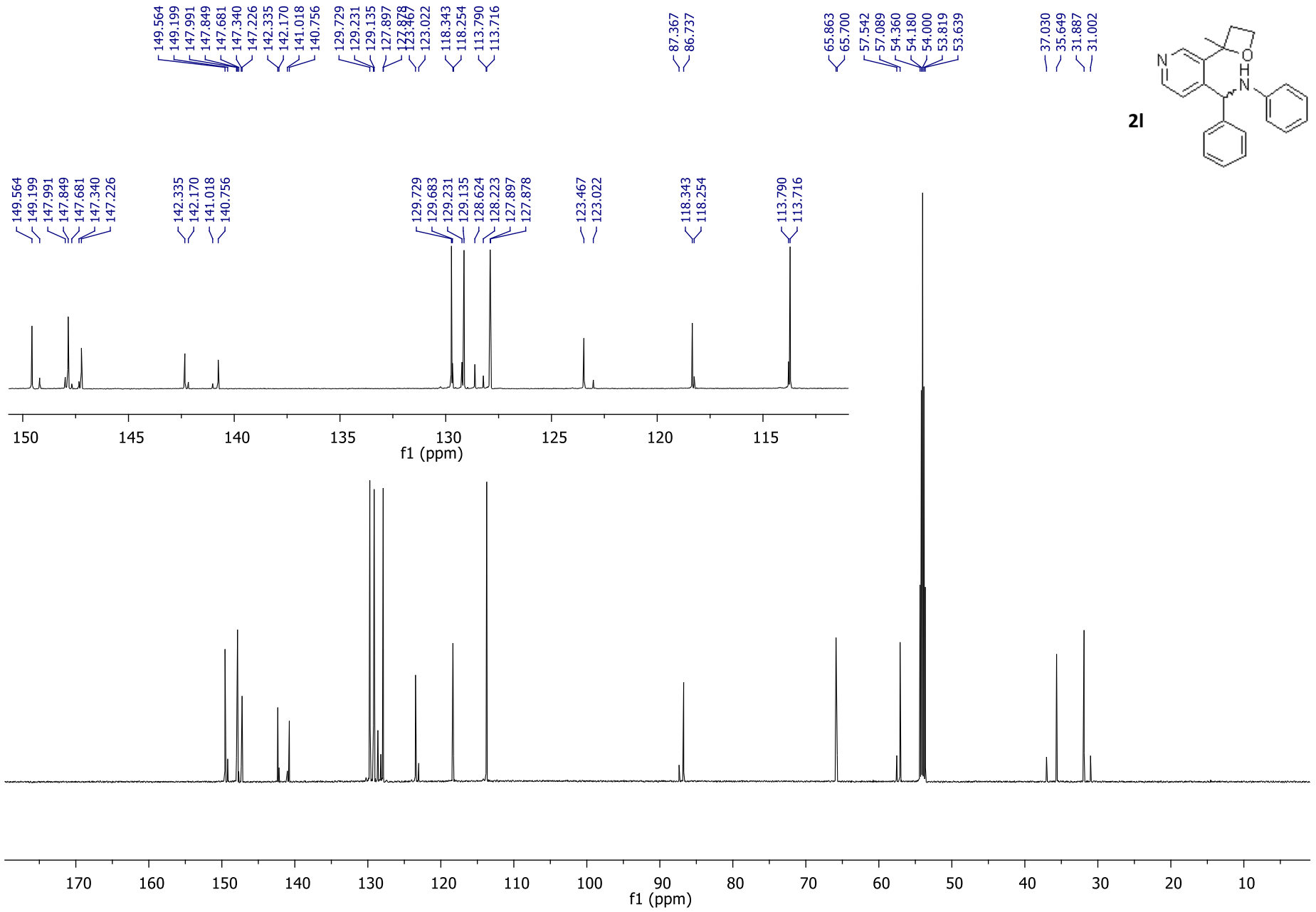
8.535
8.438
8.430
8.422
8.413
7.398
7.386
7.373
7.365
7.347
7.344
7.335
7.322
7.298
7.293
7.283
7.281
7.273
7.270
7.267
7.258
7.156
7.147
7.129
7.123
7.120
7.117
7.115
7.111
7.109
7.102
7.100
7.097
6.695
6.683
6.671
6.599
6.598
6.541
6.528
6.094
6.087
5.972
5.963
5.320
4.664
4.660
4.650
4.648
4.646
4.638
4.636
4.634
4.624
4.541
4.526
4.439
4.429
4.424
4.419
4.414
4.404
4.305
4.297
3.203
3.191
3.188
3.185
3.175
3.173
3.170
3.157
2.863
2.853
2.849
2.845
2.839
2.835
2.831
2.821
1.799
1.535
N
O
H
N
2l
0.96
0.17
0.20
0.92
2.16
2.25
1.50
0.99
2.23
1.12
