Supporting Information for

Visible-Light-Induced Selective Aerobic Oxidation of sp^3 C-H bonds Catalyzed by Heterogeneous AgI/$BiVO_4$ catalyst

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Contents:

1. General Considerations

All the reagents were purchased from commercial sources and were used without further purification, unless otherwise illustrated. Column chromatography was performed with silica gel (200-300 mesh). Analytical TLC plates were Qingdaohaiyang GF_{254} and were viewed by UV light (254 nm). ^{1}H, ^{13}C and ^{19}F NMR spectras were recorded on a Bruker DRX-400 spectrometer and all chemical shift values refer to δ_{TMS} = 0.00 ppm, DMSO-d_6 ($\delta(^{1}H)$, 2.50 ppm; $\delta(^{13}C)$, 39.52 ppm). All the melting points were uncorrected. The microstructures were tested by scanning electron microscope (SEM, JEOL JSM-7800F) and transmission electron microscope (TEM, HITACHI HT7700). The power X-ray diffraction (XRD) were performed on a Rigaku D/Max 2500PC diffractometer equipped with a Cu Ka radiation source (λ = 1.5418 Å) at a scanning rate of 5° min^{-1} (2θ from 10° to 70°). Surface compositions were determined by X-ray photoelectron spectroscopy (XPS) using Thermo VG ESCALAB250 instrument with Al Kα radiation anode (hv =1486.6 eV), and the C1s line (284.6 eV) was used as the reference to correct the binding energies (BE). The electron paramagnetic resonance (EPR) signals of the radicals spin-trapped with 5, 5-dimethyl-1-pyrroline-N-oxide (DMPO) were recorded on a Bruker ER200-SRC spectrometer at ambient temperature.

2. Setup for Photocatalytic Reactions

The reaction setup is depicted in Figure S1. The reaction setup consists of a quartz glass reactor (Figure S1**a**), light source, circulating water equipment. The light source is commercially available 16 W white LED. Circulating water equipment (ensure constant 25 °C) is cryogenic circulating pump. Magnetic stirring is performed with 200 rpm.

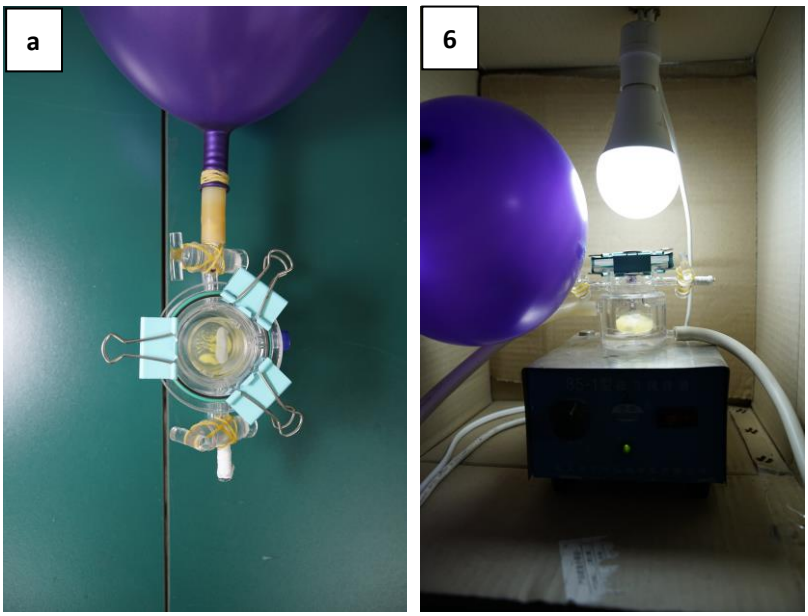
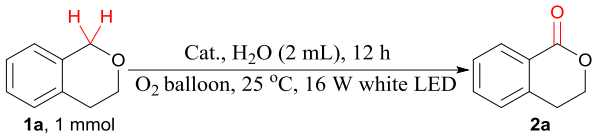


Figure S1. Photocatalytic reactions setup.

3. Table S1. Optimization of Reaction Conditions



Entry	Cat. (mg)	Yield[a] (%)
1	1wt% AgI/$BiVO_4$ (20)	17
2	5wt% AgI/$BiVO_4$ (20)	55
3	10wt% AgI/$BiVO_4$ (20)	71
4	40wt% AgI/$BiVO_4$ (20)	76
5	50wt% AgI/$BiVO_4$ (20)	77
6	60wt% AgI/$BiVO_4$ (20)	75
7	80wt% AgI/$BiVO_4$ (20)	73
8[b]	20wt% AgI/$BiVO_4$ (15)	70

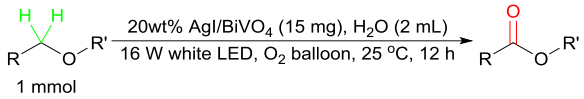
[a]Isolated yield, n. r. = no reaction. [b]The oxidant is air, and the reaction time is 16 h.

4. The preparation of AgI/$BiVO_4$ catalysts

Firstly, $BiVO_4$ was prepared through a facile water bath process. While stirring, $Bi(NO_3)_3 \cdot 5H_2O$ (1.94 g, 4 mmol) was initially dispersed into 80 mL mixed solution of ethanol: HOAc: H_2O (1:1:3, volume ratio). Meanwhile, 4 mmol NH_4VO_3 was also dispersed into 80 mL ammonia solution ($NH_3 \cdot H_2O$: H_2O = 3:1, volume ratio). After two clear solutions were achieved by ultrasonication, the latter was added dropwise into the former, followed by 12 h stirring at 25 °C. The $BiVO_4$ samples was achieved by centrifugation, washed with deionized water (5 times) and vacuum freeze-drying.

The AgI/$BiVO_4$ catalysts were prepared by an *in situ* deposition-precipitation procedure. Typically, 1 mmol $BiVO_4$ was dispersed in 50 mL deionized water under ultrasonic processing. After that, 0.345 mmol $AgNO_3$ was slowly added into the above suspension. After 30 min stirring in dark, 25 mL solution containing 0.345 mmol KI was slowly added into above suspension. The suspension was stirred for another 12 h at 25 °C to synthesize the 20wt% AgI/$BiVO_4$ catalyst (the theoretical mass ratios of AgI to (AgI+$BiVO_4$)). The 20wt% AgI/$BiVO_4$ catalyst was obtained by centrifugation, washed with deionized water (5 times) and vacuum freeze-drying. The 1wt%, 5wt%, 10wt%, 40wt%, 60wt% and 80wt% AgI/$BiVO_4$ catalysts were prepared according to the above procedure. For comparison, bare AgI nanoparticle was also fabricated under the identical conditions as AgI/$BiVO_4$ in the absence of $BiVO_4$.

5. General Experimental Procedure



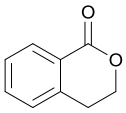
A quartz glass reactor was subsequently charged with 1.0 mmol ether, 15 mg 20wt% AgI/$BiVO_4$, 2 mL H_2O. Oxygen atmosphere was incorporated through an O_2 balloon. The resulting mixture was performed under a 16 W white LED at 25 °C for 12 h. After reaction was complete, the resulting mixture was extracted with ethyl acetate (3×10 mL). The combined organic extracts was washed with brine (3×5 mL), then dried over Na_2SO_4 and concentrated in vacuum. The resulting residue was purified by silica gel column chromatography (ethyl acetate/petroleum ether = 1:20-1:5) to afford the desired products.

6. Experimental Procedure on Gram Scale

A quartz glass reactor was subsequently charged with 10 mmol ether, 150 mg 20wt% AgI/$BiVO_4$, 20 mL H_2O. Oxygen atmosphere was incorporated through an O_2 balloon. The resulting mixture was performed under a 16 W white LED at 25 °C for 72 h. After reaction was complete, the resulting mixture was extracted with ethyl acetate (3×50 mL). The combined organic extracts was washed with brine (3×50 mL), then dried over Na_2SO_4 and concentrated in vacuum. The resulting residue was purified by silica gel column chromatography (ethyl acetate/petroleum ether = 1:20-1:5) to afford the desired products.

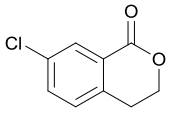
7. ^{1}H, ^{13}C and ^{19}F NMR Datas

Isochroman-1-one **2a**



Compound **2a** was isolated in 83% yield (123.3 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.93 (d, J = 7.7 Hz, 1H), 7.65-7.52 (m, 1H), 7.47-7.31 (m, 2H), 4.49 (t, J = 6.0 Hz, 2H), 3.10-2.97 (m, 2H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 164.37, 140.21, 133.58, 129.37, 127.60, 127.35, 124.95, 67.13, 26.98; Known compound.[1]

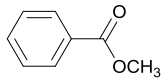
7-Chloroisochroman-1-one **2b**



Compound **2b** was isolated in 92% yield (166.9 mg). Yellow oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.91 (d, J = 8.3 Hz, 1H), 7.55 (d, J = 1.8 Hz, 1H), 7.49 (dd, J = 8.3, 2.2 Hz, 1H), 4.53-4.48 (m, 2H), 3.07 (t, J = 6.0 Hz, 2H); ^{13}C NMR (101 MHz, DMSO-d_6)

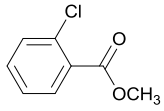
δ 163.69, 142.47, 138.39, 131.38, 127.62, 127.52, 123.85, 67.07, 26.78; Known compound.[1]

Methyl benzoate **2c**



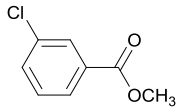
Compound **2c** was isolated in 87% yield (118.9 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.96 (dd, J = 8.2, 1.1 Hz, 2H), 7.69-7.61 (m, 1H), 7.51 (t, J = 7.3 Hz, 2H), 3.85 (s, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 166.16, 133.18, 129.60, 129.04, 128.65, 51.99; Known compound.[2]

Methyl 2-chlorobenzoate **2d**



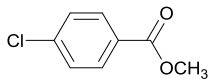
Compound **2d** was isolated in 71% yield (120.4 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.79 (d, J = 7.6 Hz, 1H), 7.61-7.32 (m, 3H), 3.86 (s, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 165.46, 133.05, 131.73, 130.91, 130.67, 130.04, 127.25, 52.39; Known compound.[6]

Methyl 3-chlorobenzoate **2e**



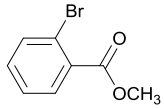
Compound **2e** was isolated in 74% yield (126.0 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.87 (d, J = 14.0 Hz, 2H), 7.75-7.64 (m, 1H), 7.59-7.48 (m, 1H), 3.86 (s, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 164.96, 133.45, 133.06, 131.59, 130.74, 128.61, 127.71, 52.42; Known compound.[6]

Methyl 4-chlorobenzoate **2f**



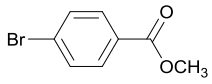
Compound **2f** was isolated in 72% yield (122.7 mg). White solid; mp: 42-44 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.88 (dd, J = 8.5, 1.8 Hz, 2H), 7.56-7.46 (m, 2H), 3.83 (s, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 165.23, 138.19, 130.78, 128.67, 128.32, 52.06; Known compound.[3]

Methyl 2-bromobenzoate **2g**



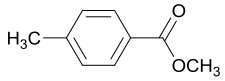
Compound **2g** was isolated in 80% yield (171.8 mg). Yellow oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.81-7.67 (m, 2H), 7.52-7.37 (m, 2H), 3.86 (s, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 165.93, 133.80, 132.85, 132.15, 130.75, 127.76, 120.00, 52.30; Known compound.[5]

Methyl 4-bromobenzoate **2h**



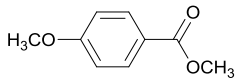
Compound **2h** was isolated in 88% yield (188.7 mg). White solid; mp: 77-80 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.85 (d, J = 8.6 Hz, 2H), 7.70 (d, J = 8.6 Hz, 2H), 3.84 (s, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 165.46, 131.79, 130.99, 128.73, 127.27, 52.24; Known compound.[3]

Methyl 4-methylbenzoate **2i**



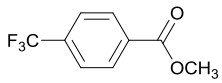
Compound **2i** was isolated in 89% yield (133.1 mg). White solid; mp: 33-35 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.84 (d, J = 8.2 Hz, 2H), 7.28 (d, J = 8.3 Hz, 2H), 3.82 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 166.13, 143.41, 129.14, 129.07, 126.90, 51.74, 20.99; Known compound.[3]

Methyl 4-methoxybenzoate **2j**



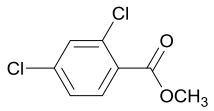
Compound **2j** was isolated in 91% yield (151.7 mg). White solid; mp: 52-53 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.90 (d, J = 8.8 Hz, 2H), 7.02 (d, J = 8.2 Hz, 2H), 3.82 (s, 3H), 3.81 (s, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 165.84, 163.08, 131.15, 121.84, 113.91, 55.39, 51.66; Known compound.[3]

Methyl 4-(trifluoromethyl)benzoate **2k**



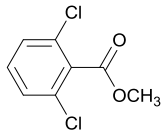
Compound **2k** was isolated in 67% yield (136.9 mg). Yellow oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.15 (d, J = 7.6 Hz, 2H), 7.89 (d, J = 7.2 Hz, 2H), 3.90 (s, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 165.12, 133.27, 132.79 (d, J = 32.0 Hz), 132.32, 127.84 (d, J = 427.8 Hz), 122.31, 52.57; ^{19}F NMR (377 MHz, DMSO-d_6) δ -61.72; Known compound.[4]

Methyl 2,4-dichlorobenzoate **2l**



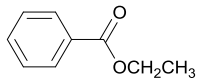
Compound **2l** was isolated in 74% yield (150.5 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.81 (ddd, J = 15.1, 8.4, 5.1 Hz, 1H), 7.75-7.59 (m, 1H), 7.51 (ddd, J = 26.7, 15.9, 6.3 Hz, 1H), 3.86 (s, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 164.56, 137.15, 133.17, 132.46, 130.30, 128.64, 127.58, 52.59; Known compound.[7]

Methyl 2,6-dichlorobenzoate **2m**



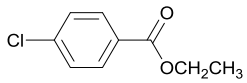
Compound **2m** was isolated in 71% yield (145.4 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.60-7.43 (m, 3H), 3.92 (s, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 164.50, 132.79, 132.24, 130.51, 128.30, 53.12; Known compound.[5]

Ethyl benzoate **2n**



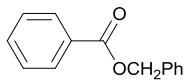
Compound **2n** was isolated in 80% yield (120.5 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.96 (d, J = 7.0 Hz, 2H), 7.64 (s, 1H), 7.51 (t, J = 6.9 Hz, 2H), 4.30 (q, J = 7.1 Hz, 2H), 1.31 (t, J = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 165.64, 133.09, 129.87, 128.99, 128.59, 60.60, 14.04; Known compound.[8]

Ethyl 4-chlorobenzoate **2o**



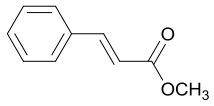
Compound **2o** was isolated in 83% yield (153.3 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.98-7.89 (m, 2H), 7.61-7.52 (m, 2H), 4.30 (q, J = 7.1 Hz, 2H), 1.31 (t, J = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 164.79, 138.11, 130.83, 128.78, 128.65, 60.91, 13.99; Known compound.[9]

Benzyl benzoate **2p**



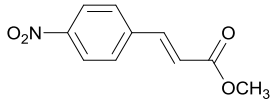
Compound **2p** was isolated in 78% yield (165.6 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.04-7.98 (m, 2H), 7.62 (dd, J = 9.5, 4.0 Hz, 1H), 7.56-7.44 (m, 4H), 7.37 (ddd, J = 14.5, 10.3, 3.6 Hz, 3H), 5.36 (s, 2H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 165.51, 136.08, 133.24, 129.58, 129.14, 128.62, 128.42, 128.00, 127.88, 66.10; Known compound.[10]

Methyl cinnamate **2q**



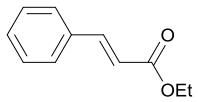
Compound **2q** was isolated in 71% yield (115.7 mg). White solid; mp: 35-37 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.73-7.69 (m, 2H), 7.67 (d, J = 16.2 Hz, 1H), 7.44-7.39 (m, 3H), 6.63 (d, J = 16.1 Hz, 1H), 3.73 (s, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 166.59, 144.46, 133.96, 130.39, 128.84, 128.27, 117.78, 51.37; Known compound.[6]

Methyl 3-(4-nitrophenyl)acrylate **2r**



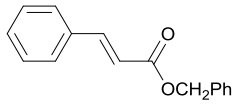
Compound **2r** was isolated in 49% yield (100.8 mg). White solid; mp: 161-164 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.23 (d, J = 8.8 Hz, 2H), 7.99 (d, J = 8.8 Hz, 2H), 7.75 (d, J = 16.1 Hz, 1H), 6.84 (d, J = 16.1 Hz, 1H), 3.75 (s, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 166.09, 148.05, 141.89, 140.39, 129.40, 123.87, 122.07, 51.71; HRMS (ESI) for $C_{10}H_9NO_4$, calcd: 207.0532, found: 207.0567.

Ethyl cinnamate **2s**



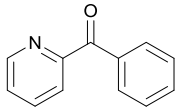
Compound **2s** was isolated in 69% yield (121.8 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.74-7.68 (m, 2H), 7.65 (d, J = 16.1 Hz, 1H), 7.46-7.38 (m, 3H), 6.61 (d, J = 16.0 Hz, 1H), 4.19 (q, J = 7.1 Hz, 2H), 1.25 (t, J = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 166.10, 144.26, 133.99, 130.32, 128.82, 128.23, 118.12, 59.93, 14.11; HRMS (ESI) for $C_{11}H_{12}O_2$, calcd: 176.0837, found: 176.0811.

Benzyl cinnamate **2t**



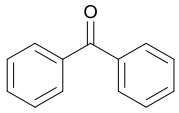
Compound **2t** was isolated in 59% yield (140.2 mg). White solid; mp: 33-36 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.77-7.67 (m, 3H), 7.47-7.31 (m, 8H), 6.70 (d, J = 16.1 Hz, 1H), 5.24 (s, 2H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 166.00, 144.80, 136.18, 133.94, 130.45, 128.84, 128.40, 128.33, 128.03, 128.00, 117.83, 65.57; HRMS (ESI) for $C_{16}H_{14}O_2$, calcd: 238.0994, found: 238.0951.