0.35
1.96
0.17
0.98
1.16
0.18
1.00
0.18
0.95
0.99
0.18
0.18
1.00
3.00
0.53
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)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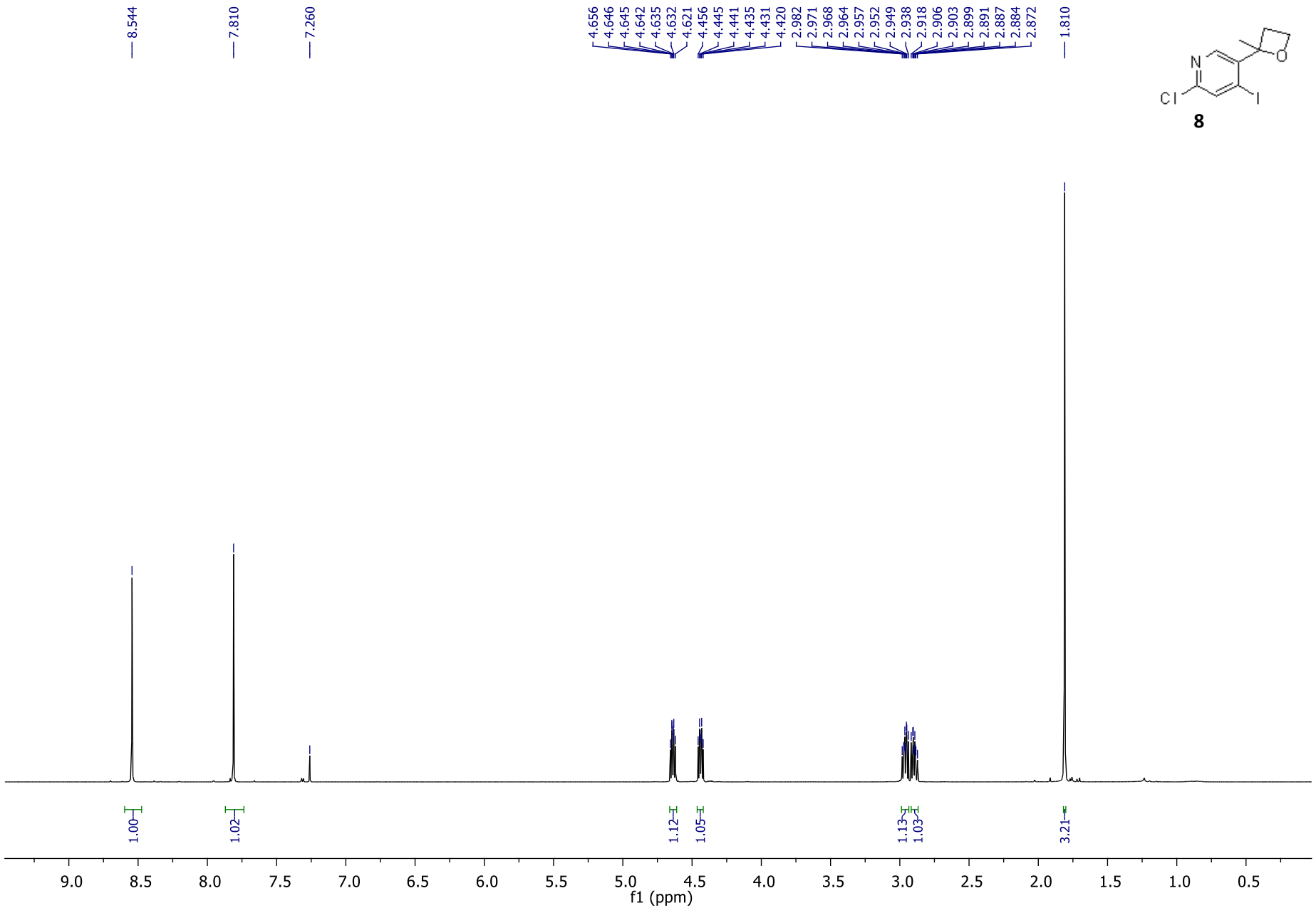
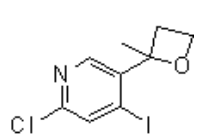
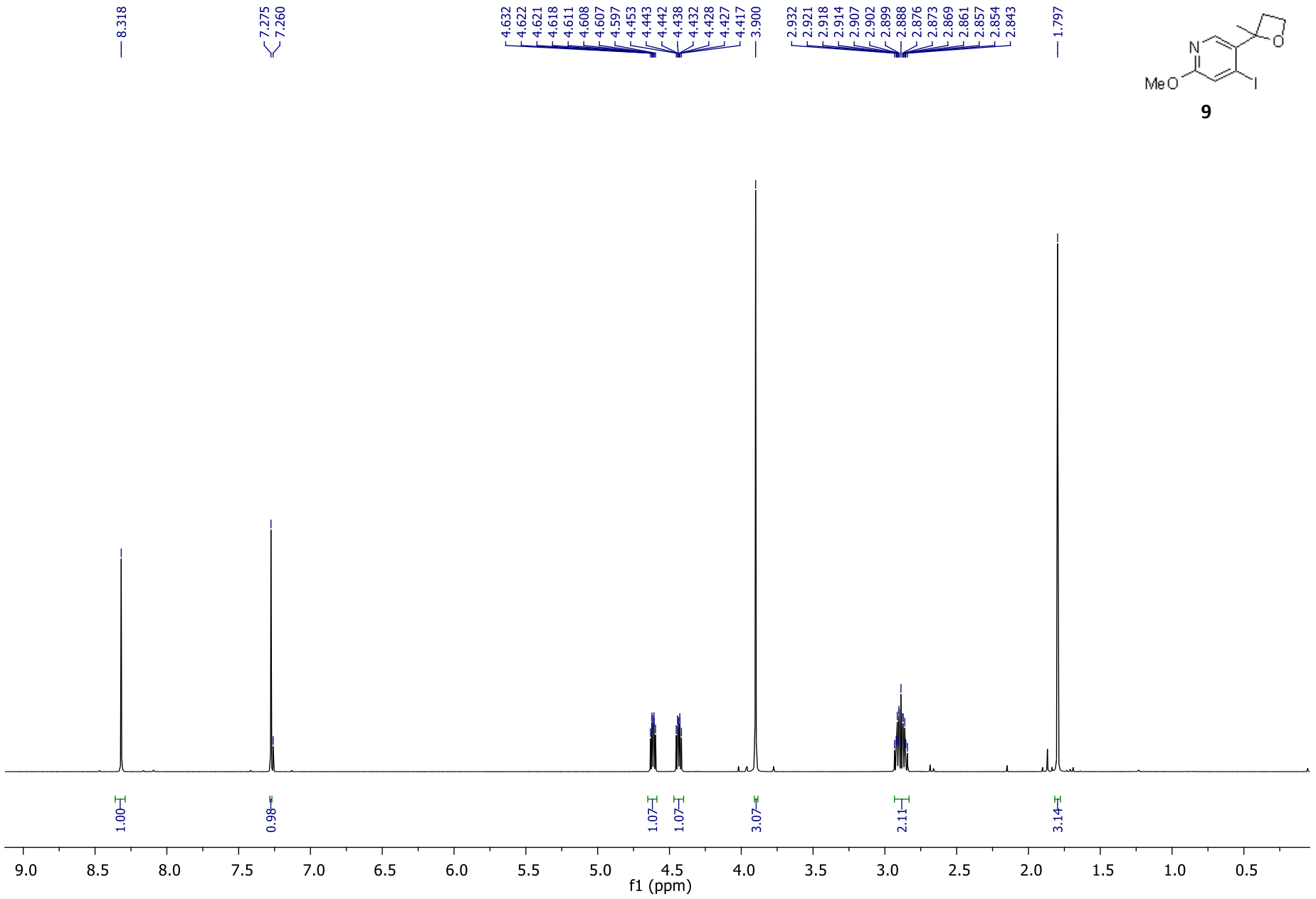
149.199
147.991
147.849
147.681
147.340
147.226
142.335
142.170
141.018
140.756
129.729
129.231
129.135
127.897
123.467
123.022
118.343
118.254
113.790
113.716
87.367
86.737
65.863
65.700
57.542
57.089
54.360
54.180
54.000
53.819
53.639
37.030
35.649
31.887
31.002
2l
f1 (ppm)
f1 (ppm)


8.544
7.810
7.260
4.656
4.646
4.645
4.642
4.635
4.632
4.621
4.456
4.445
4.441
4.435
4.431
4.420
2.982
2.971
2.968
2.964
2.957
2.952
2.949
2.938
2.918
2.906
2.903
2.899
2.891
2.887
2.884
2.872
1.810
N
O
Cl
I
8
1.00
1.02
1.12
1.05
1.13
1.03
3.21
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)


N
O
Cl
I

8

149.877
146.633
143.902
134.555
104.665
87.635
77.372
77.160
76.948
65.429
34.960
28.609
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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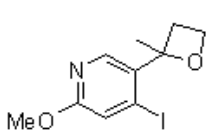
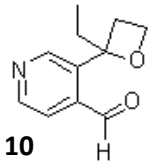
7.275
7.260
4.632
4.622
4.621
4.618
4.611
4.608
4.607
4.597
4.453
4.443
4.442
4.438
4.432
4.428
4.427
4.417
3.900
2.932
2.921
2.918
2.914
2.907
2.902
2.899
2.888
2.876
2.873
2.869
2.861
2.857
2.854
2.843
1.797
N
O
MeO
I
1.00
0.98
1.07
1.07
3.07
2.11
3.14
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

9


N
O
MeO
I

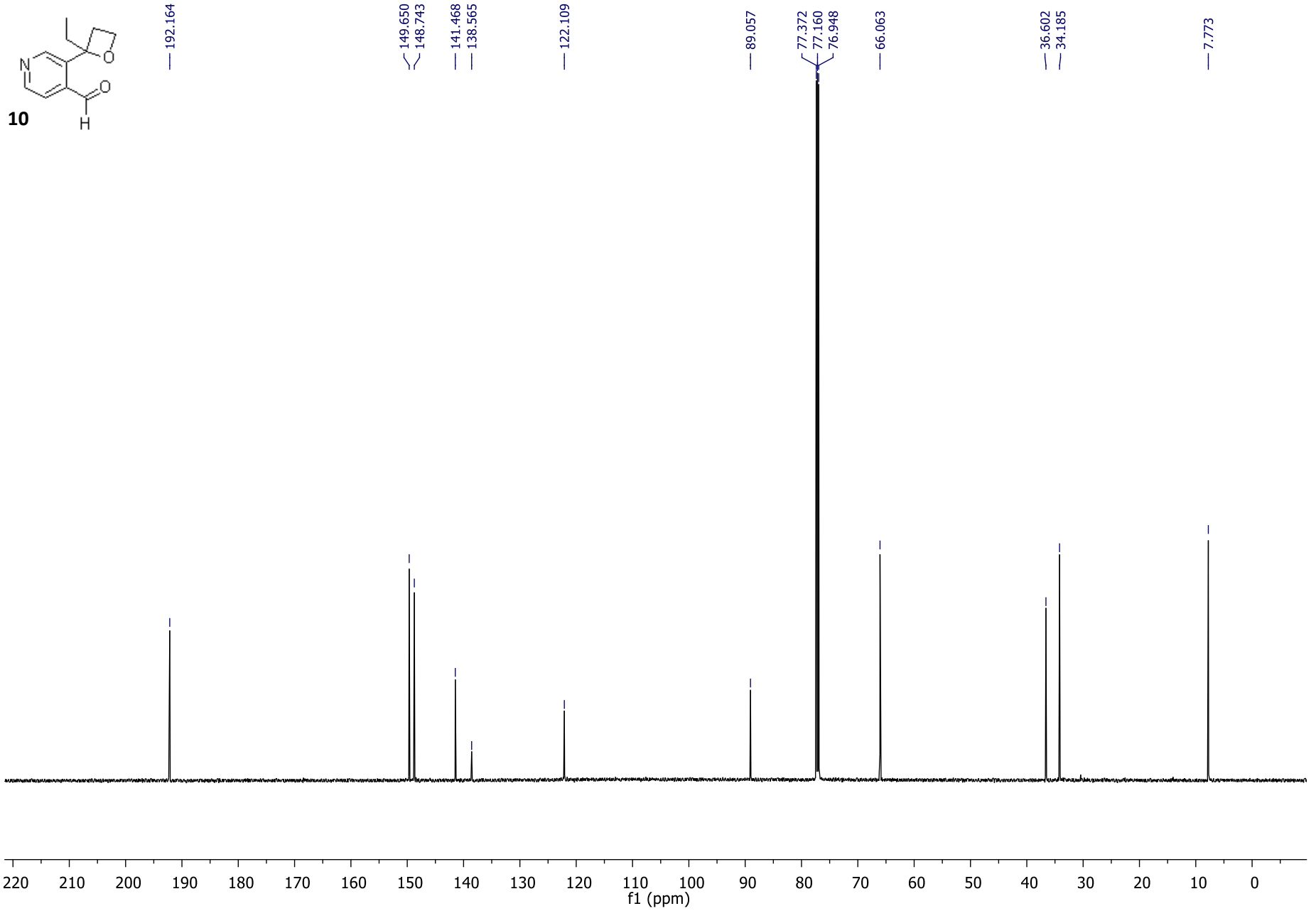
9

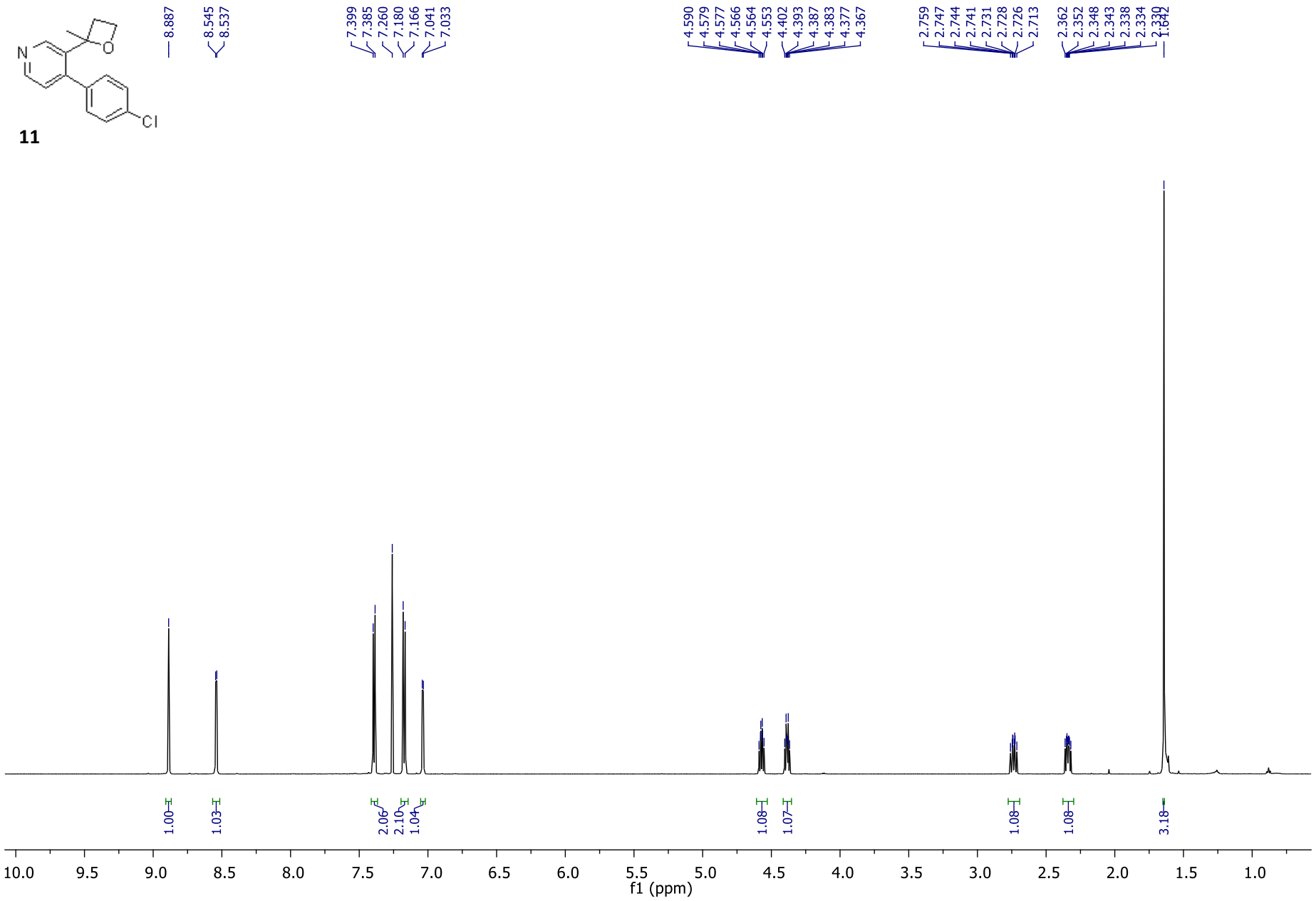
163.109
143.497
137.218
121.955
104.469
87.889
77.372
77.160
76.948
65.377
53.840
35.167
29.349
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