2-Benzoylpyridine **4a**



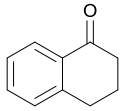
Compound **4a** was isolated in 31% yield (5.7 mg). White solid; mp: 40-42 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.72 (d, J = 4.7 Hz, 1H), 8.11-8.03 (m, 1H), 8.03-7.93 (m, 3H), 7.66 (dd, J = 8.8, 5.2 Hz, 2H), 7.54 (t, J = 7.6 Hz, 2H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 193.41, 154.48, 148.56, 137.64, 136.00, 132.97, 130.59, 128.20, 126.72, 124.14; Known compound.[11]

Benzophenone **4b**



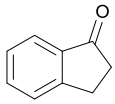
Compound **4b** was isolated in 44% yield (8.0 mg). White solid; mp: 48-50 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.76-7.71 (m, 4H), 7.71-7.65 (m, 2H), 7.57 (t, J = 7.6 Hz, 4H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 195.82, 137.00, 132.69, 129.60, 128.57; Known compound.[12]

3,4-Dihydronaphthalen-1(2*H*)-one **4c**



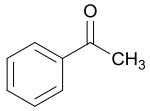
Compound **4c** was isolated in 40% yield (5.9 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.86 (dd, J = 8.1, 1.4 Hz, 1H), 7.54 (td, J = 7.5, 1.4 Hz, 1H), 7.34 (t, J = 7.1 Hz, 2H), 2.94 (t, J = 6.1 Hz, 2H), 2.63-2.56 (m, 2H), 2.09-1.98 (m, 2H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 197.49, 144.68, 133.43, 132.14, 129.01, 126.51, 126.22, 38.60, 28.86, 22.85; Known compound.[12]

1-Indanone **4d**



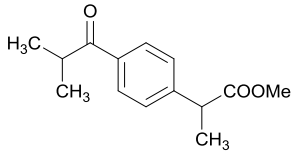
Compound **4d** was isolated in 42% yield (5.6 mg). White solid; mp: 38-40 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.69-7.54 (m, 3H), 7.44-7.38 (m, 1H), 3.13-3.07 (m, 2H), 2.65-2.59 (m, 2H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 206.35, 155.29, 136.67, 134.65, 127.25, 127.03, 122.89, 35.85, 25.42; Known compound.[13]

Acetophenone **4e**



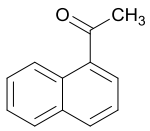
Compound **4e** was isolated in 46% yield (5.5 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.96 (dt, J = 8.4, 1.7 Hz, 2H), 7.66-7.61 (m, 1H), 7.52 (dd, J = 10.7, 4.8 Hz, 2H), 2.58 (s, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 197.95, 136.81, 133.18, 128.68, 128.15, 26.71; Known compound.[13]

Methyl 2-(4-isobutyrylphenyl)propanoate **4f**



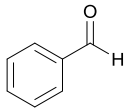
Compound **4f** was isolated in 32% yield (7.6 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.93 (d, J = 8.4 Hz, 2H), 7.43 (d, J = 8.3 Hz, 2H), 3.92 (q, J = 7.1 Hz, 1H), 3.64 (dd, J = 13.6, 6.8 Hz, 1H), 3.59 (s, 3H), 1.41 (d, J = 7.1 Hz, 3H), 1.10 (d, J = 6.8 Hz, 6H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 203.39, 173.78, 145.74, 134.49, 128.57, 127.90, 51.95, 44.31, 34.51, 19.00, 18.31; Known compound.[14]

Methyl 2-(4-isobutyrylphenyl)propanoate **4g**



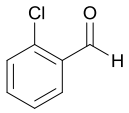
Compound **4g** was isolated in 41% yield (7.1 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.62 (d, J = 8.3 Hz, 1H), 8.17-8.11 (m, 2H), 8.04-7.98 (m, 1H), 7.67-7.54 (m, 3H), 2.72 (s, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 201.72, 134.88, 133.50, 132.79, 129.38, 129.21, 128.50, 127.85, 126.35, 125.48, 124.83, 30.04; Known compound.[15]

Benzaldehyde **6a**



Compound **6a** was isolated in 48% yield (6.2 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.02 (s, 1H), 7.92 (dt, J = 8.3, 1.5 Hz, 2H), 7.76-7.69 (m, 1H), 7.61 (dd, J = 10.5, 4.6 Hz, 2H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 193.24, 136.19, 134.58, 129.48, 129.16; Known compound.[16]

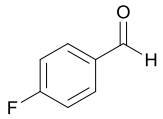
2-Chlorobenzaldehyde **6b**



Compound **6b** was isolated in 45% yield (6.3 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.32 (s, 1H), 7.89-7.81 (m, 1H), 7.72-7.64 (m, 1H), 7.59 (t, J = 8.9 Hz,

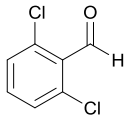
1H), 7.51 (q, *J* = 6.8 Hz, 1H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 189.68, 136.27, 135.70, 132.01, 130.67, 129.59, 127.82; Known compound.[17]

4-Fluorobenzaldehyde **6c**



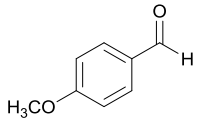
Compound **6c** was isolated in 44% yield (5.5 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.98 (s, 1H), 8.04-7.97 (m, 2H), 7.48-7.41 (m, 2H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 191.68, 167.00, 164.49, 133.08 (d, *J* = 2.3 Hz), 132.38 (d, *J* = 10.0 Hz), 116.49, 116.27; ^{19}F NMR (377 MHz, DMSO-d_6) δ -103.57; Known compound.[16]

2,6-Dichlorobenzaldehyde **6d**



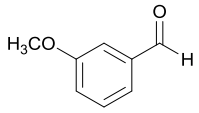
Compound **6d** was isolated in 42% yield (7.3 mg). White solid; mp: 70-73 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.35 (s, 1H), 7.62-7.58 (m, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 189.45, 135.07, 134.58, 130.30, 129.96; Known compound.[17]

4-Methoxybenzaldehyde **6e**



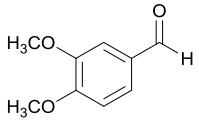
Compound **6e** was isolated in 53% yield (7.2 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.87 (s, 1H), 7.90-7.84 (m, 2H), 7.16-7.10 (m, 2H), 3.86 (s, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 191.31, 164.22, 131.81, 129.65, 114.51, 55.69; Known compound.[16]

3-Methoxybenzaldehyde **6f**



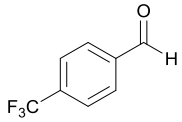
Compound **6f** was isolated in 51% yield (7.0 mg). Colourless oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.98 (s, 1H), 7.56-7.50 (m, 2H), 7.44-7.41 (m, 1H), 7.28 (dt, *J* = 6.9, 2.6 Hz, 1H), 3.83 (s, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 192.98, 159.76, 137.64, 130.36, 122.48, 120.97, 112.92, 55.39; Known compound.[18]

3,4-Dimethoxybenzaldehyde **6g**



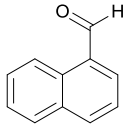
Compound **6g** was isolated in 57% yield (9.5 mg). White solid; mp: 41-43 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.85 (s, 1H), 7.56 (d, J = 8.2 Hz, 1H), 7.40 (s, 1H), 7.18 (d, J = 8.2 Hz, 1H), 3.88 (s, 3H), 3.84 (s, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 191.37, 154.20, 149.18, 129.65, 126.12, 111.26, 109.40, 55.87, 55.50; Known compound.[19]

4-(Trifluoromethyl)benzaldehyde **6h**



Compound **6h** was isolated in 37% yield (6.5 mg). Yellow oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.13 (s, 1H), 8.12 (d, J = 8.0 Hz, 2H), 7.98 (d, J = 8.2 Hz, 2H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 192.67, 138.91, 133.70, 133.38, 130.15, 126.13 (q, J = 3.8 Hz), 125.02; ^{19}F NMR (377 MHz, DMSO-d_6) δ -61.69; Known compound.[16]

1-Naphthaldehyde **6i**



Compound **6i** was isolated in 40% yield (6.3 mg). Yellow oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.41 (s, 1H), 9.16 (d, J = 8.5 Hz, 1H), 8.29 (d, J = 8.2 Hz, 1H), 8.20 (d, J = 7.0 Hz, 1H), 8.09 (d, J = 8.1 Hz, 1H), 7.75 (dd, J = 15.4, 7.3 Hz, 2H), 7.66 (t, J = 7.5 Hz, 1H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 194.43, 136.80, 135.27, 133.32, 130.85, 129.76, 129.04, 128.73, 126.94, 125.41, 124.11; Known compound.[16]

Reference

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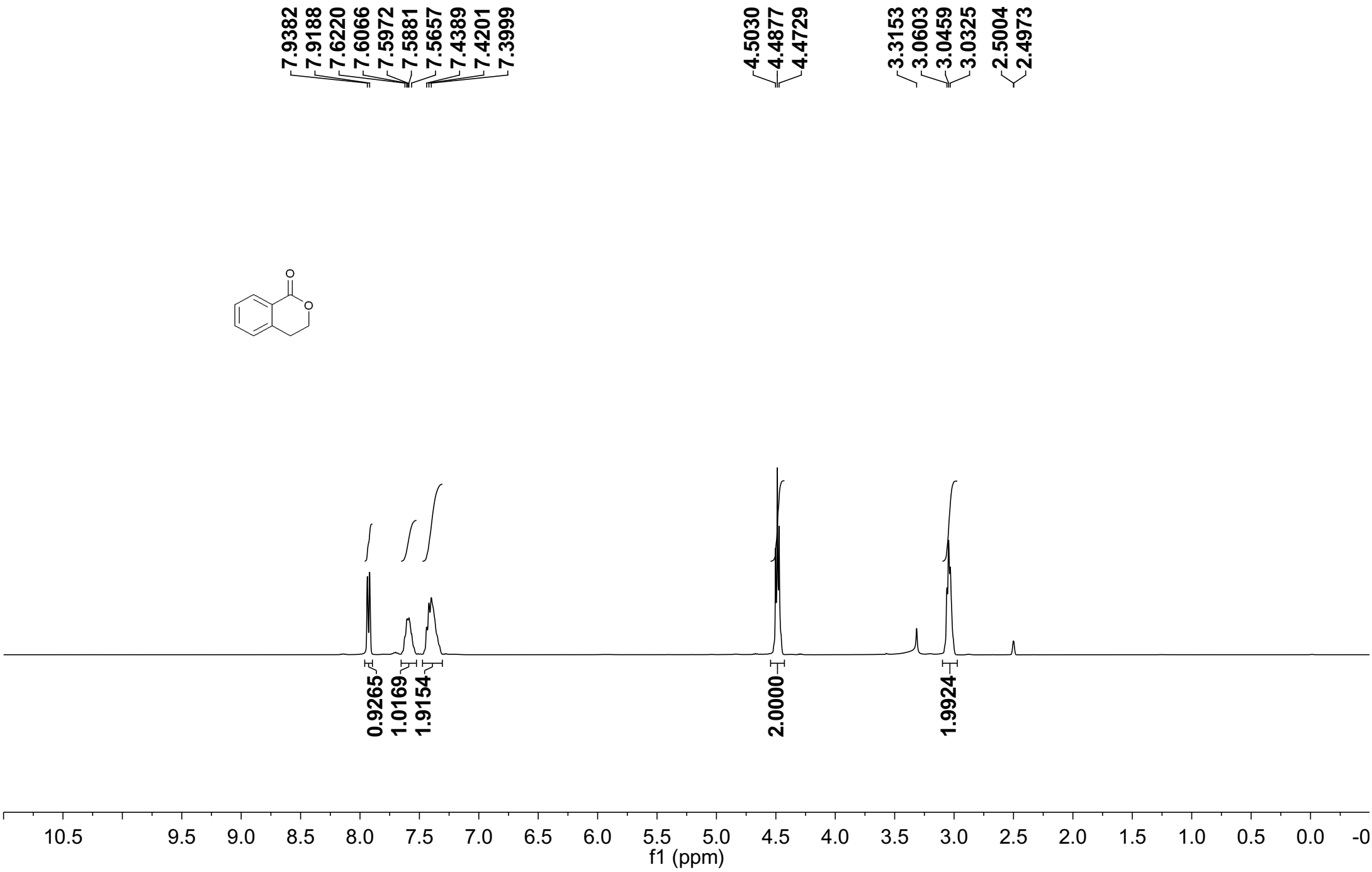
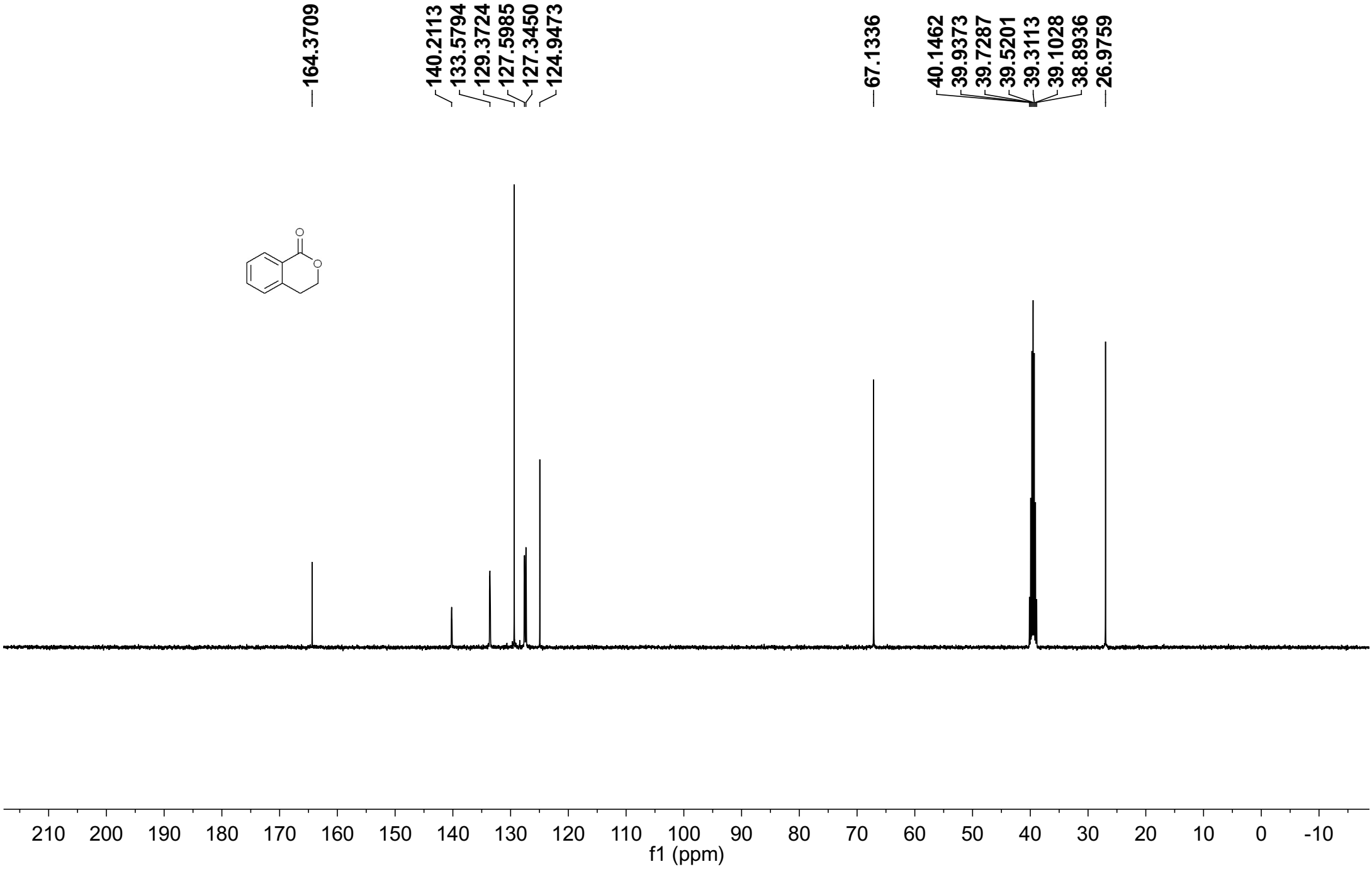
8. ^{1}H, ^{13}C and ^{19}F NMR Spectras

Compound 2a



Compound 2a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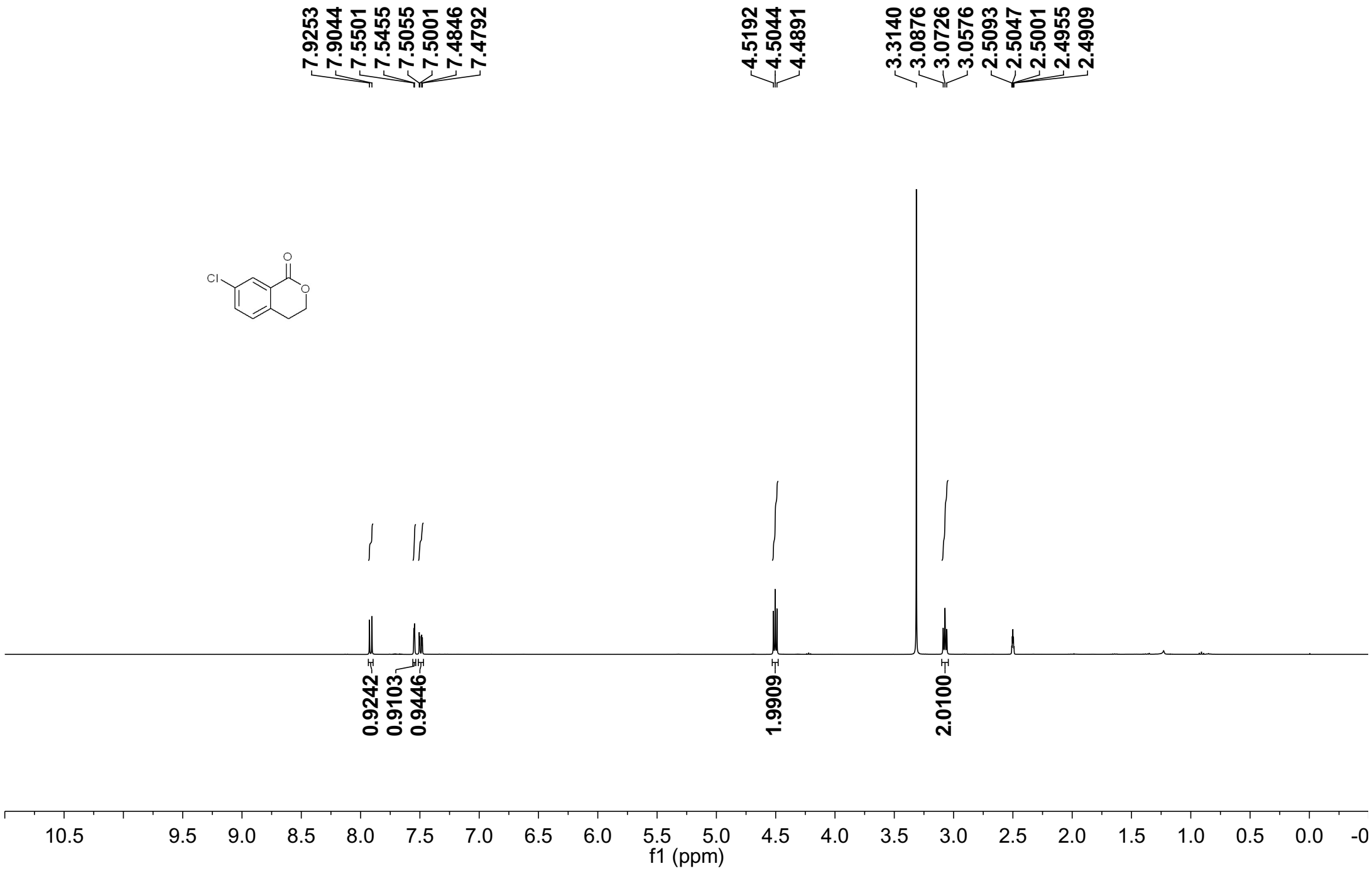
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Compound 2b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4.5
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3.5
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2.5
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1.5
1.0
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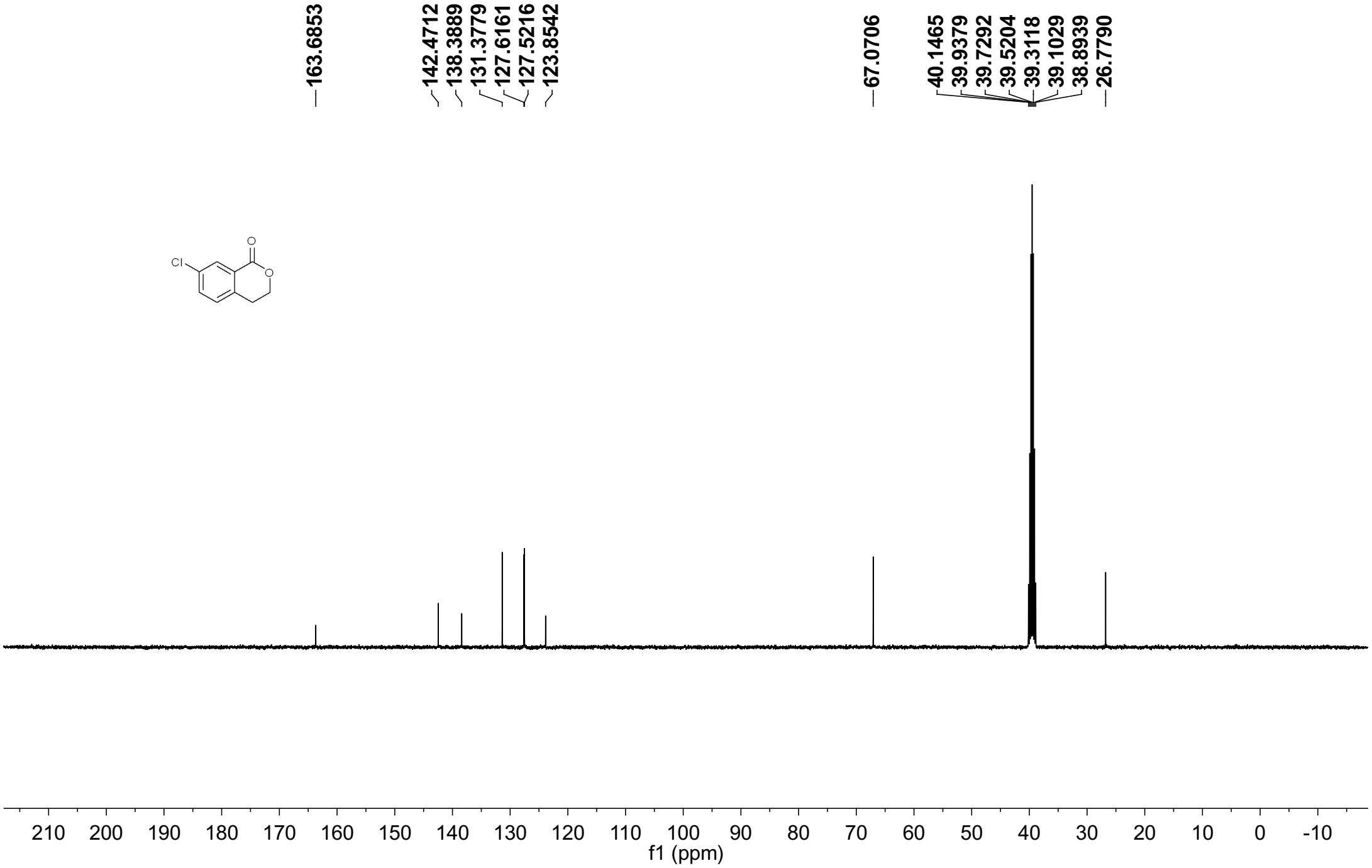
-0
f1 (ppm)