N
O
O
H

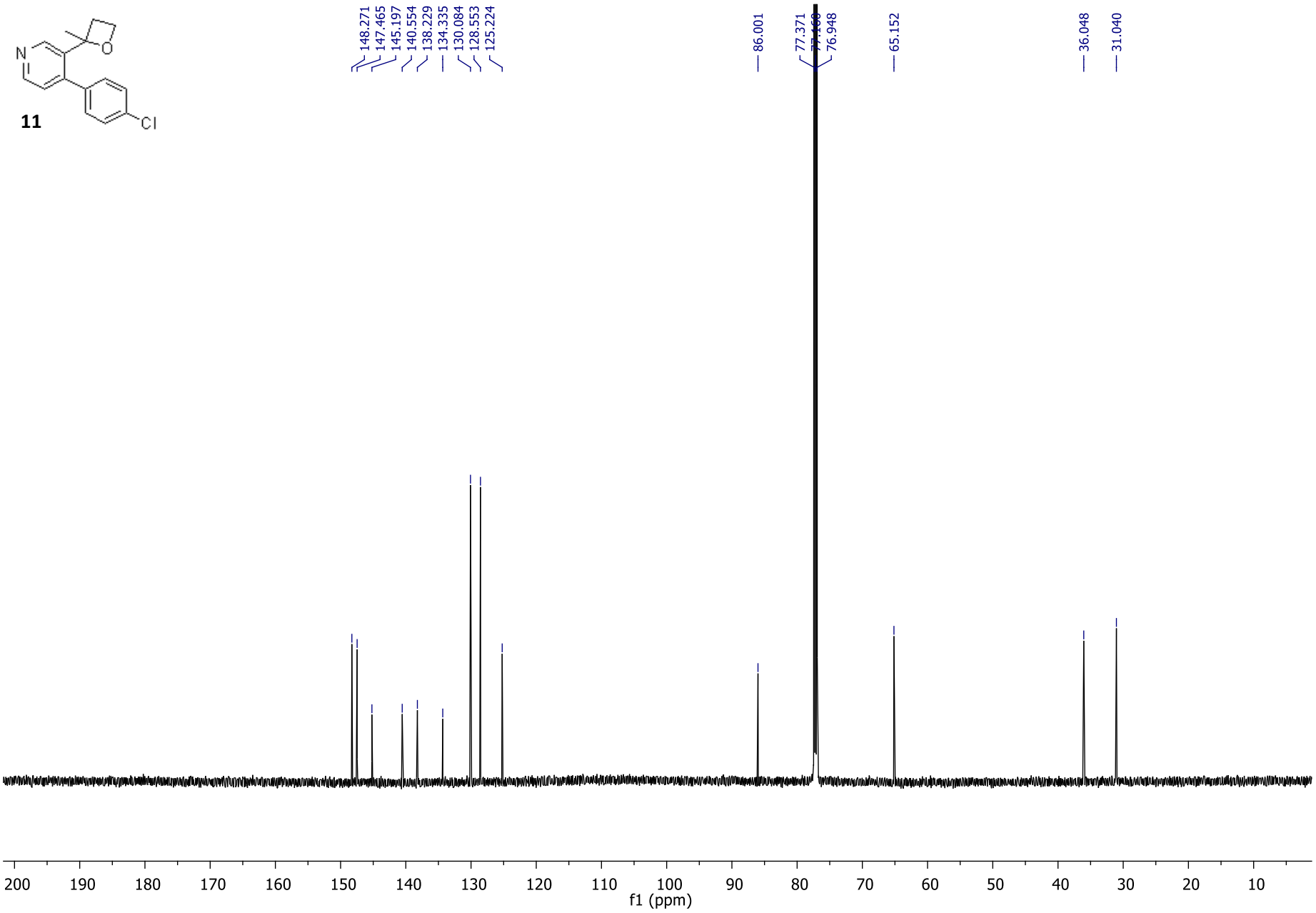
10

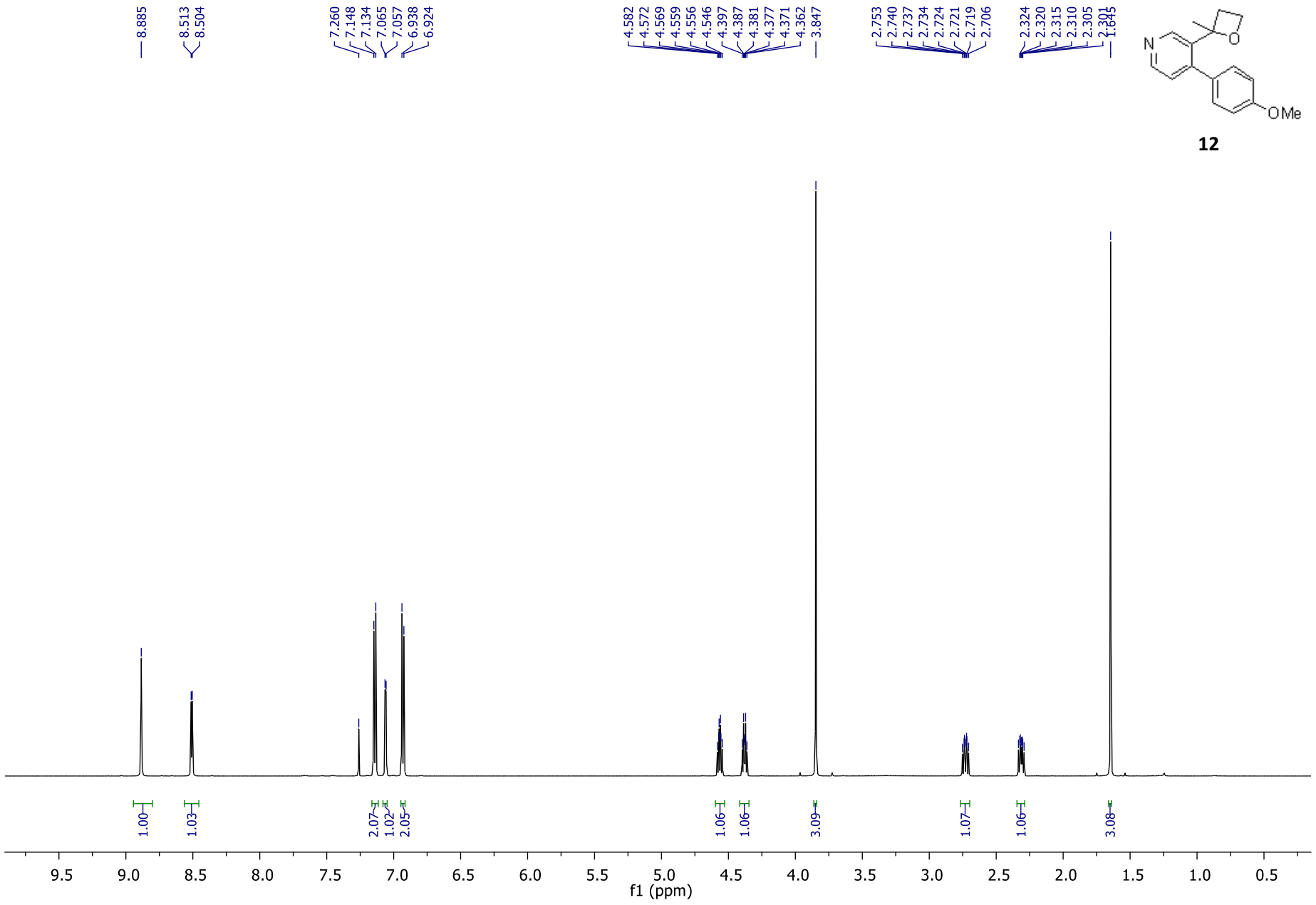
10.331
8.764
8.742
8.734
7.646
7.645
7.637
7.636
7.260
4.625
4.615
4.611
4.604
4.601
4.590
4.541
4.530
4.526
4.520
4.515
4.515
4.504
3.083
3.080
3.076
3.065
3.061
3.050
2.849
2.845
2.833
2.190
2.177
2.166
2.154
2.141
2.032
2.020
2.008
1.996
0.952
0.940
0.928
1.00
0.95
1.00
1.01
1.10
1.10
1.06
1.07
1.10
1.09
3.17
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
0.0
f1 (ppm)