Compound 2b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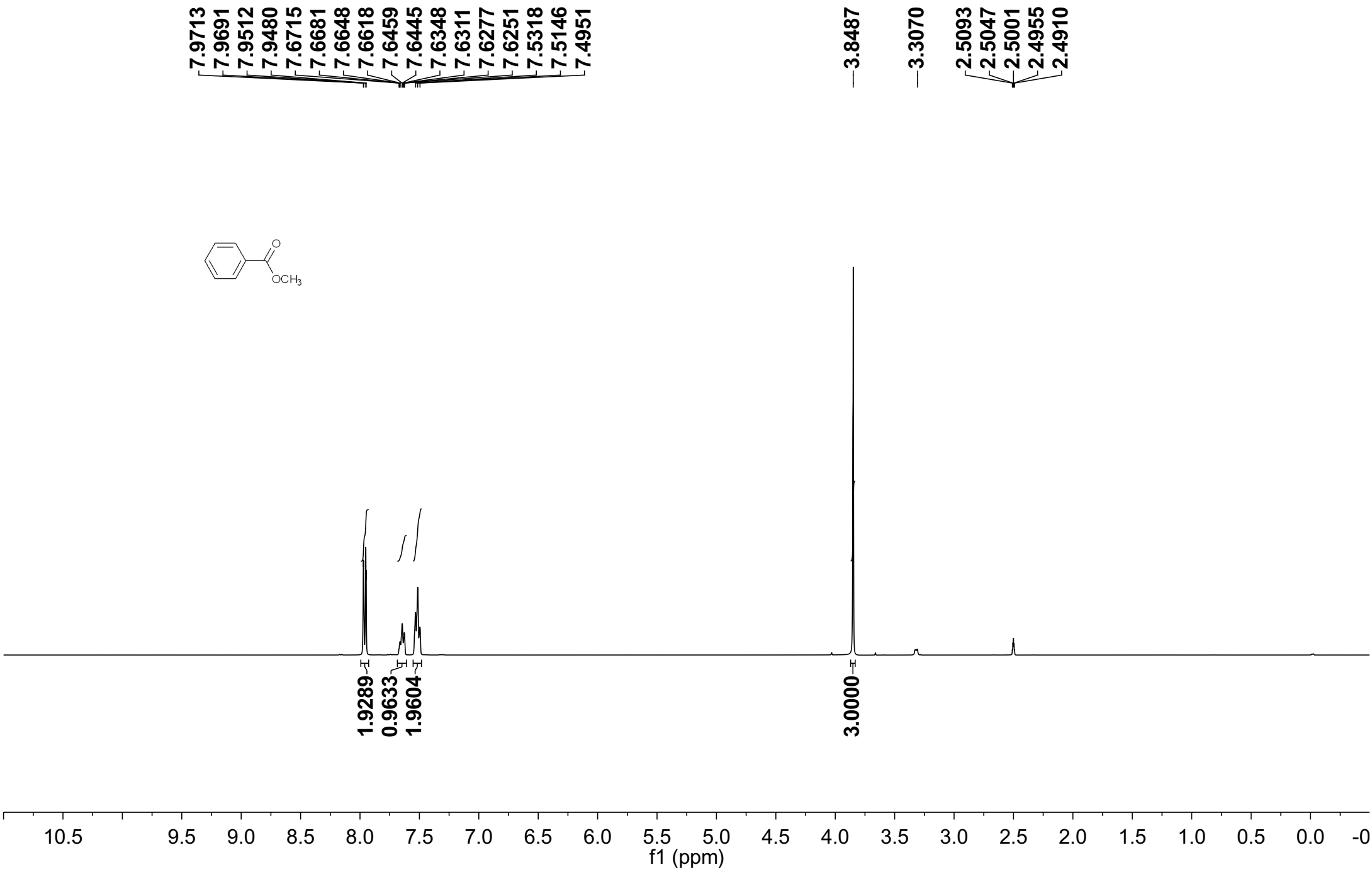
f1 (ppm)

Compound 2c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6.0
5.5
5.0
4.5
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2.5
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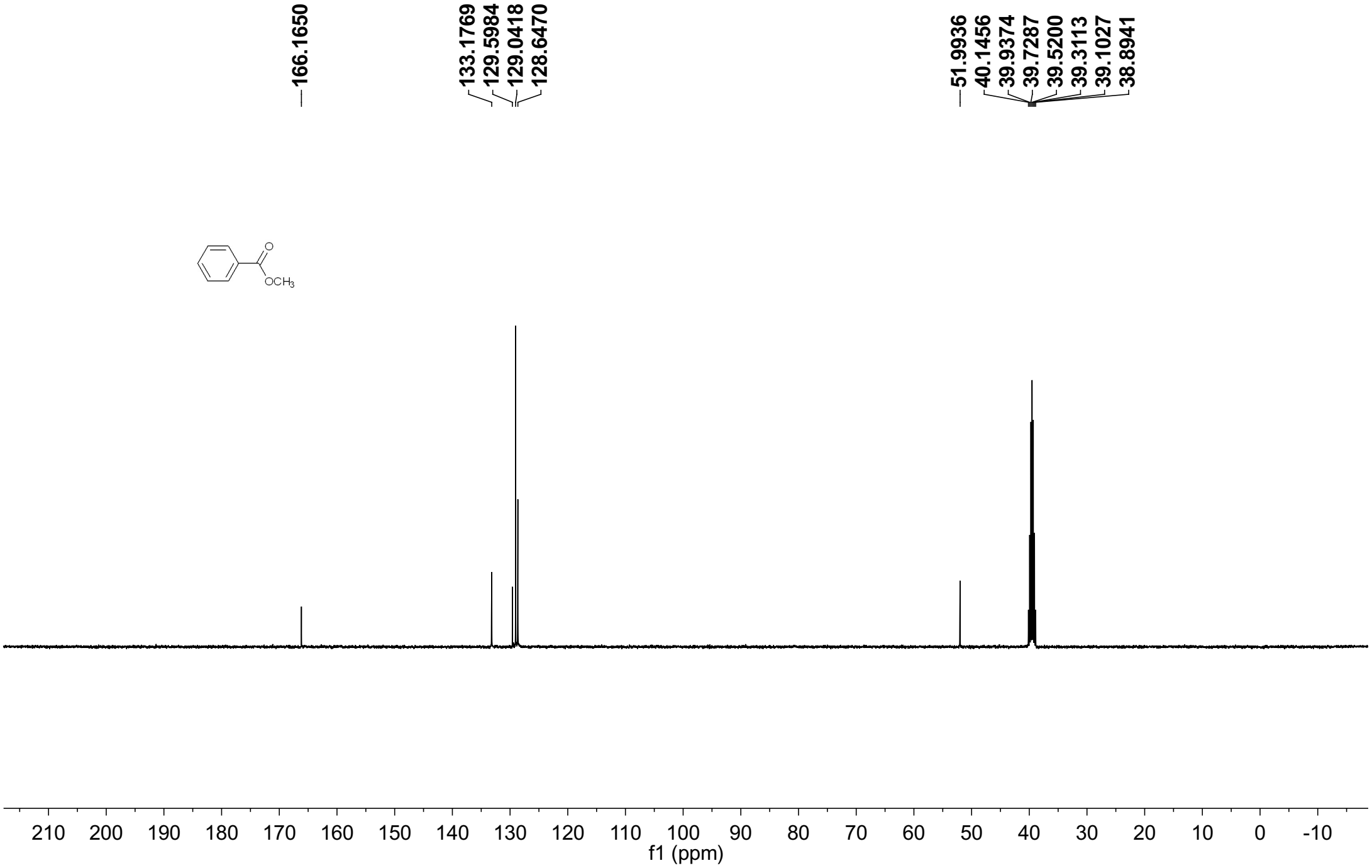
0.5
0.0
-0
f1 (ppm)

Compound 2c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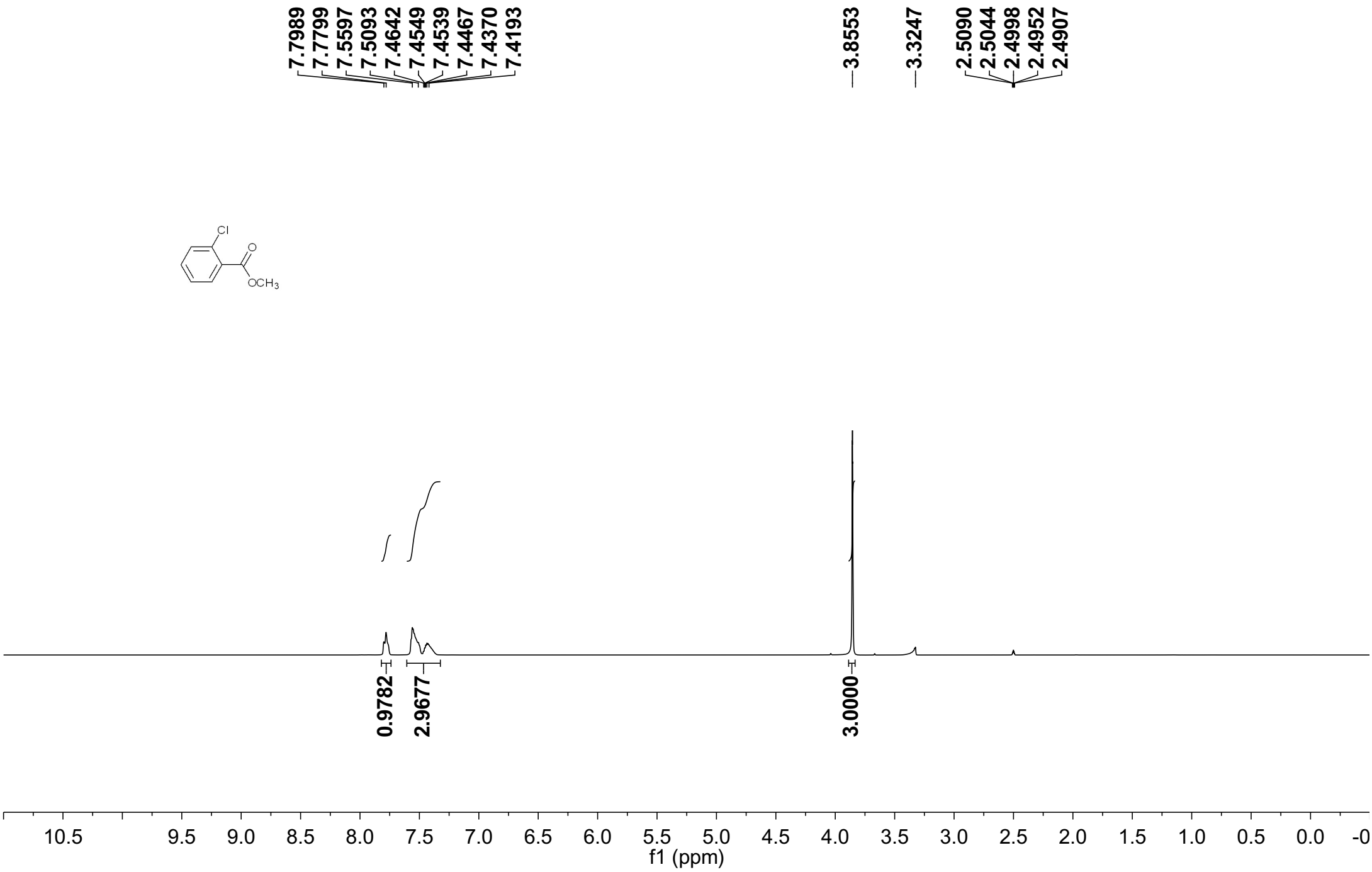
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Compound 2d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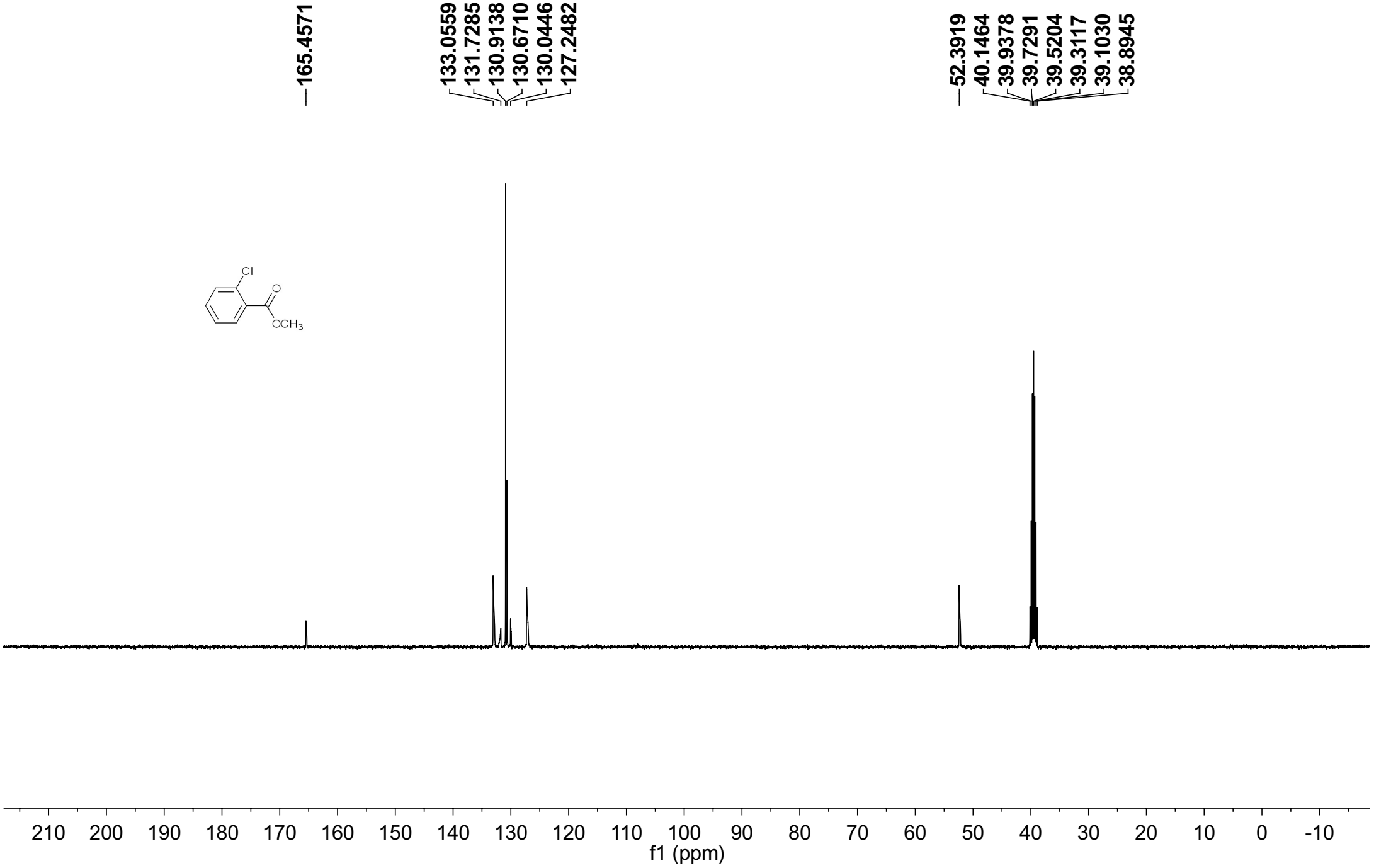
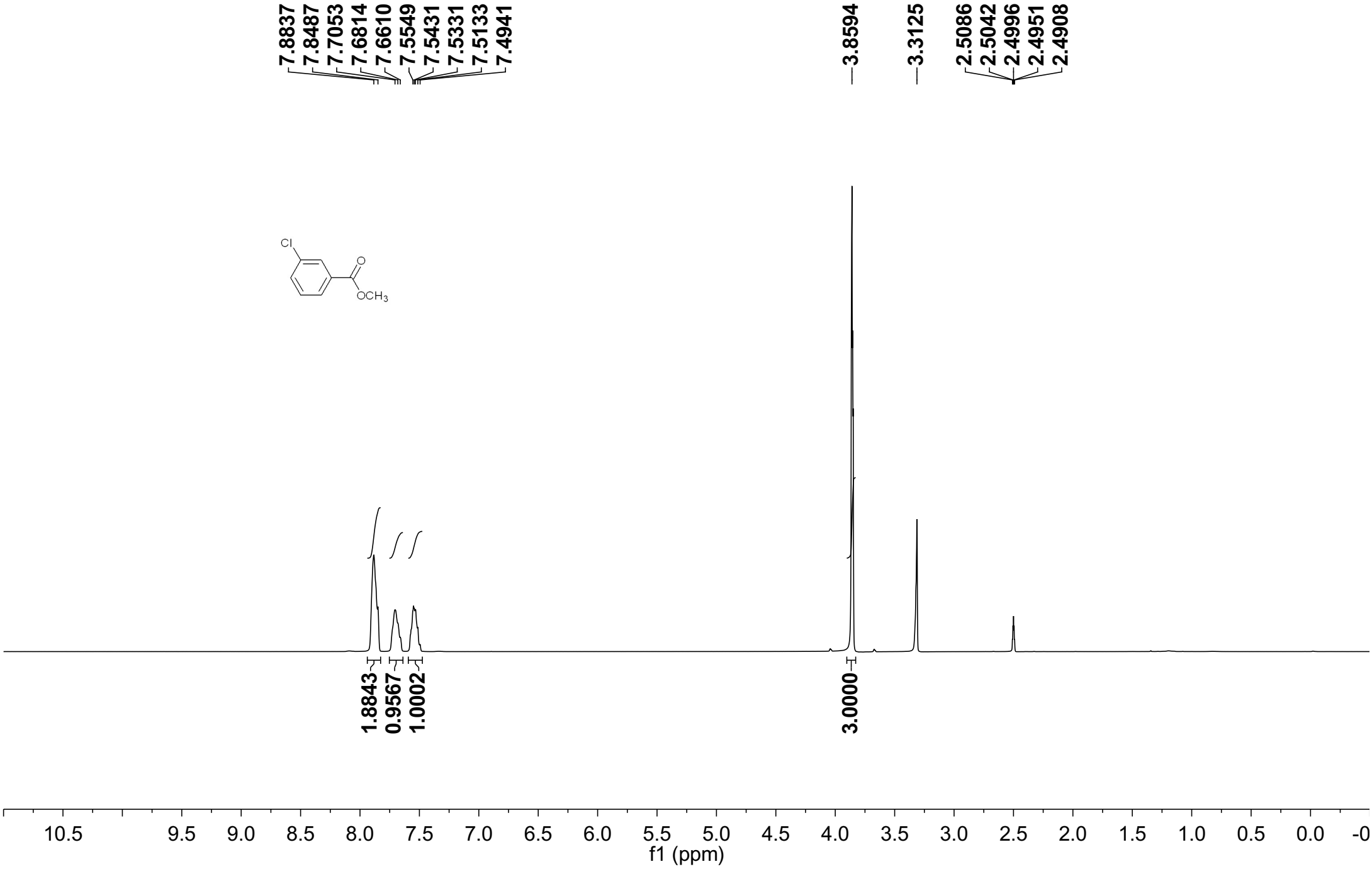
7.4539
7.4467
7.4370
7.4193
3.8553
3.3247
2.5090
2.5044
2.4998
2.4952
2.4907
Cl
O
OCH3
0.9782
2.9677
3.0000
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

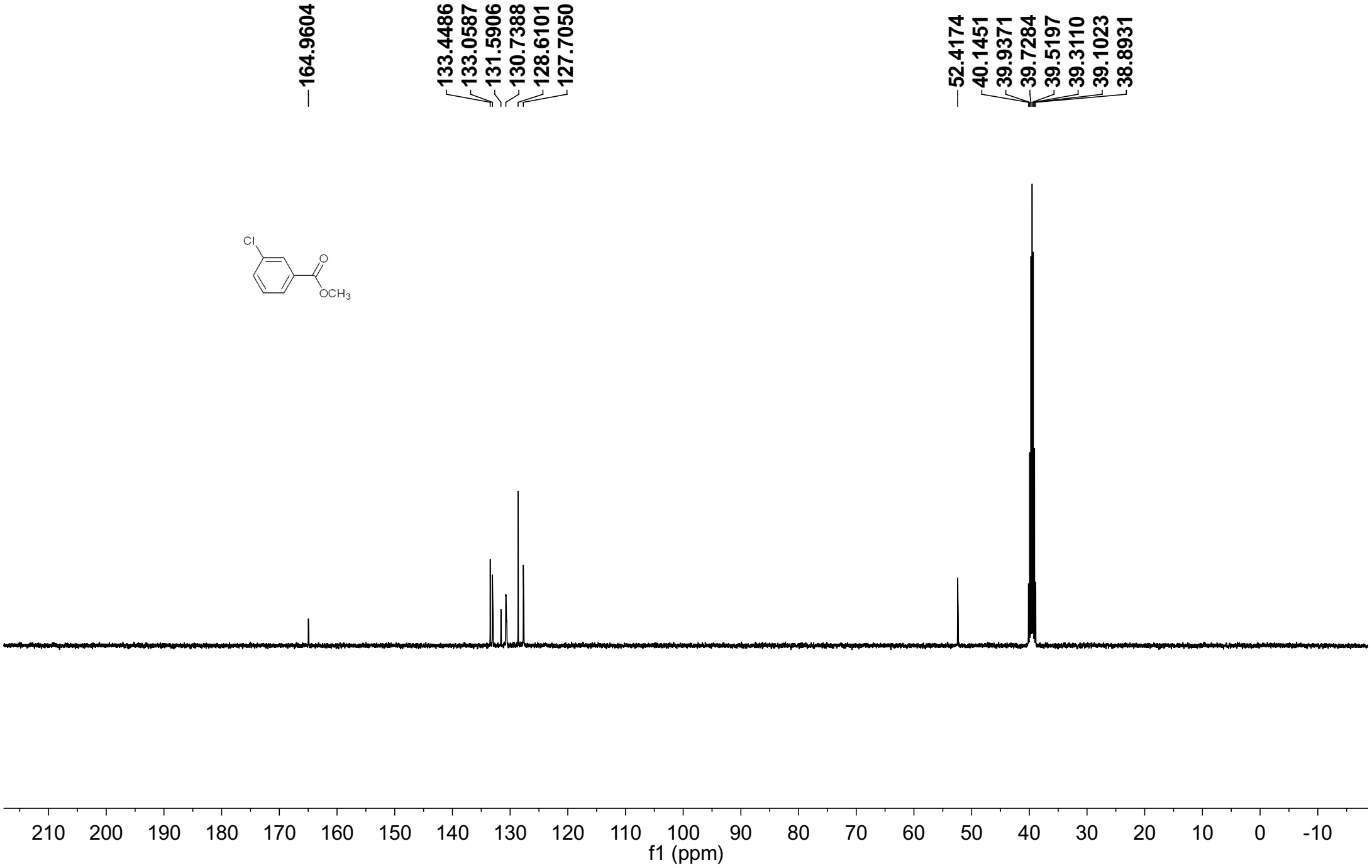
Compound 2d


165.4571
133.0559
131.7285
130.9138
130.6710
130.0446
127.2482
52.3919
40.1464
39.9378
39.7291
39.5204
39.3117
39.1030
38.8945
Cl
O
OCH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

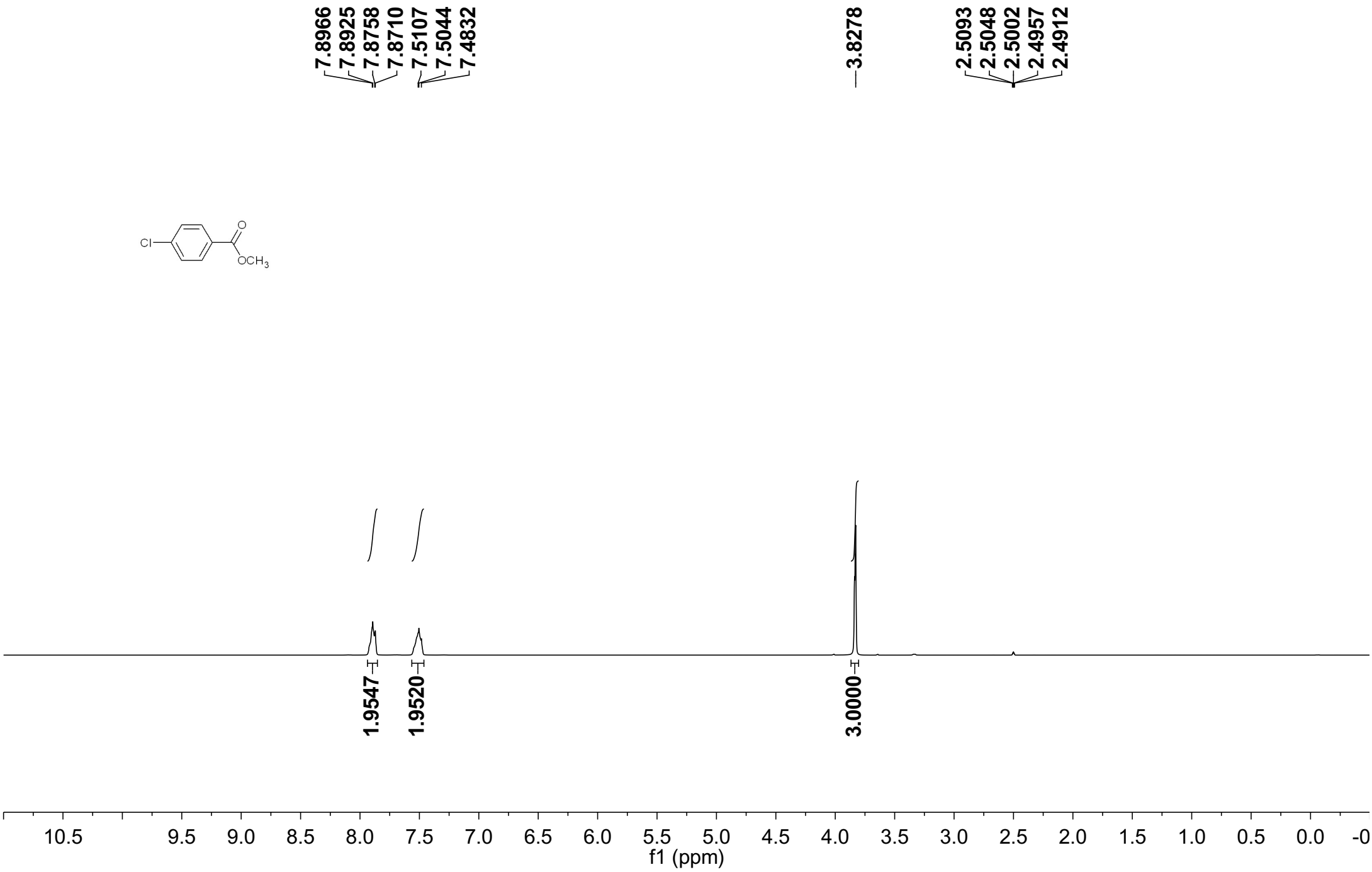
Compound 2e


7.8837
7.8487
7.7053
7.6814
7.6610
7.5549
7.5431
7.5331
7.5133
7.4941
3.8594
3.3125
2.5086
2.5042
2.4996
2.4951
2.4908
Cl
O
OCH3
1.8843
0.9567
1.0002
3.0000
10.5
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
f1 (ppm)

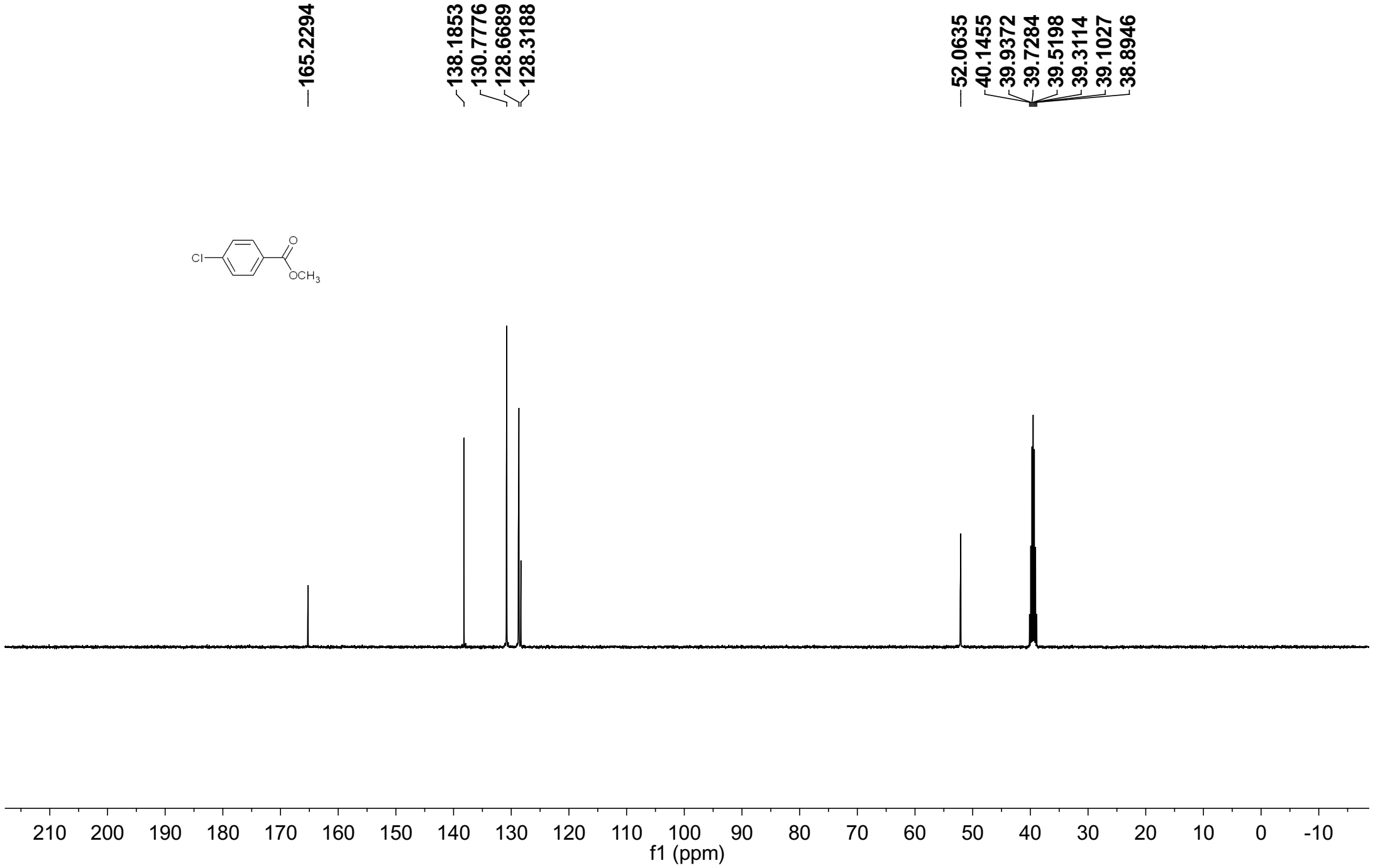
Compound 2e


164.9604
133.4486
133.0587
131.5906
130.7388
128.6101
127.7050
52.4174
40.1451
39.9371
39.7284
39.5197
39.3110
39.1023
38.8931
Cl
O
OCH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

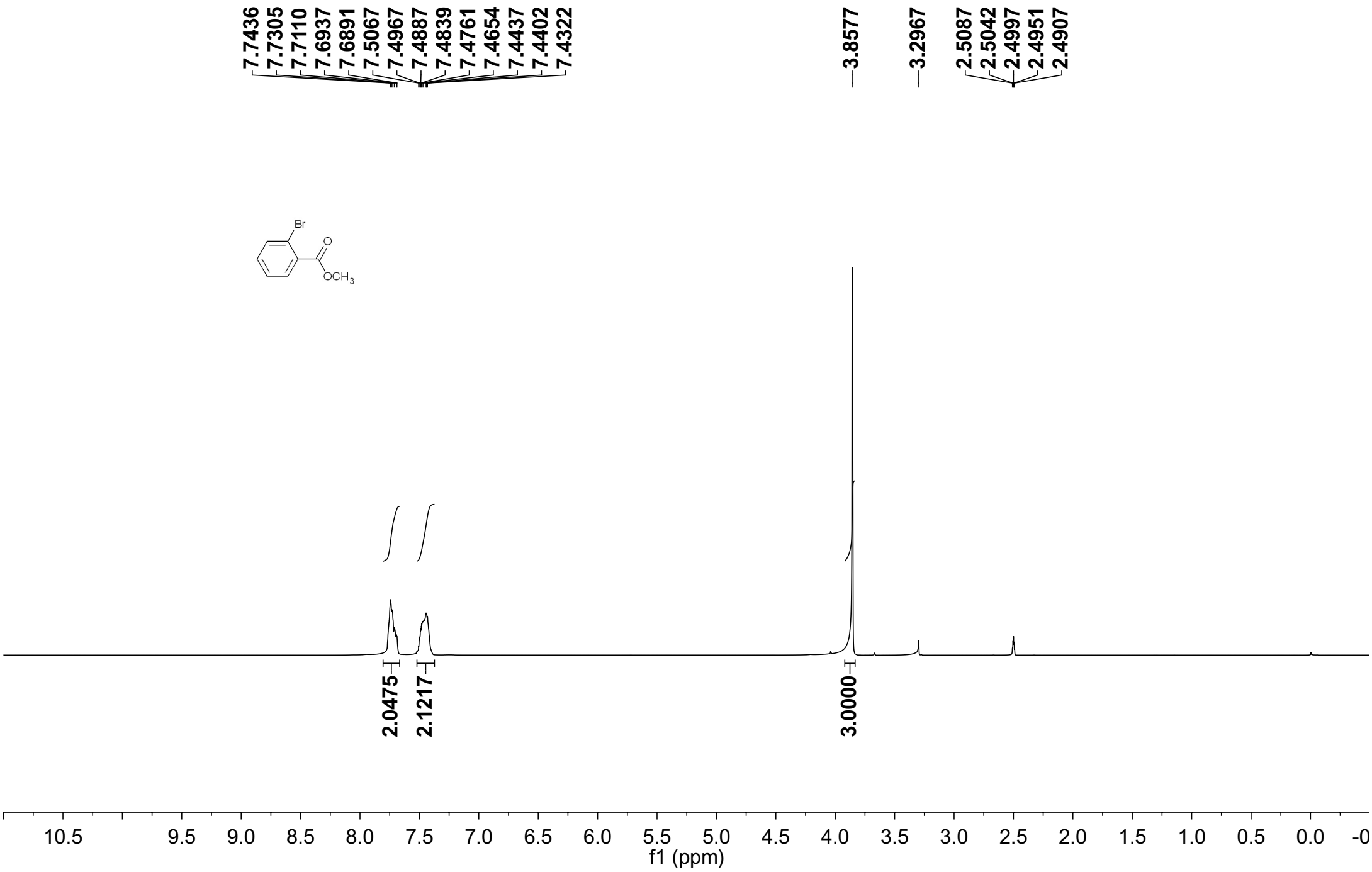
Compound 2f


7.8966
7.8925
7.8758
7.8710
7.5107
7.5044
7.4832
3.8278
2.5093
2.5048
2.5002
2.4957
2.4912
Cl
O
OCH3
1.9547
1.9520
3.0000
10.5
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
f1 (ppm)

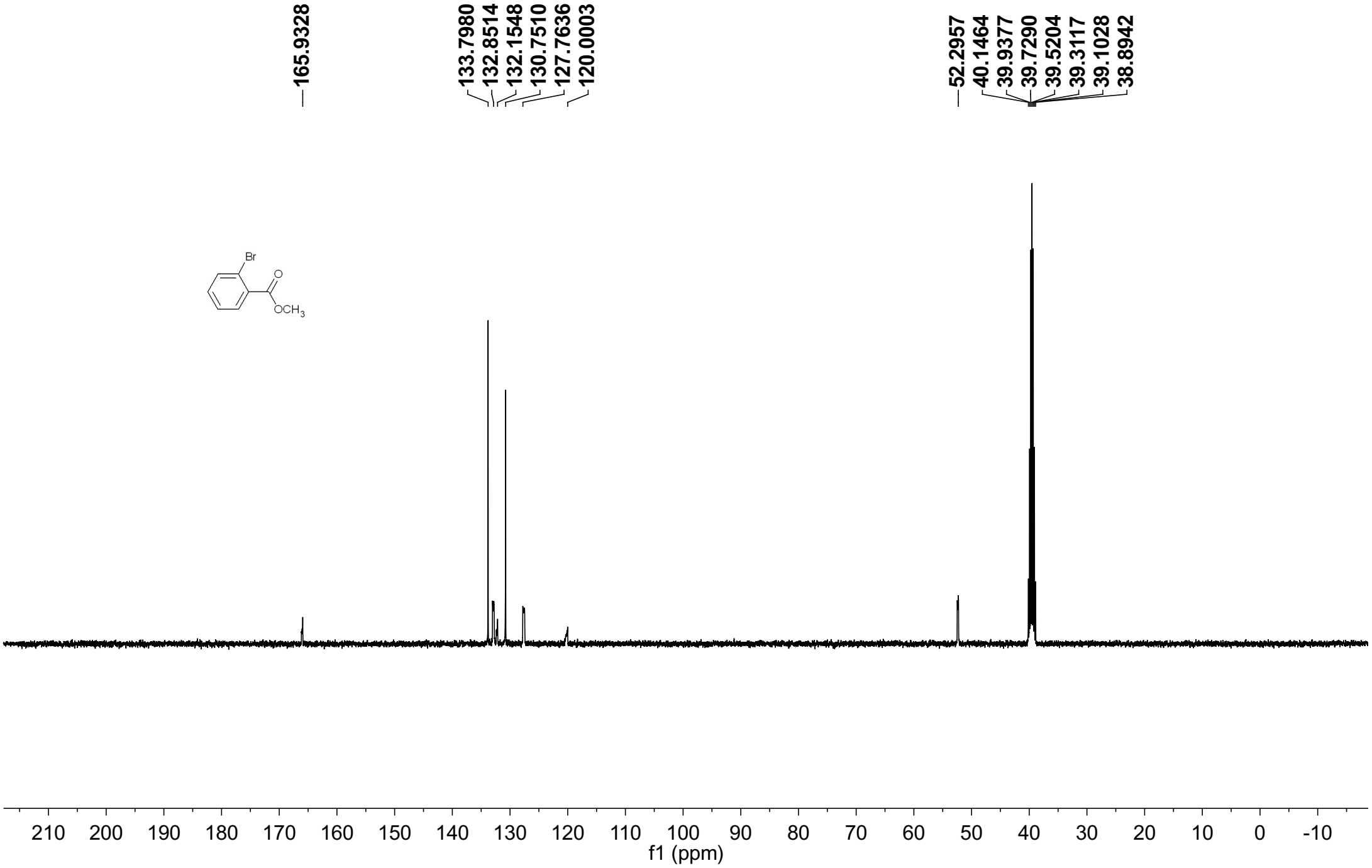
Compound 2f


165.2294
138.1853
130.7776
128.6689
128.3188
52.0635
40.1455
39.9372
39.7284
39.5198
39.3114
39.1027
38.8946
Cl
O
OCH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

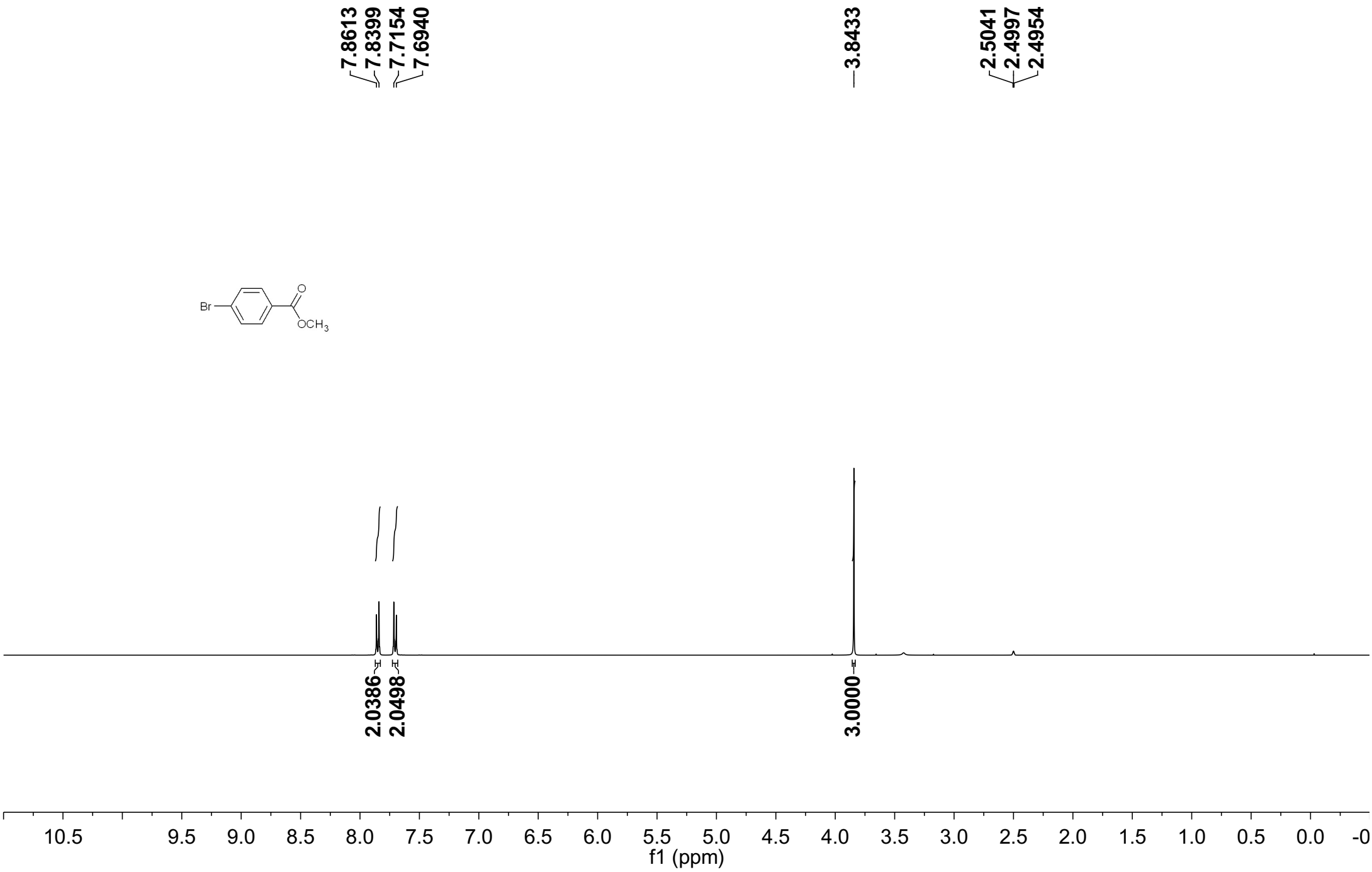
Compound 2g


7.7436
7.7305
7.7110
7.6937
7.6891
7.5067
7.4967
7.4887
7.4839
7.4761
7.4654
7.4437
7.4402
7.4322
3.8577
3.2967
2.5087
2.5042
2.4997
2.4951
2.4907
Br
O
OCH3
2.0475
2.1217
3.0000
10.5
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
f1 (ppm)