10
192.164
149.650
148.743
141.468
138.565
122.109
89.057
77.372
77.160
76.948
66.063
36.602
34.185
7.773
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

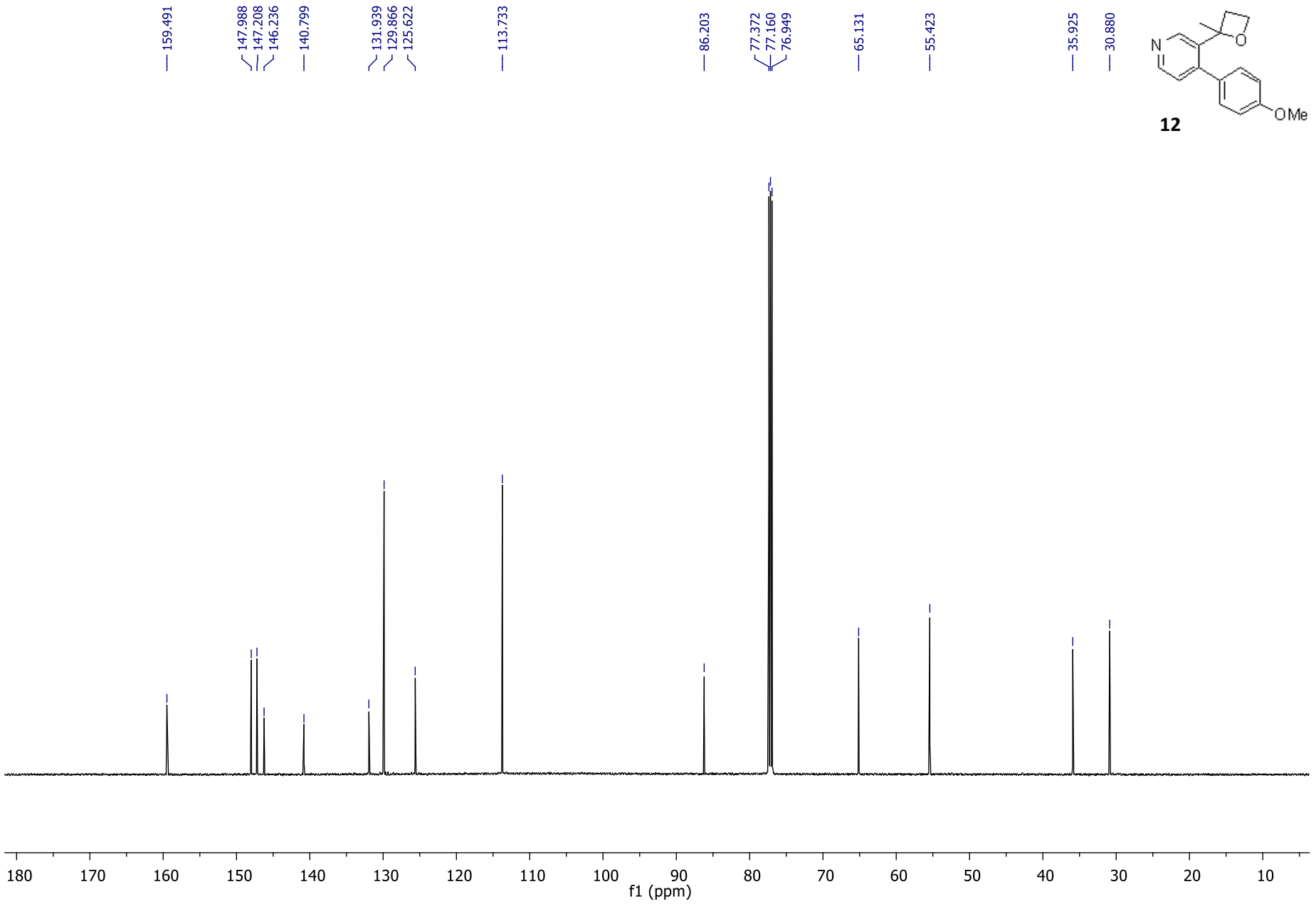

8.887
8.545
8.537
7.399
7.385
7.260
7.180
7.166
7.041
7.033
4.590
4.579
4.577
4.566
4.564
4.553
4.402
4.393
4.387
4.383
4.377
4.367
2.759
2.747
2.744
2.741
2.731
2.728
2.726
2.713
2.362
2.352
2.348
2.343
2.338
2.334
2.330
1.842
N
O
Cl
1.00
1.03
2.06
2.10
1.04
1.08
1.07
1.08
1.08
3.18
10.0
9.5
9.0
8.5
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
f1 (ppm)

11


11
N
O
Cl
148.271
147.465
145.197
140.554
138.229
134.335
130.084
128.553
125.224
86.001
77.371
77.160
76.948
65.152
36.048
31.040
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


8.885
8.513
8.504
7.260
7.148
7.134
7.065
7.057
6.938
6.924
4.582
4.572
4.569
4.559
4.556
4.546
4.397
4.387
4.381
4.377
4.371
4.362
3.847
2.753
2.740
2.737
2.734
2.724
2.721
2.719
2.706
2.324
2.320
2.315
2.310
2.305
2.301
1.645
N
O
OMe
1.00
1.03
2.07
1.02
2.05
1.06
1.06
3.09
1.07
1.06
3.08
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)

12


159.491
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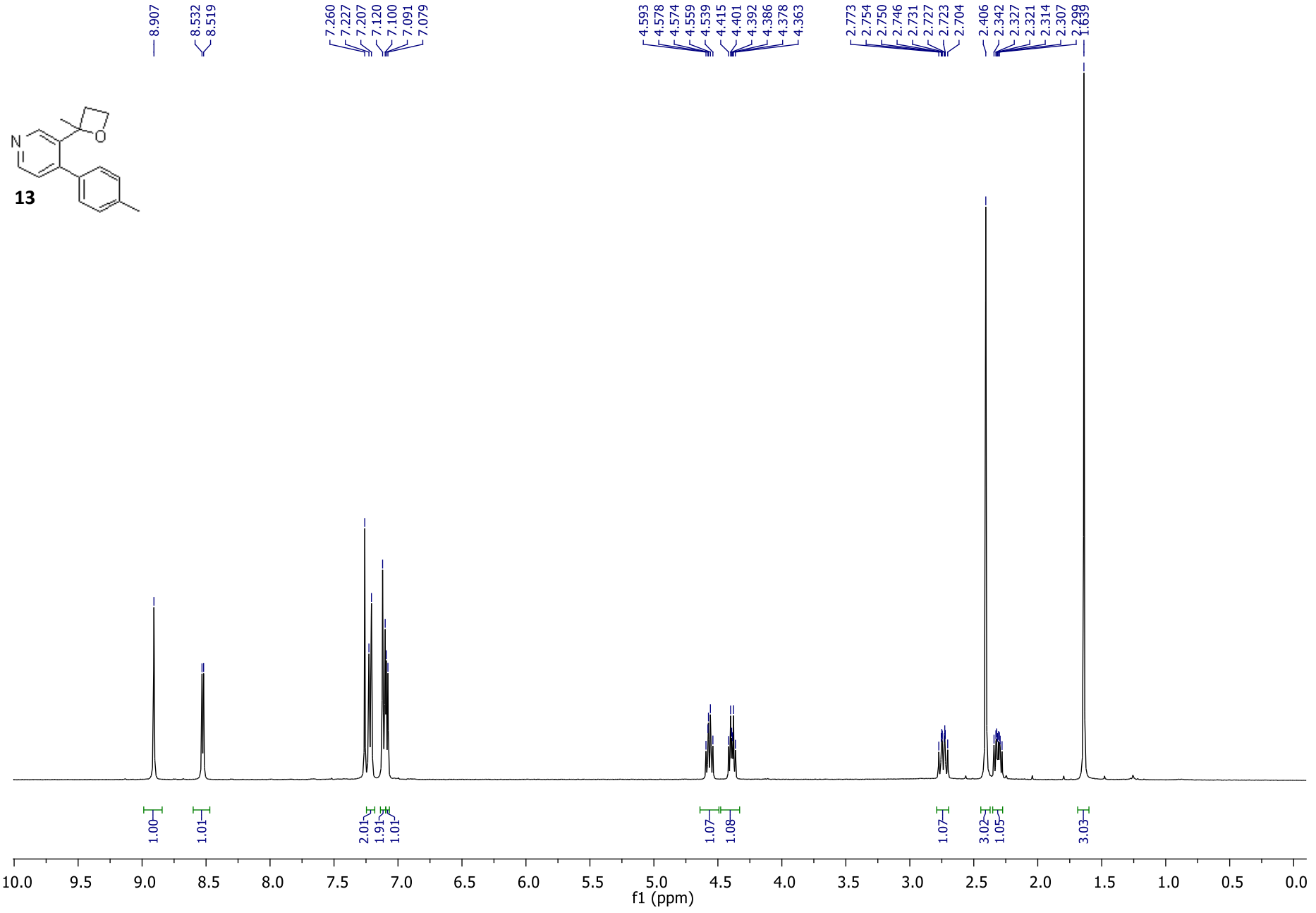
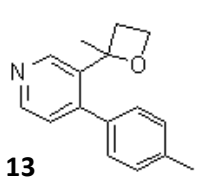
147.208
146.236
140.799
131.939
129.866
125.622
113.733
86.203
77.372
77.160
76.949
65.131
55.423
35.925
30.880
N
O
OMe
12
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


8.907
8.532
8.519
7.260
7.227
7.207
7.120
7.100
7.091
7.079
4.593
4.578
4.574
4.559
4.539
4.415
4.401
4.392
4.386
4.378
4.363
2.773
2.754
2.750
2.746
2.731
2.727
2.723
2.704
2.406
2.342
2.327
2.321
2.314
2.307
2.298
1.639
N
O
13
1.00
1.01
2.01
1.91
1.01
1.07
1.08
1.07
3.02
1.05
3.03
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
0.0
f1 (ppm)



13

147.988
147.171
146.455
140.645
137.907
136.772
128.950
128.523
125.435
86.187
77.478
77.160
76.842
65.105
35.945
30.885
21.303