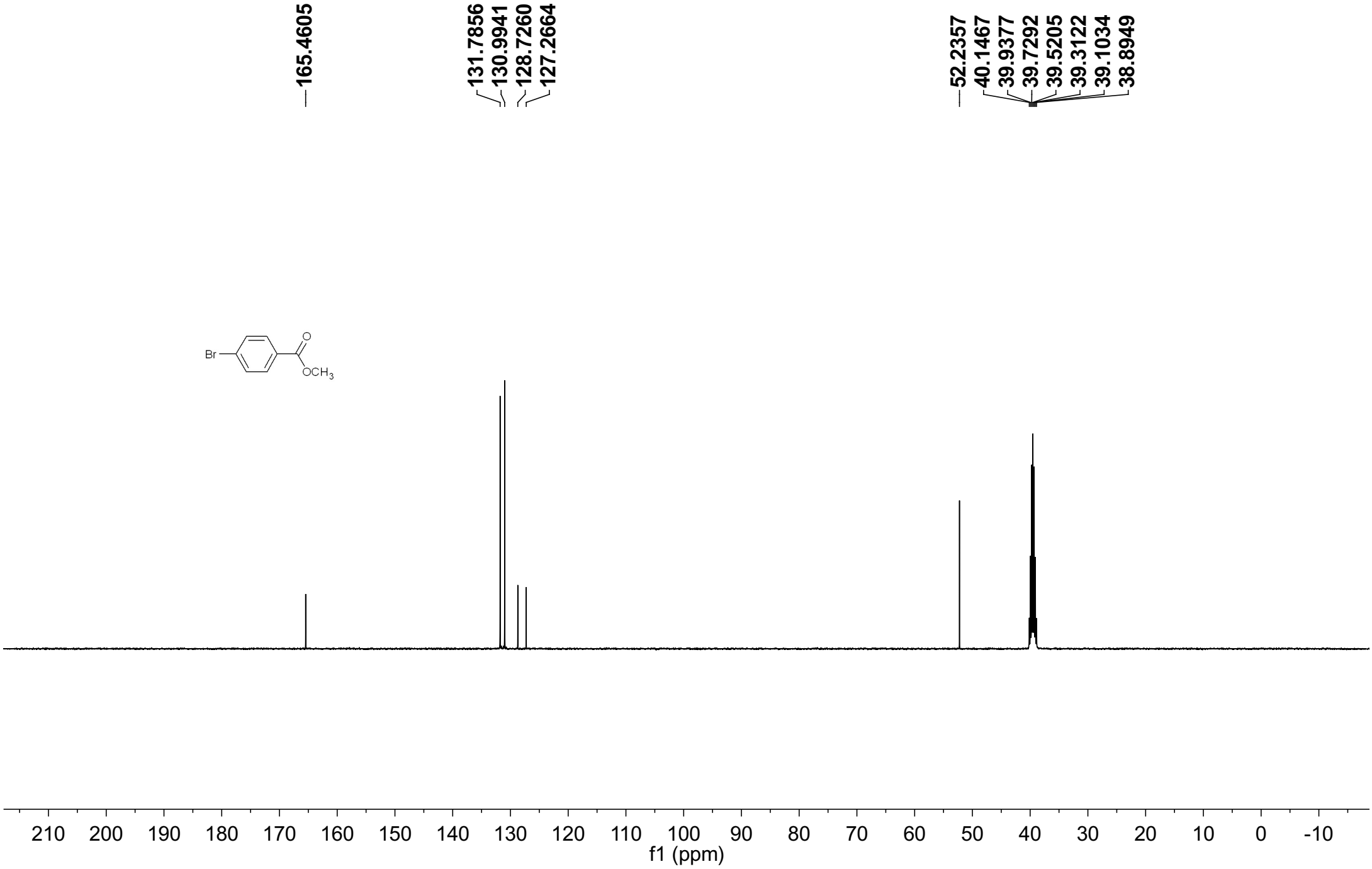
Compound 2g


165.9328
133.7980
132.8514
132.1548
130.7510
127.7636
120.0003
52.2957
40.1464
39.9377
39.7290
39.5204
39.3117
39.1028
38.8942
Br
O
OCH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

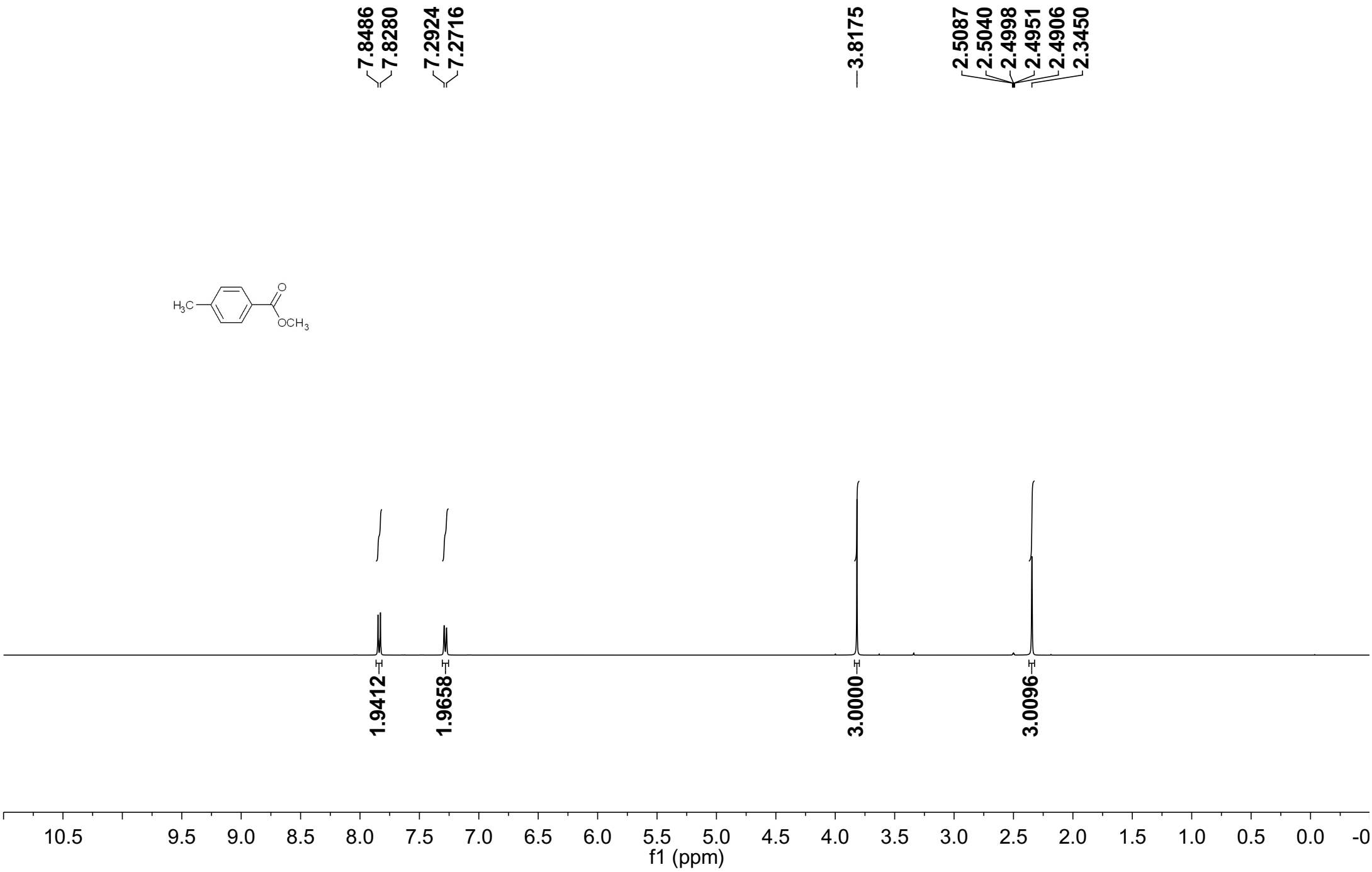
Compound 2h


7.8613
7.8399
7.7154
7.6940
3.8433
2.5041
2.4997
2.4954
Br
O
OCH3
2.0386
2.0498
3.0000
10.5
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
f1 (ppm)

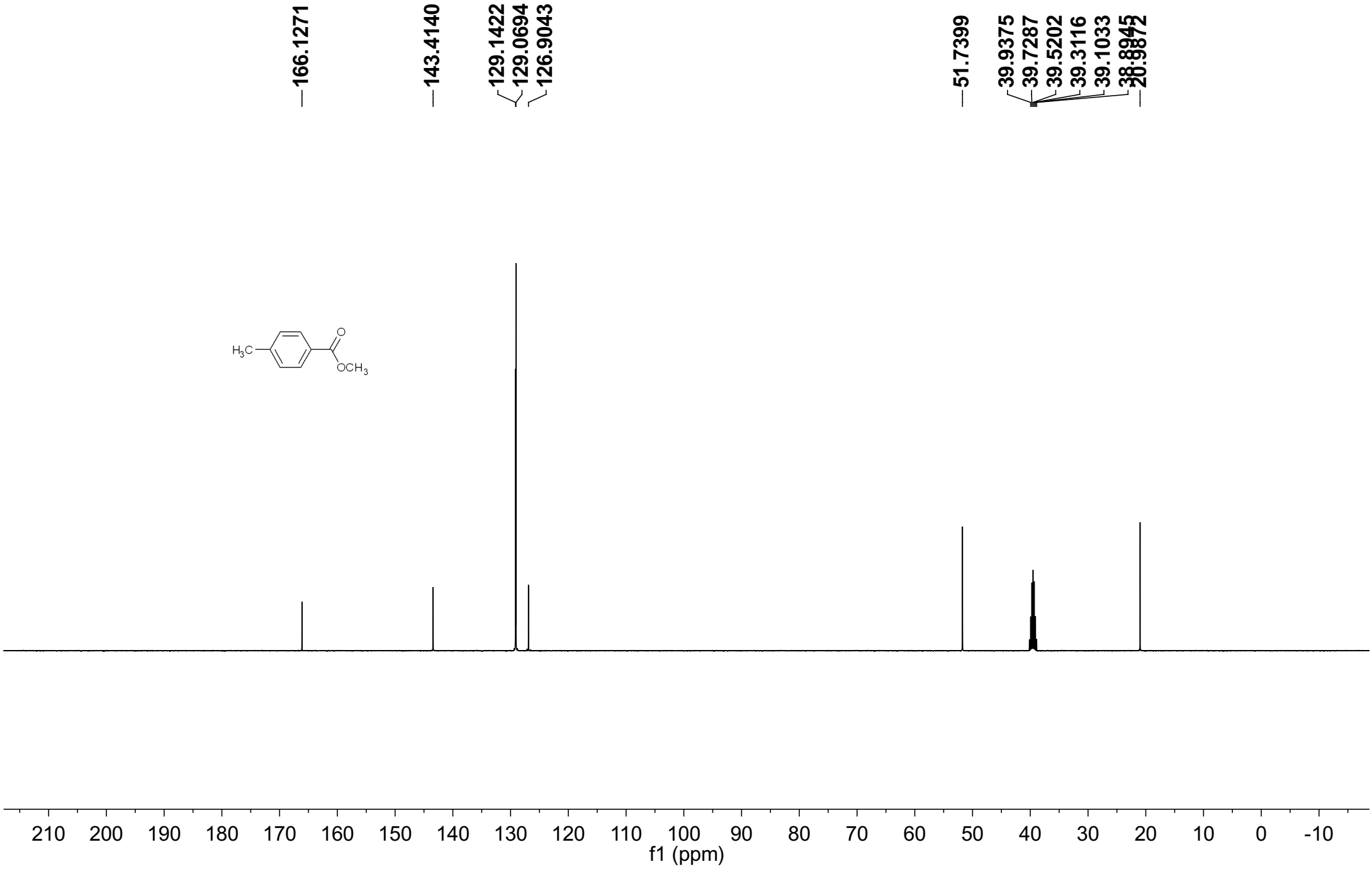
Compound 2h


165.4605
131.7856
130.9941
128.7260
127.2664
52.2357
40.1467
39.9377
39.7292
39.5205
39.3122
39.1034
38.8949
Br
O
OCH3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

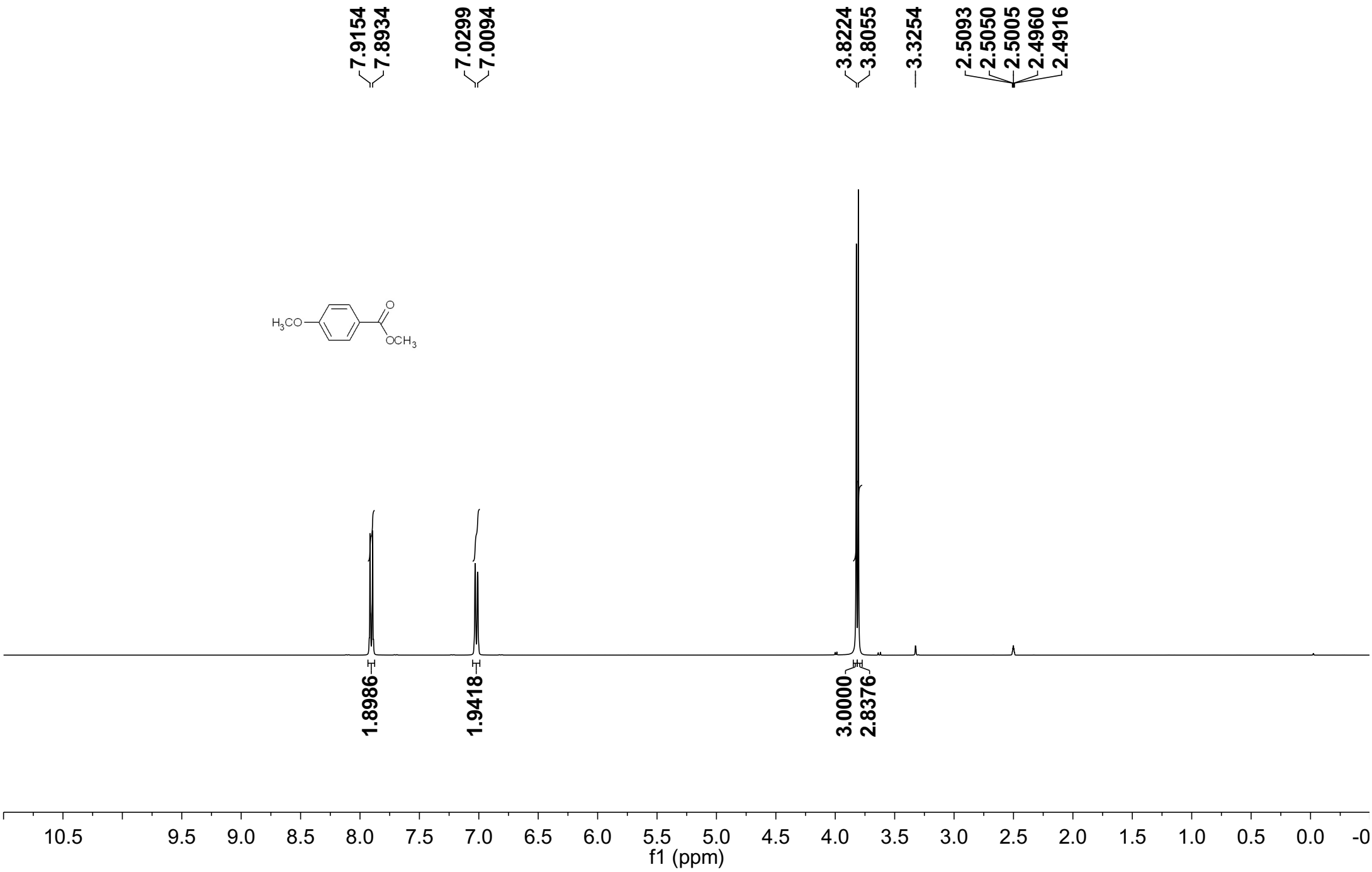
Compound 2i


7.8486
7.8280
7.2924
7.2716
3.8175
2.5087
2.5040
2.4998
2.4951
2.4906
2.3450
H3C
O
OCH3
1.9412
1.9658
3.0000
3.0096
10.5
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
f1 (ppm)

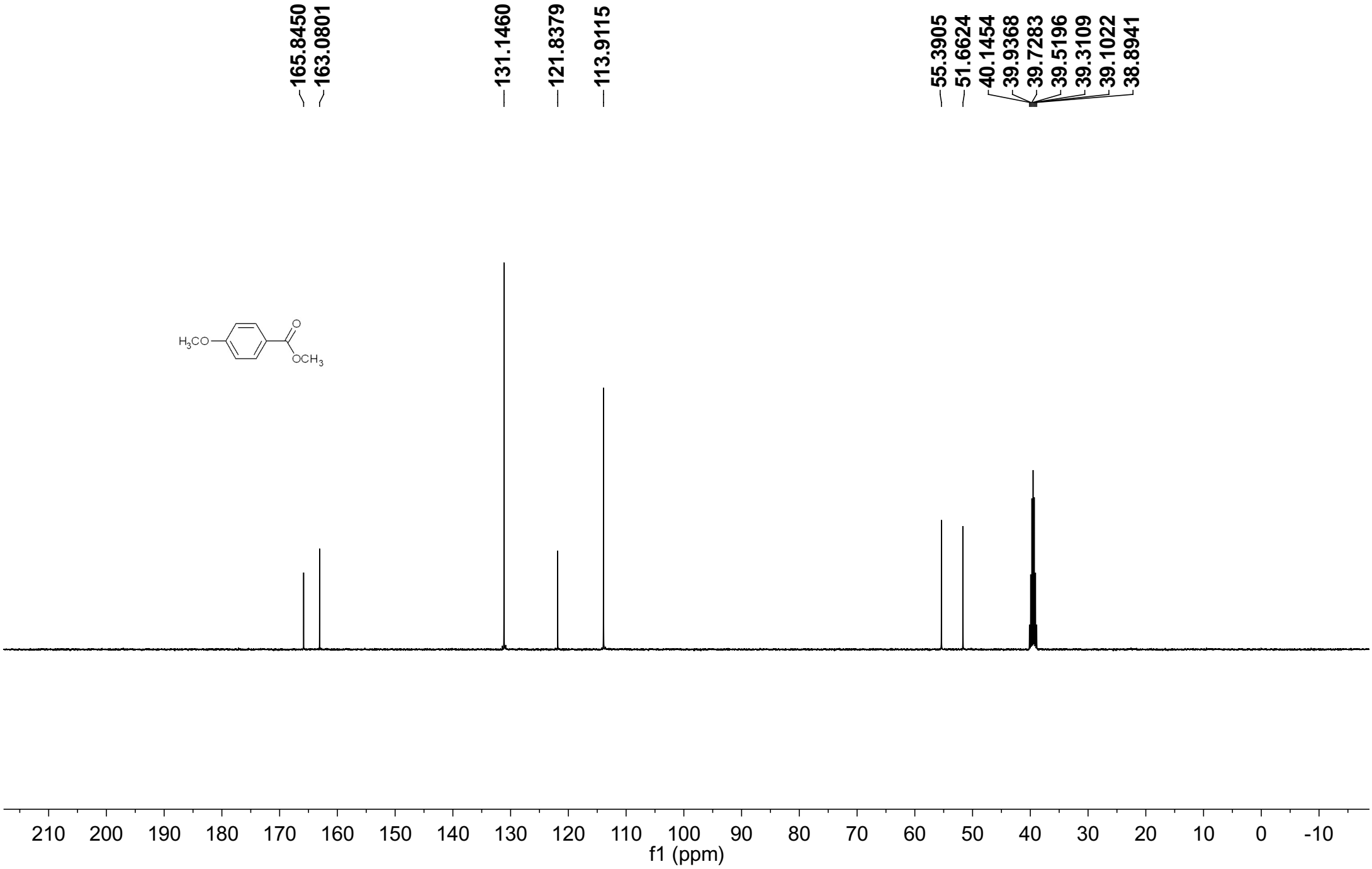
Compound 2i


166.1271
143.4140
129.1422
129.0694
126.9043
51.7399
39.9375
39.7287
39.5202
39.3116
39.1033
38.8945
20.9872
H3C
O
OCH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

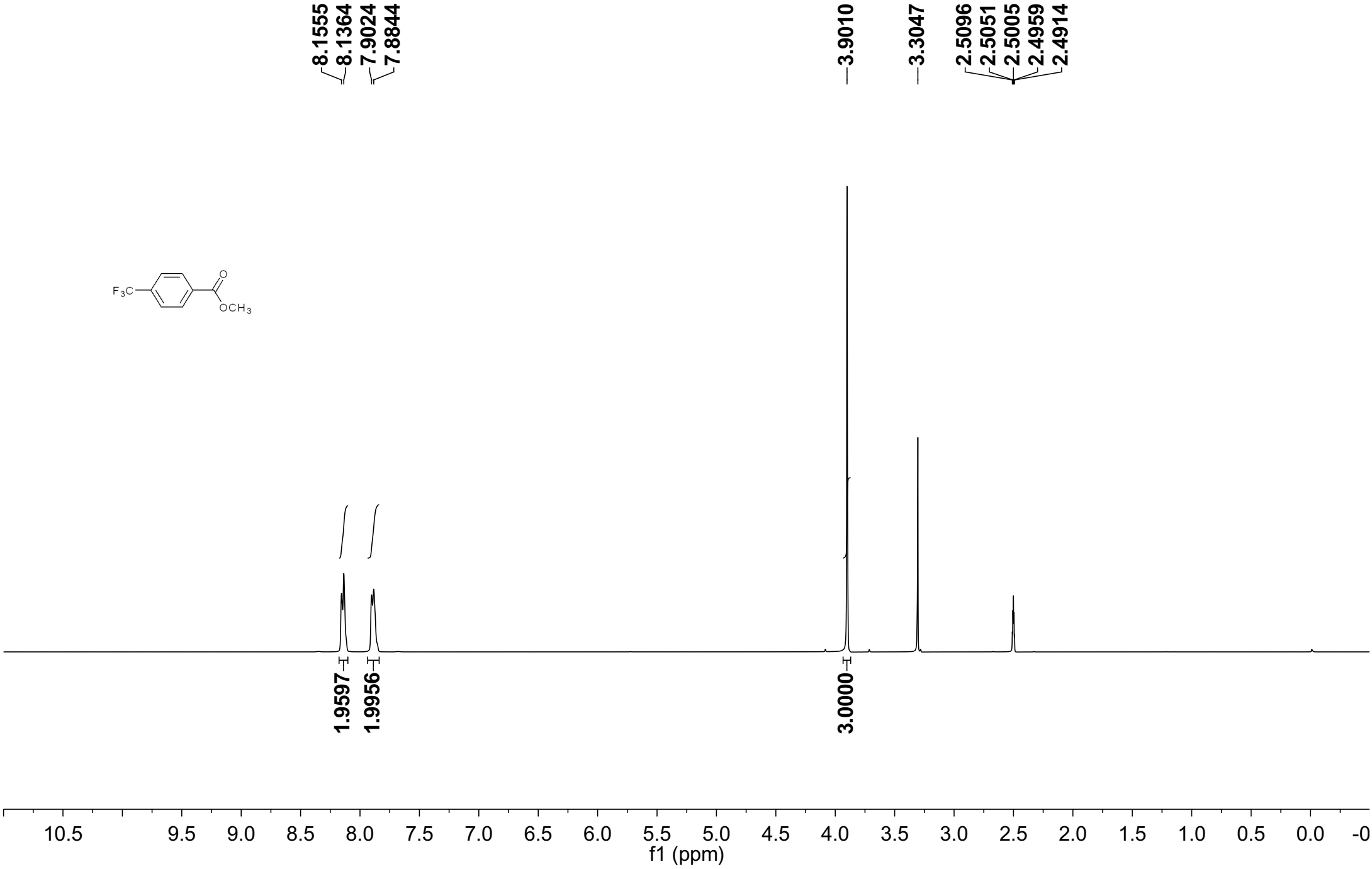
Compound 2j


7.9154
7.8934
7.0299
7.0094
3.8224
3.8055
3.3254
2.5093
2.5050
2.5005
2.4960
2.4916
H3CO
O
OCH3
1.8986
1.9418
3.0000
2.8376
10.5
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
f1 (ppm)

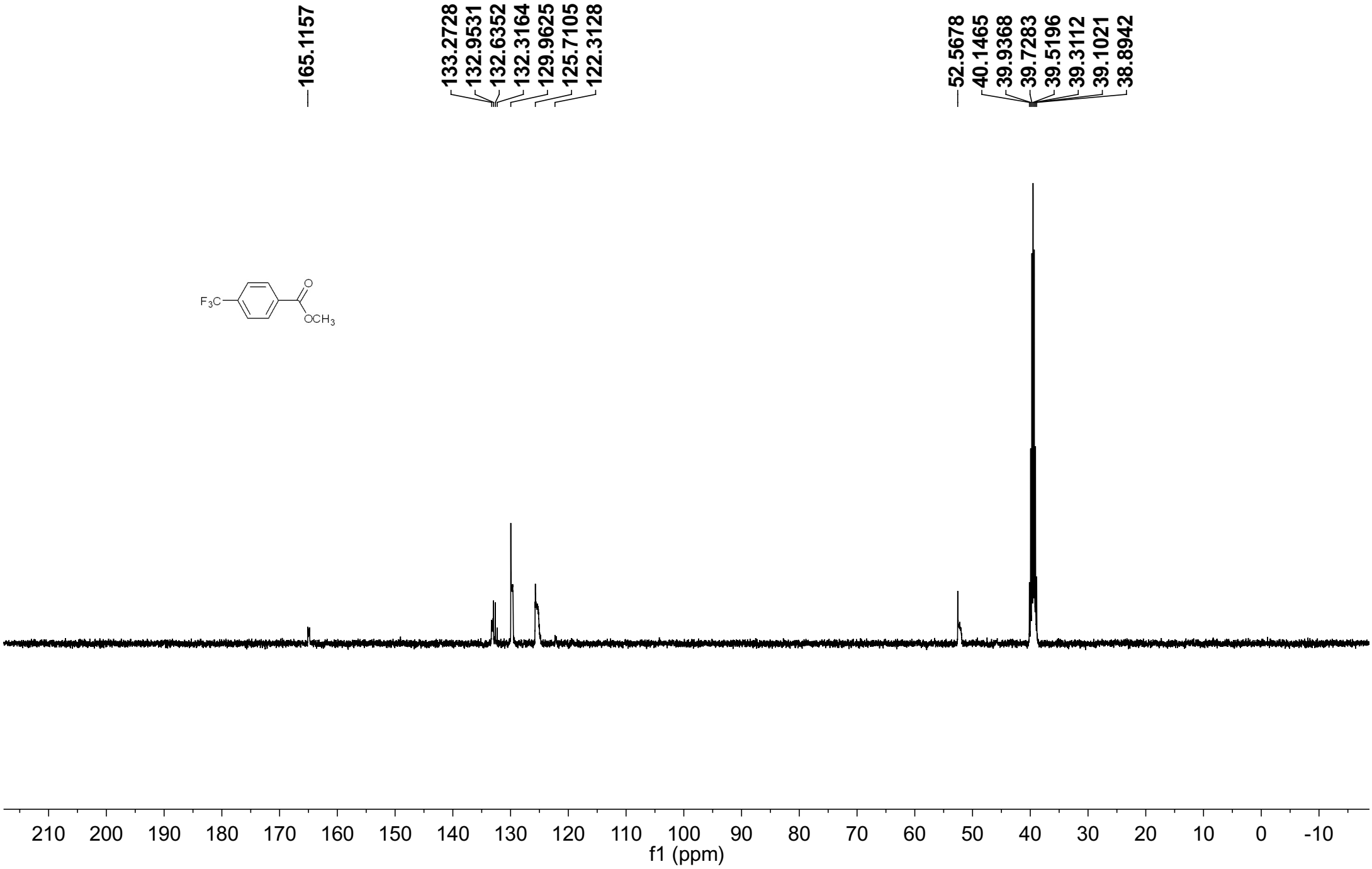
Compound 2j


165.8450
163.0801
131.1460
121.8379
113.9115
55.3905
51.6624
40.1454
39.9368
39.7283
39.5196
39.3109
39.1022
38.8941
H3CO
OCH3
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

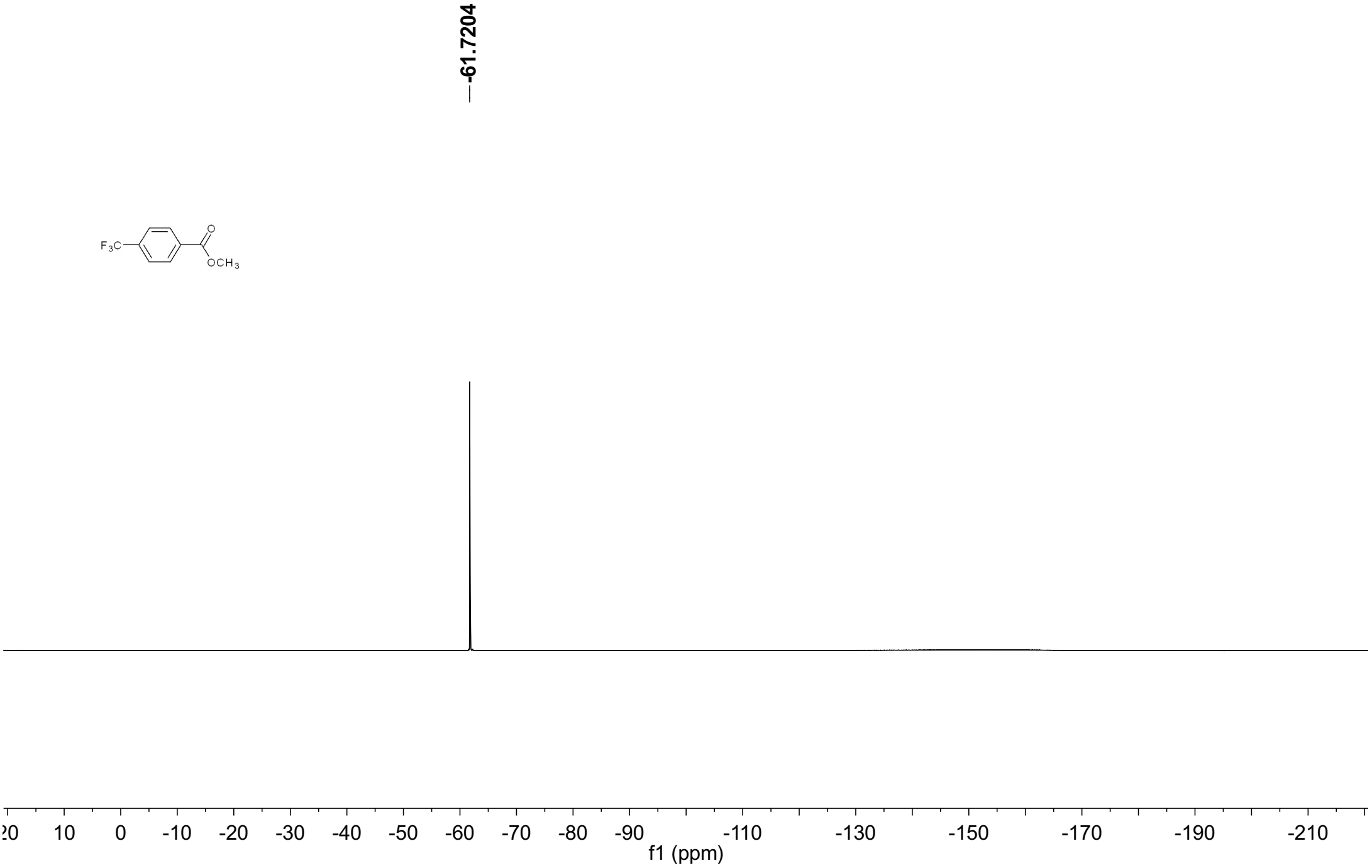
Compound 2k


8.1555
8.1364
7.9024
7.8844
3.9010
3.3047
2.5096
2.5051
2.5005
2.4959
2.4914
F3C
O
OCH3
1.9597
1.9956
3.0000
10.5
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
f1 (ppm)

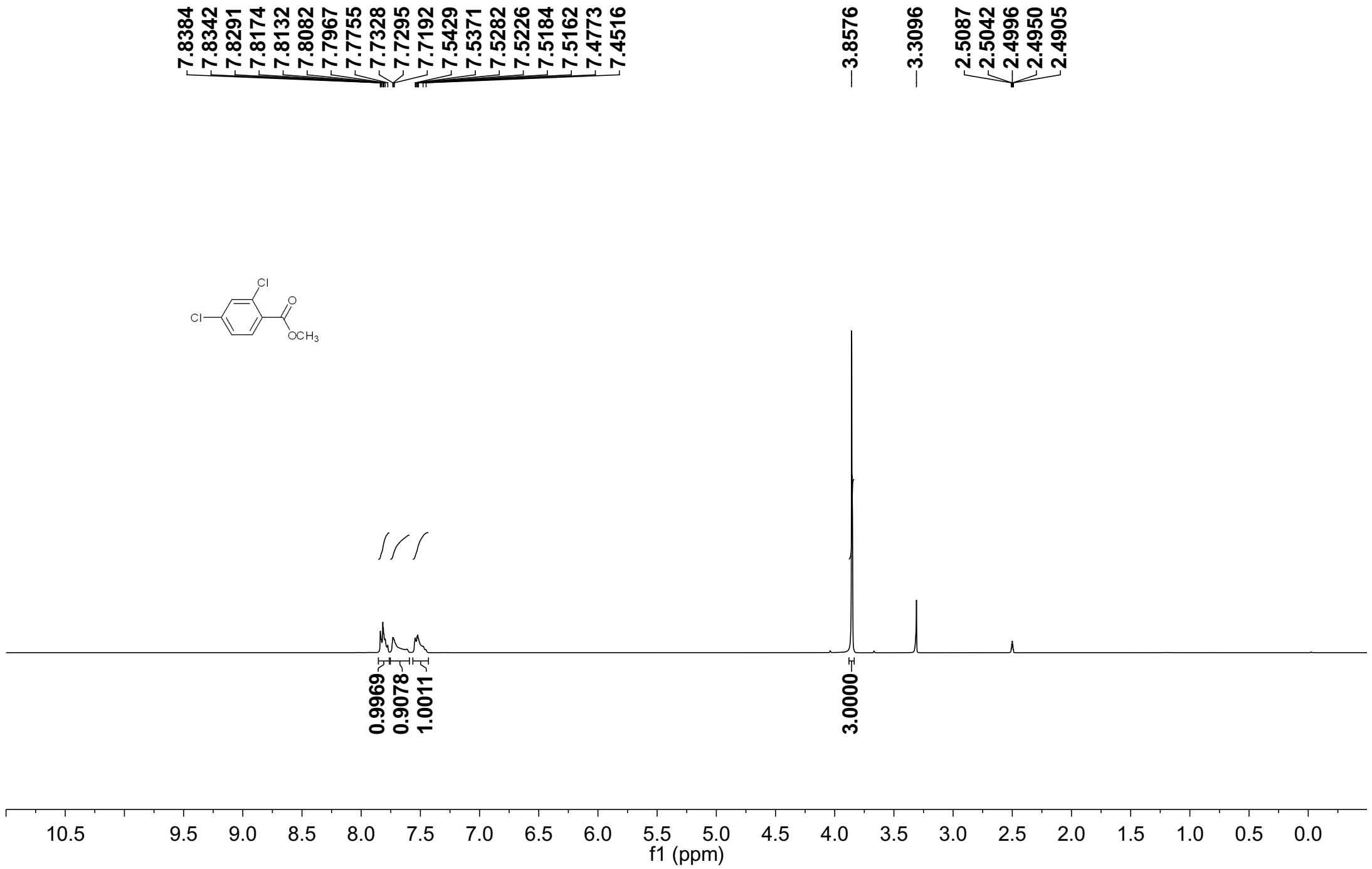
Compound 2k


165.1157
133.2728
132.9531
132.6352
132.3164
129.9625
125.7105
122.3128
52.5678
40.1465
39.9368
39.7283
39.5196
39.3112
39.1021
38.8942
F3C
O
OCH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

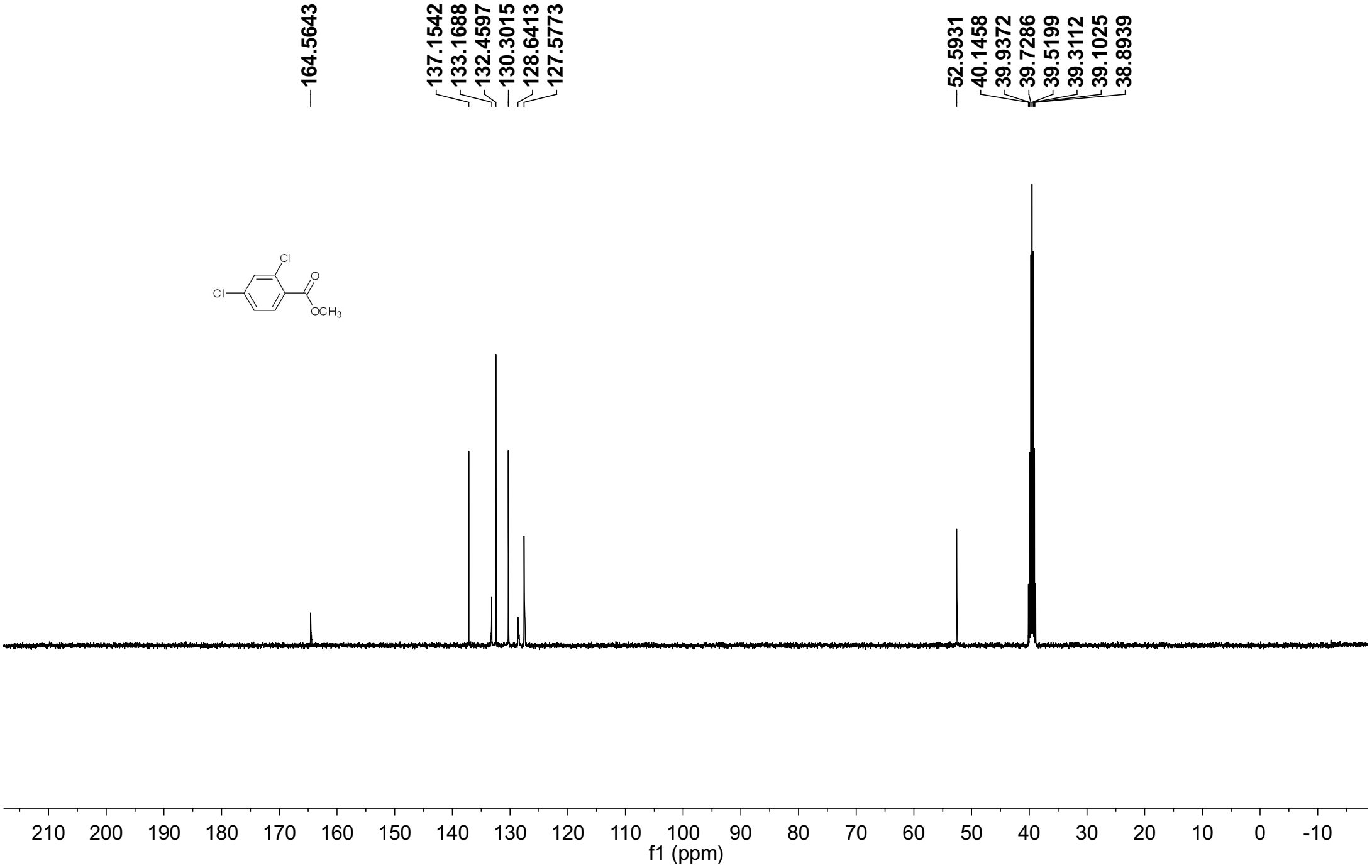
Compound 2k


-61.7204
F3C
O
OCH3
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
-210
f1 (ppm)

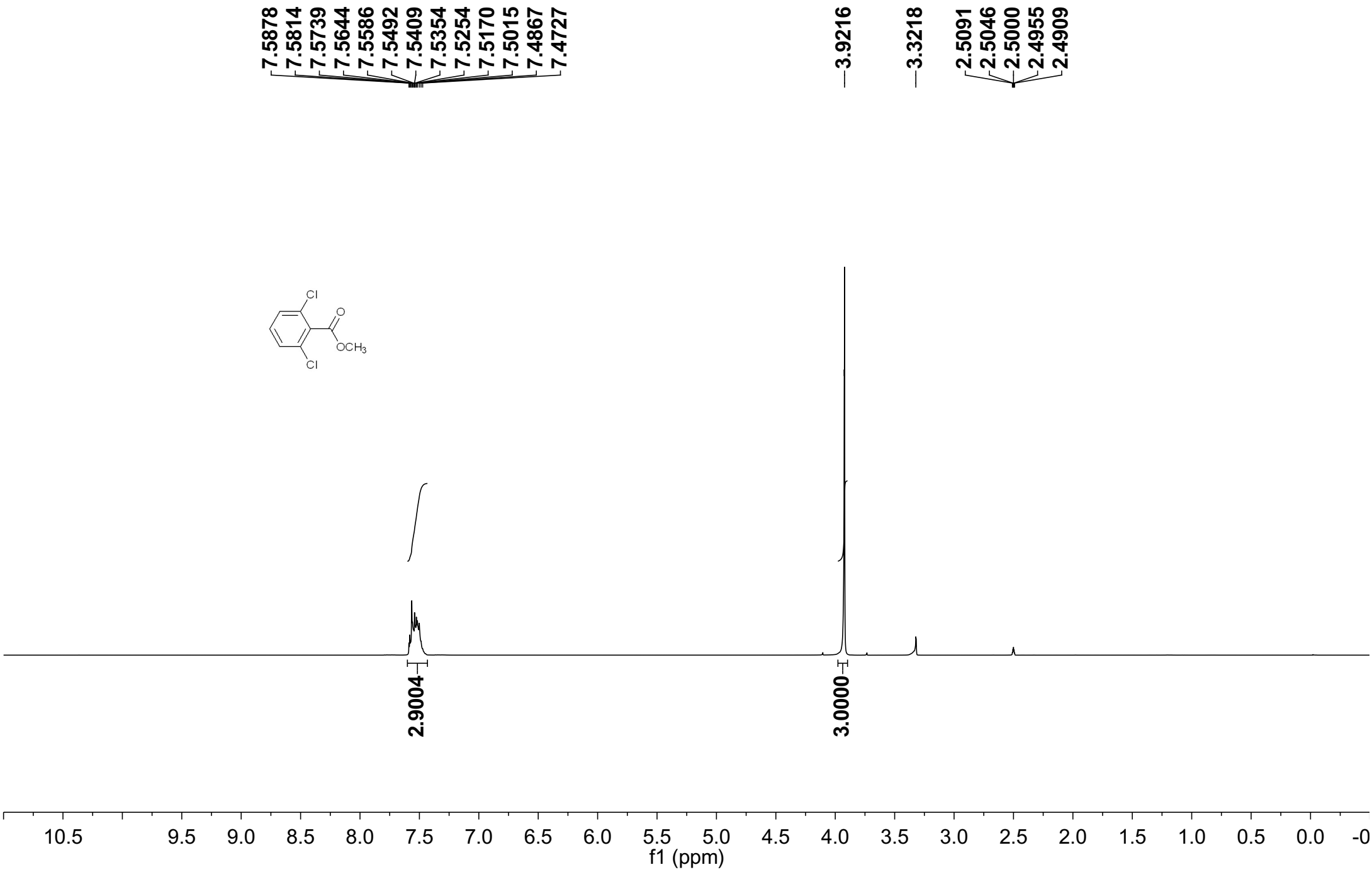
Compound 2l


7.8384
7.8342
7.8291
7.8174
7.8132
7.8082
7.7967
7.7755
7.7328
7.7295
7.7192
7.5429
7.5371
7.5282
7.5226
7.5184
7.5162
7.4773
7.4516
3.8576
3.3096
2.5087
2.5042
2.4996
2.4950
2.4905
Cl
Cl
O
OCH3
0.9969
0.9078
1.0011
3.0000
10.5
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)

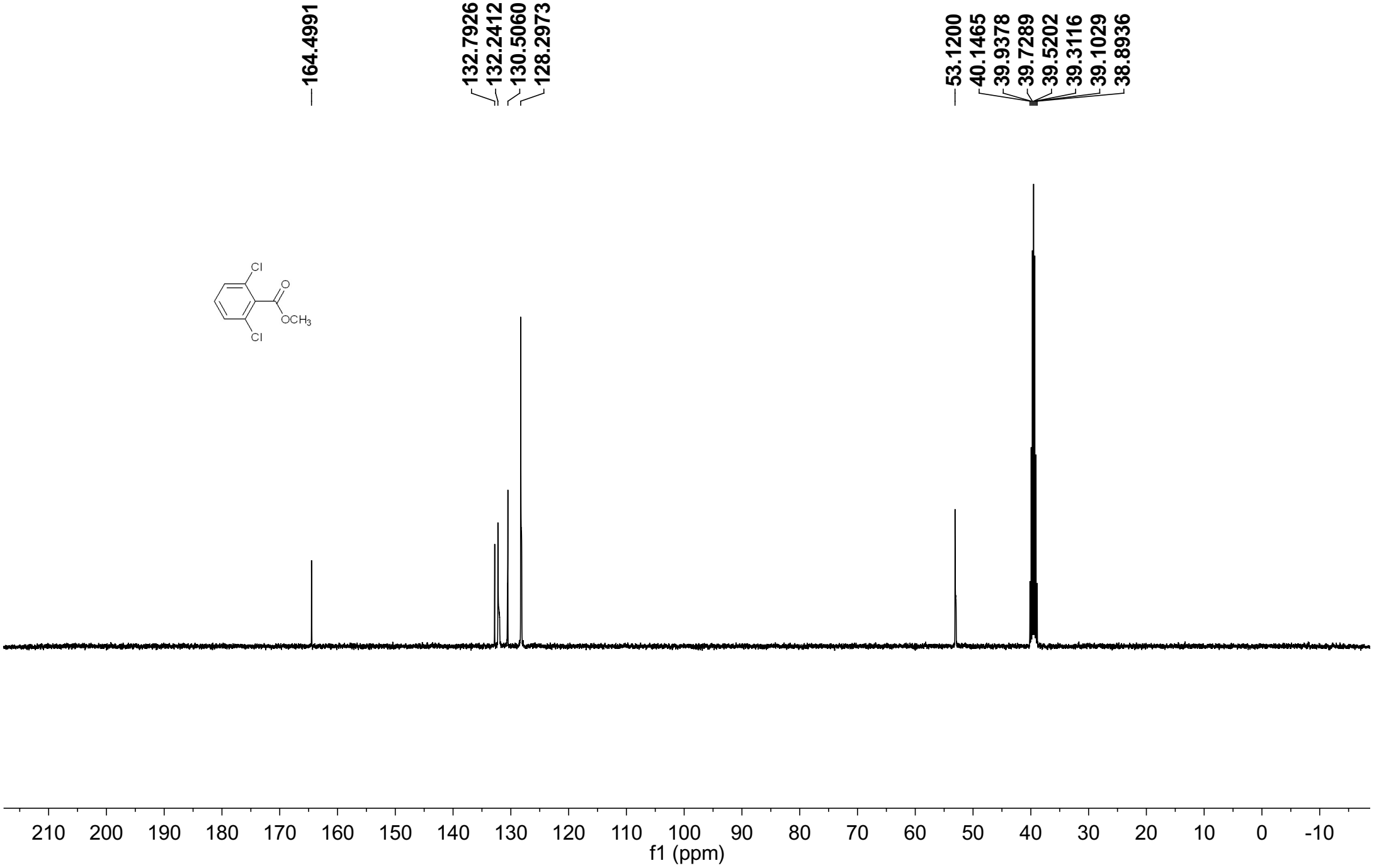
Compound 2l


164.5643
137.1542
133.1688
132.4597
130.3015
128.6413
127.5773
52.5931
40.1458
39.9372
39.7286
39.5199
39.3112
39.1025
38.8939
Cl
Cl
O
OCH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

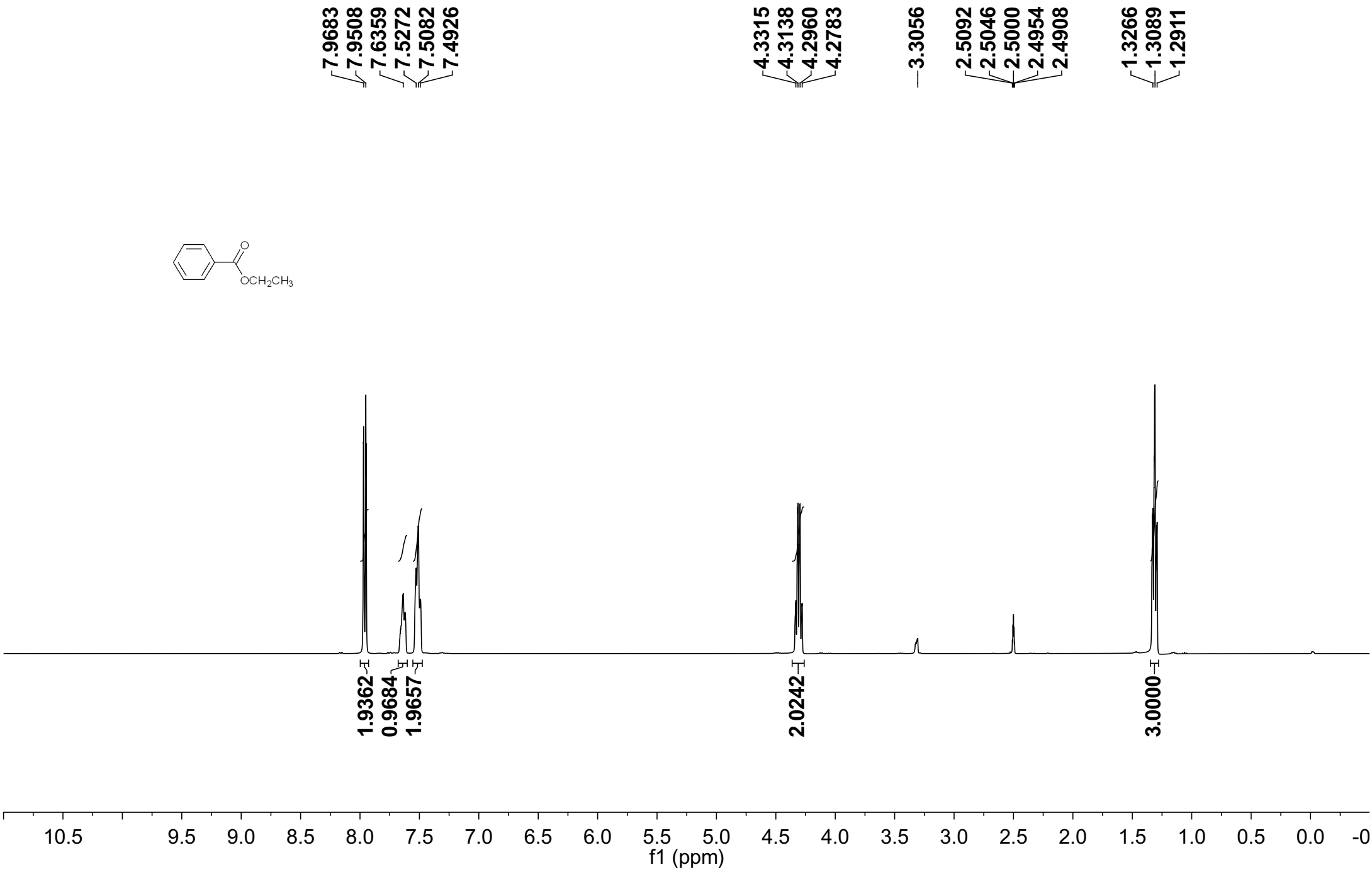
Compound 2m


7.5878
7.5814
7.5739
7.5644
7.5586
7.5492
7.5409
7.5354
7.5254
7.5170
7.5015
7.4867
7.4727
3.9216
3.3218
2.5091
2.5046
2.5000
2.4955
2.4909
Cl
O
OCH3
Cl
2.9004
3.0000
10.5
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
f1 (ppm)

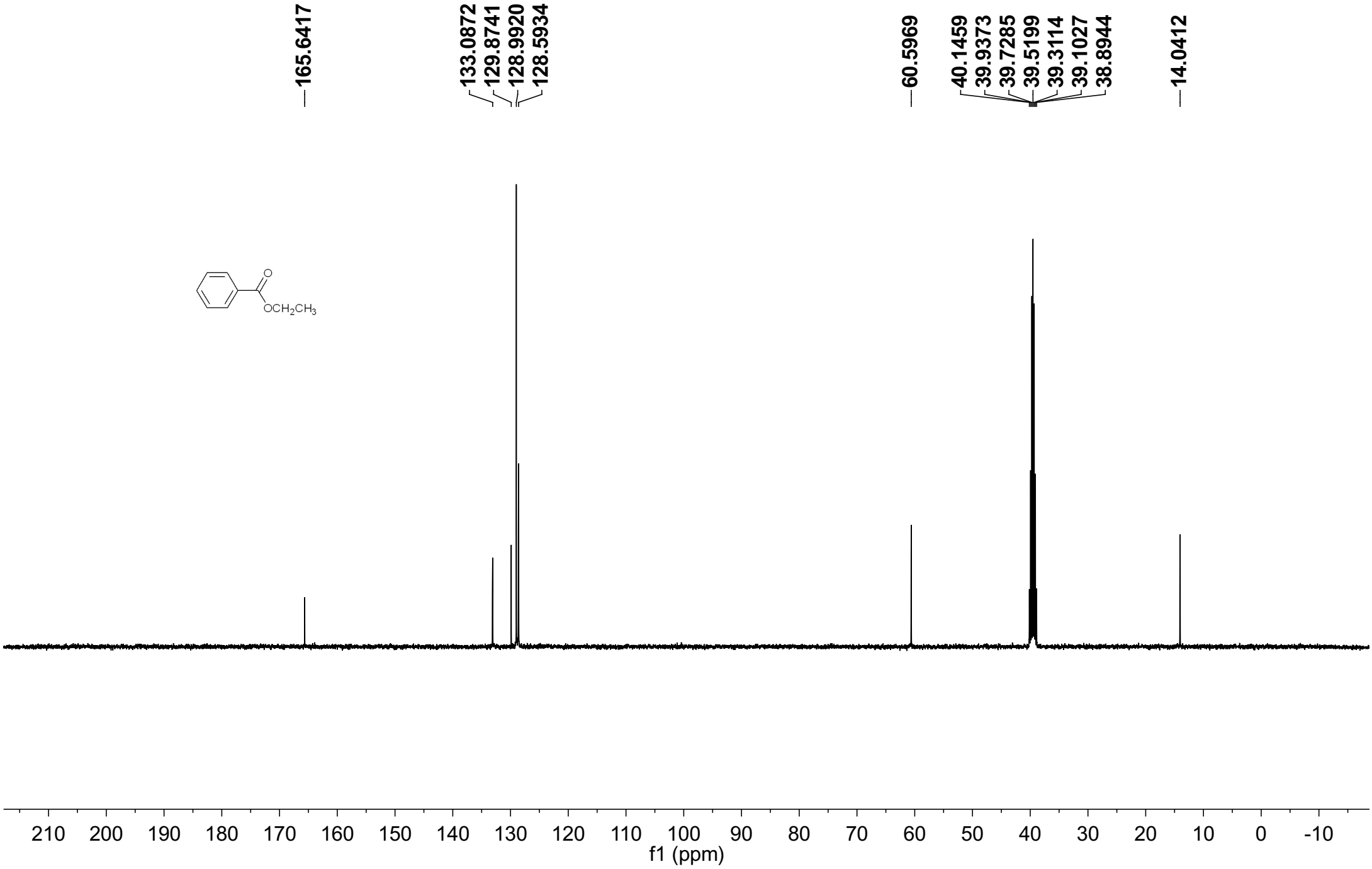
Compound 2m


164.4991
132.7926
132.2412
130.5060
128.2973
53.1200
40.1465
39.9378
39.7289
39.5202
39.3116
39.1029
38.8936
Cl
O
OCH3
Cl
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

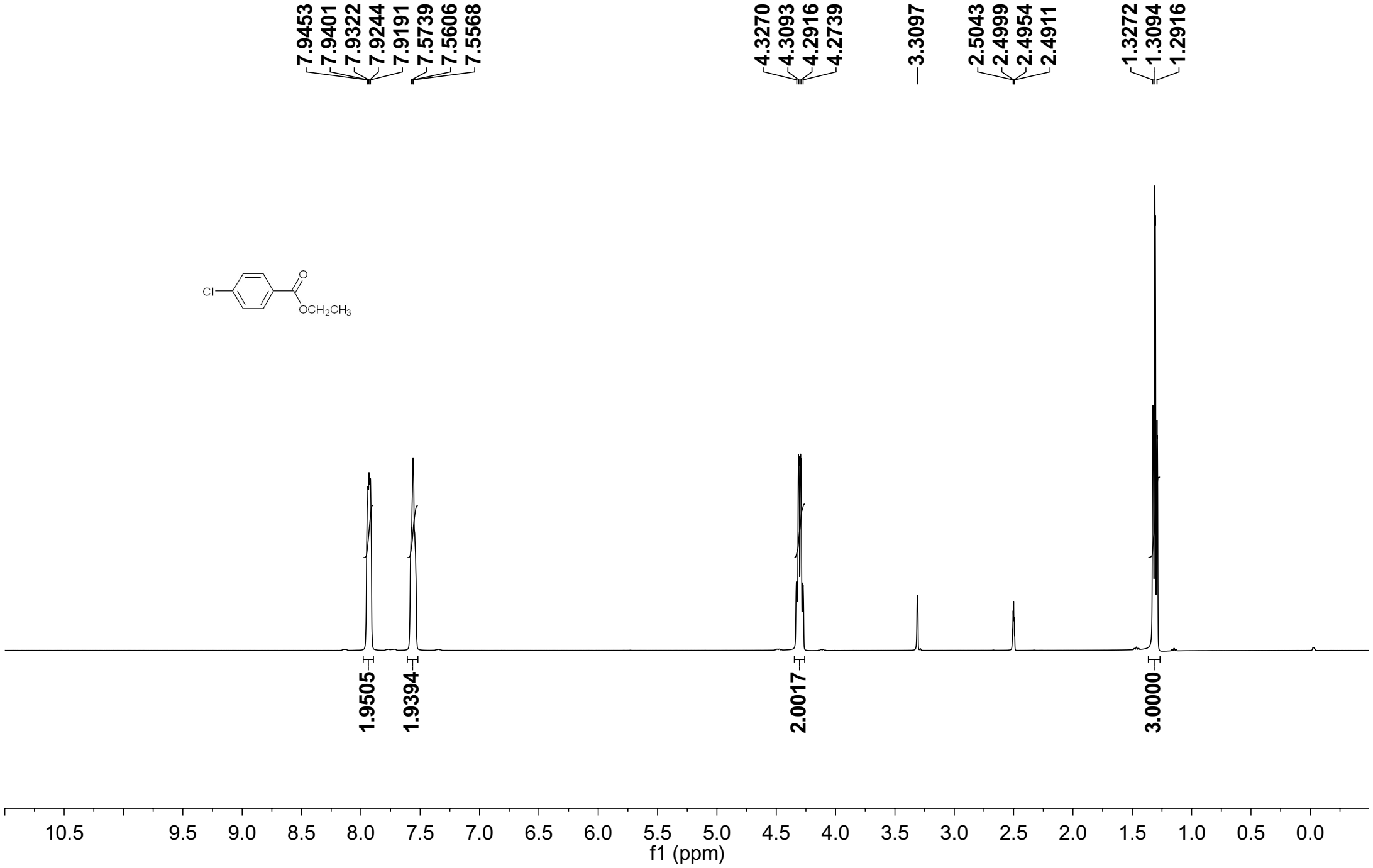
Compound 2n


7.9683
7.9508
7.6359
7.5272
7.5082
7.4926
4.3315
4.3138
4.2960
4.2783
3.3056
2.5092
2.5046
2.5000
2.4954
2.4908
1.3266
1.3089
1.2911
OCH2CH3
1.9362
0.9684
1.9657
2.0242
3.0000
10.5
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
f1 (ppm)

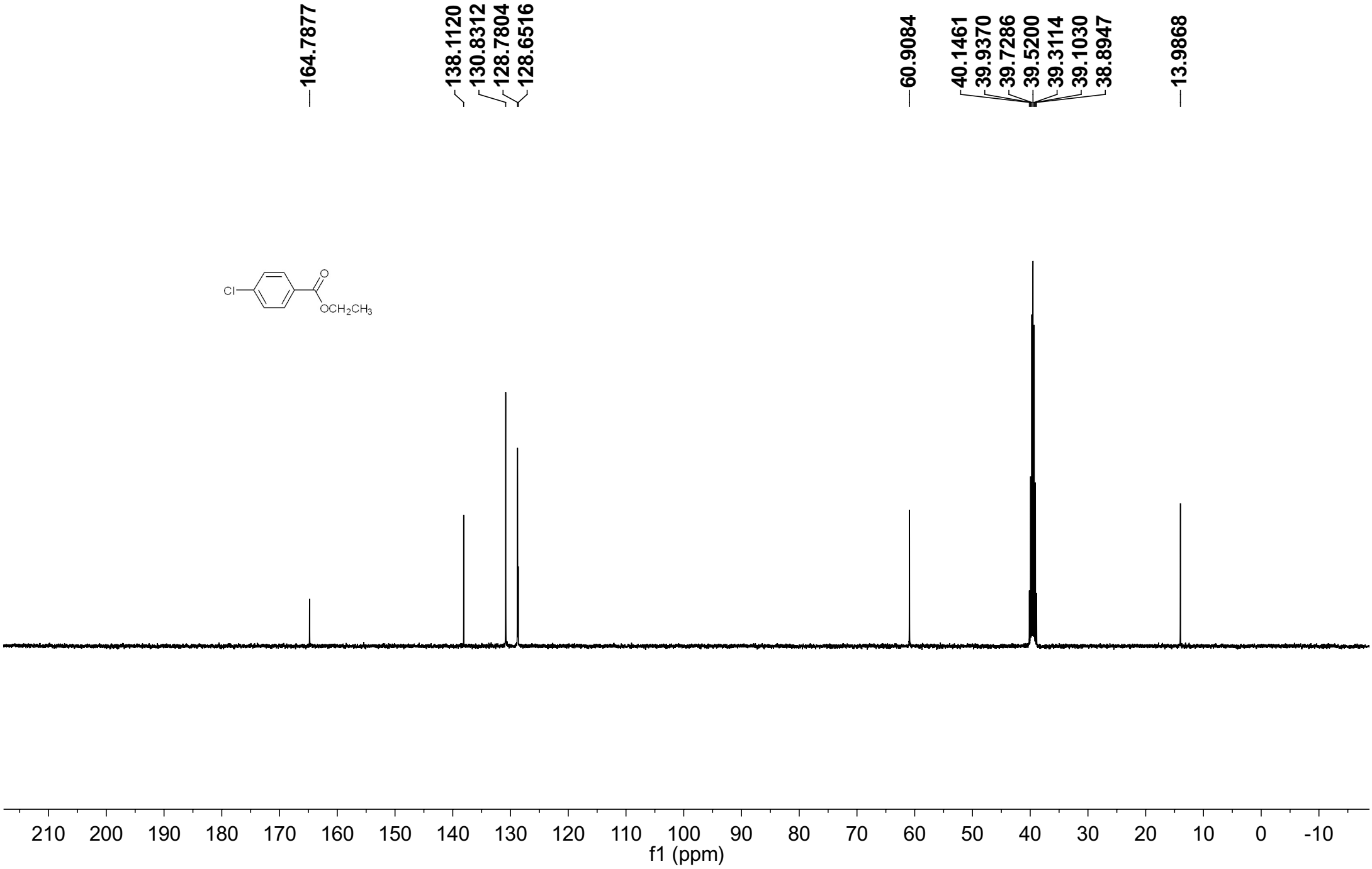
Compound 2n


165.6417
133.0872
129.8741
128.9920
128.5934
60.5969
40.1459
39.9373
39.7285
39.5199
39.3114
39.1027
38.8944
14.0412
OCH2CH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

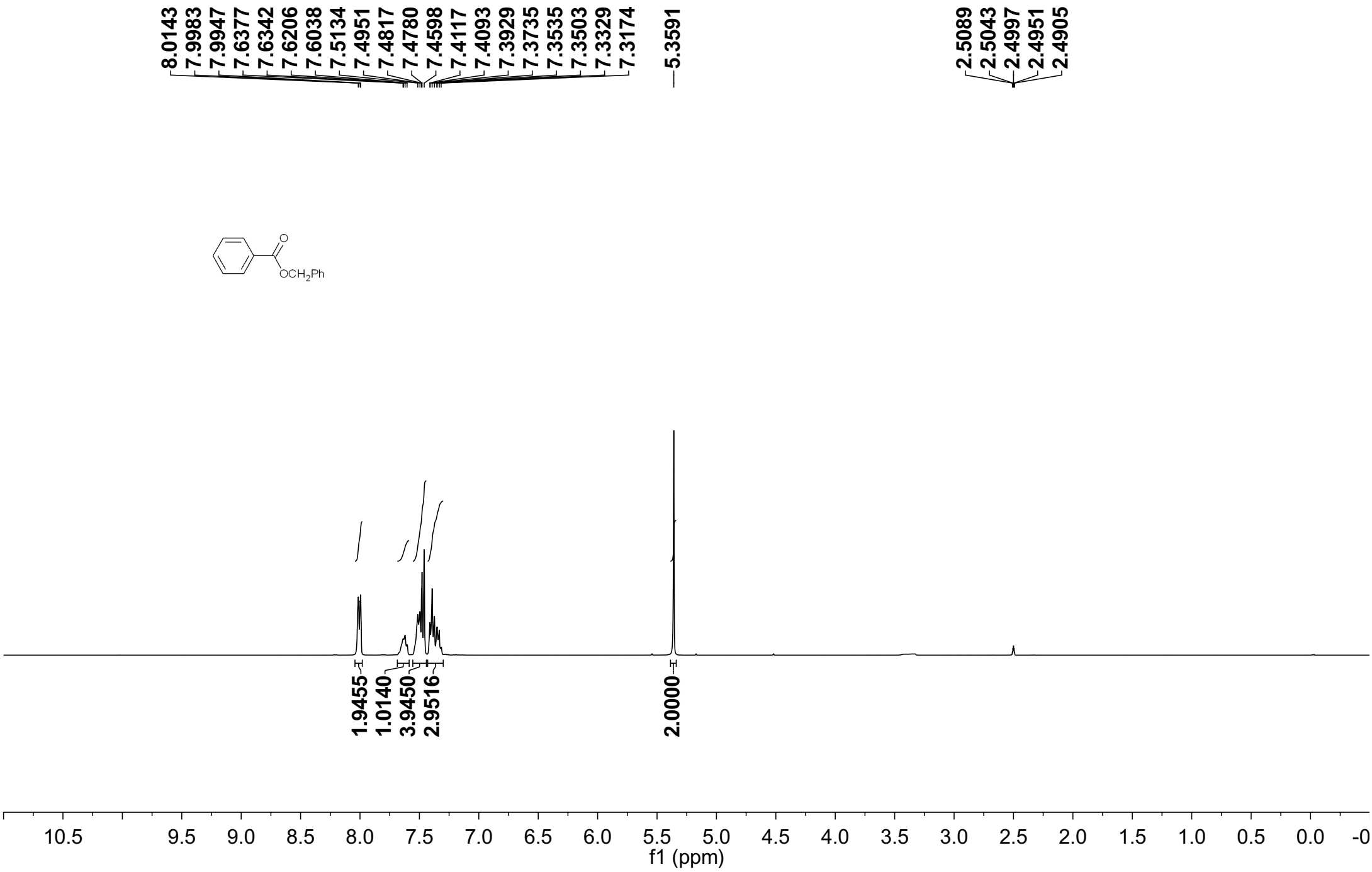
Compound 2o


7.9453
7.9401
7.9322
7.9244
7.9191
7.5739
7.5606
7.5568
4.3270
4.3093
4.2916
4.2739
3.3097
2.5043
2.4999
2.4954
2.4911
1.3272
1.3094
1.2916
Cl
O
OCH2CH3
1.9505
1.9394
2.0017
3.0000
10.5
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)

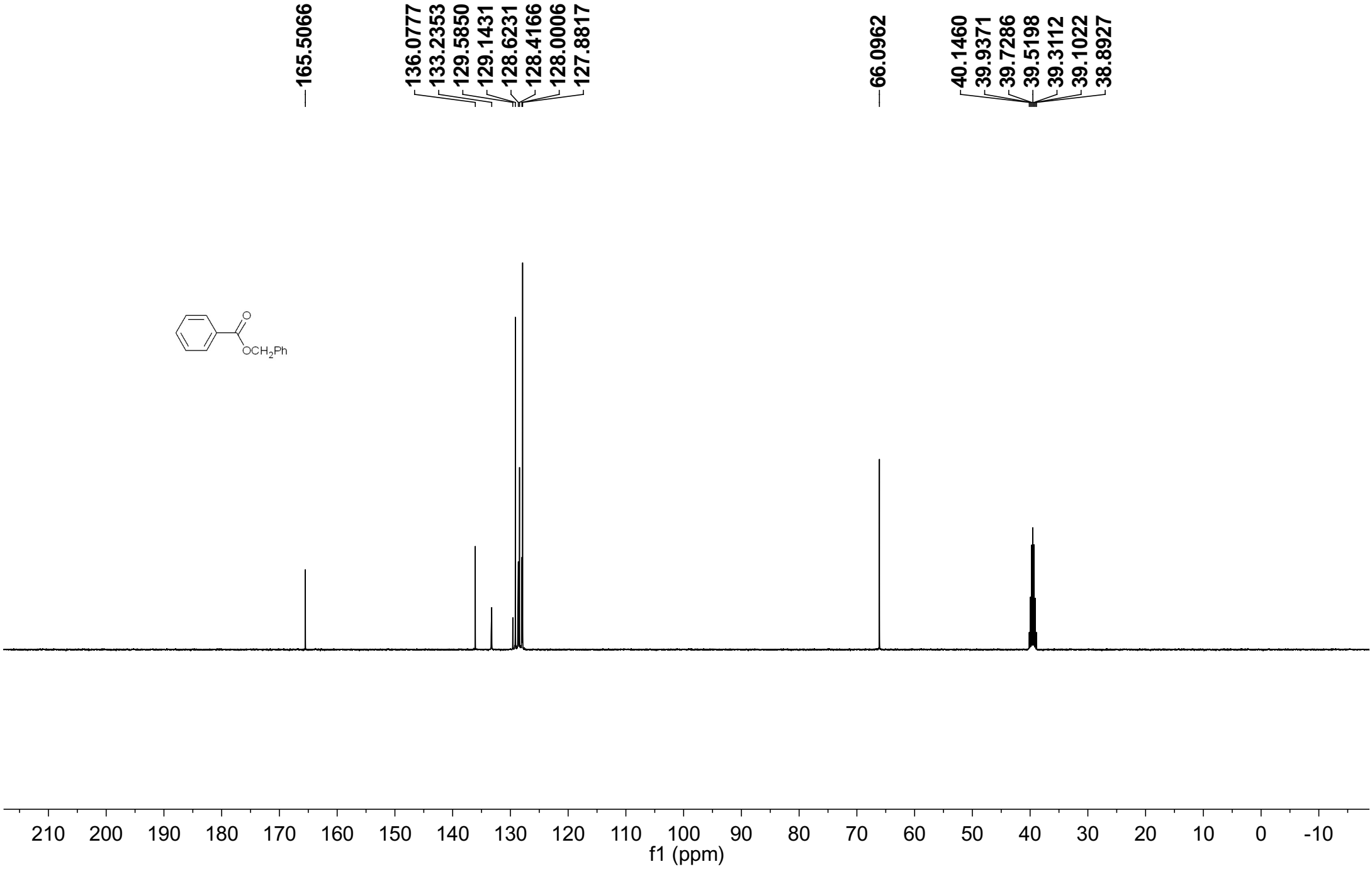
Compound 2o


164.7877
138.1120
130.8312
128.7804
128.6516
60.9084
40.1461
39.9370
39.7286
39.5200
39.3114
39.1030
38.8947
13.9868
Cl
O
OCH2CH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

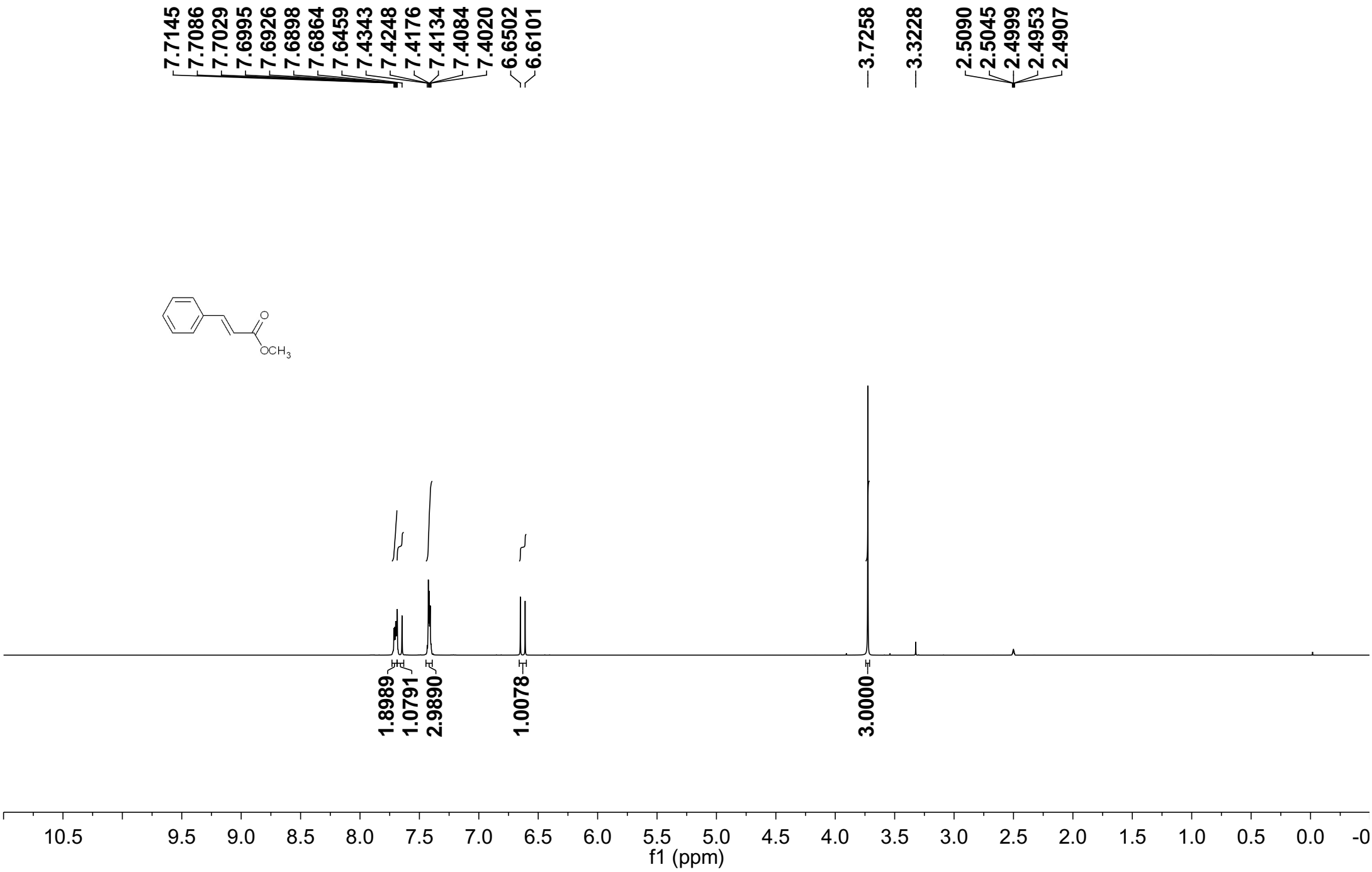
Compound 2p


8.0143
7.9983
7.9947
7.6377
7.6342
7.6206
7.6038
7.5134
7.4951
7.4817
7.4780
7.4598
7.4117
7.4093
7.3929
7.3735
7.3535
7.3503
7.3329
7.3174
5.3591
2.5089
2.5043
2.4997
2.4951
2.4905
OCH2Ph
1.9455
1.0140
3.9450
2.9516
2.0000
10.5
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
f1 (ppm)

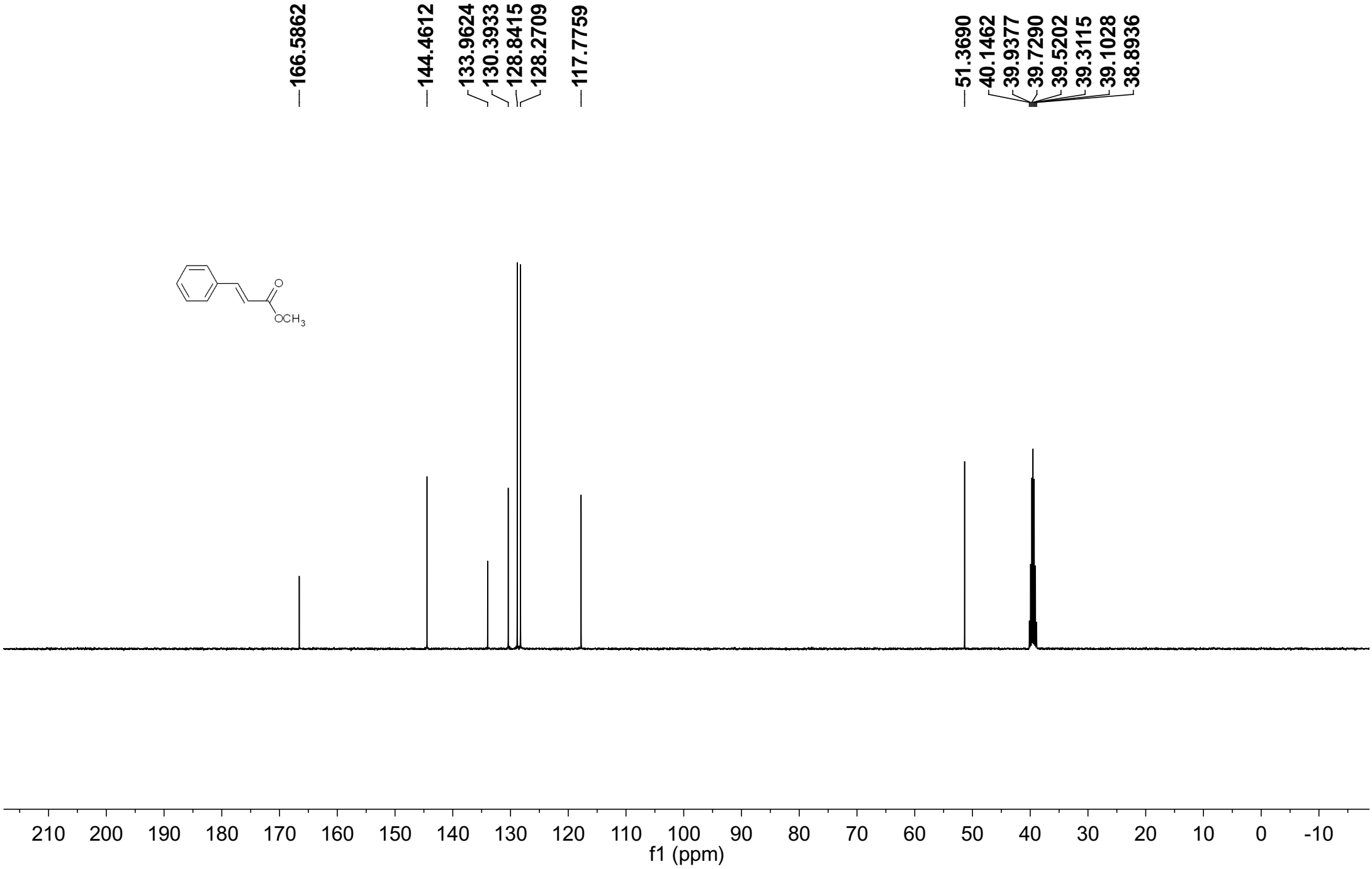
Compound 2p


165.5066
136.0777
133.2353
129.5850
129.1431
128.6231
128.4166
128.0006
127.8817
66.0962
40.1460
39.9371
39.7286
39.5198
39.3112
39.1022
38.8927
OCH2Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

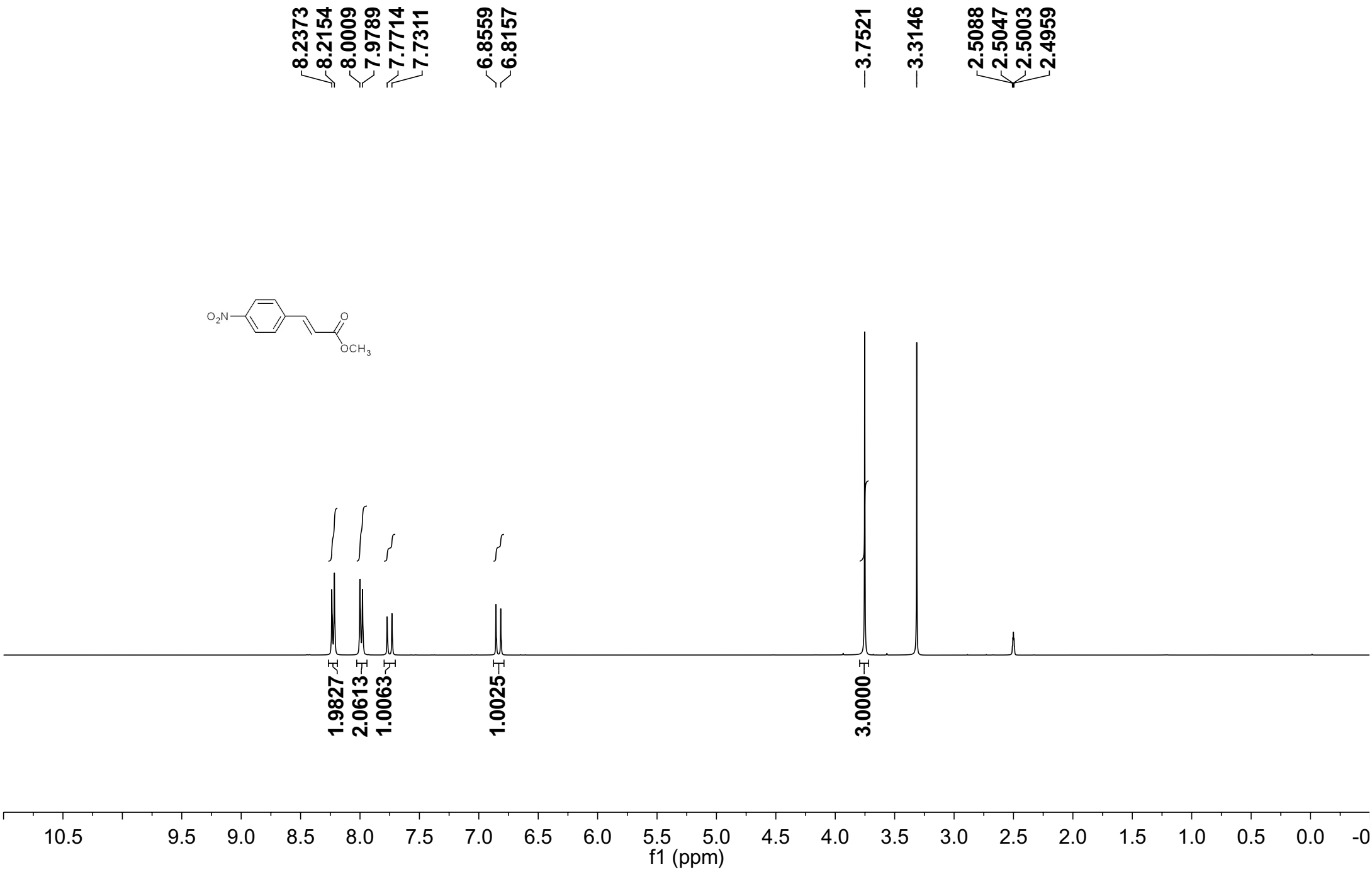
Compound 2q


7.7145
7.7086
7.7029
7.6995
7.6926
7.6898
7.6864
7.6459
7.4343
7.4248
7.4176
7.4134
7.4084
7.4020
6.6502
6.6101
3.7258
3.3228
2.5090
2.5045
2.4999
2.4953
2.4907
O
OCH3
1.8989
1.0791
2.9890
1.0078
3.0000
10.5
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
f1 (ppm)

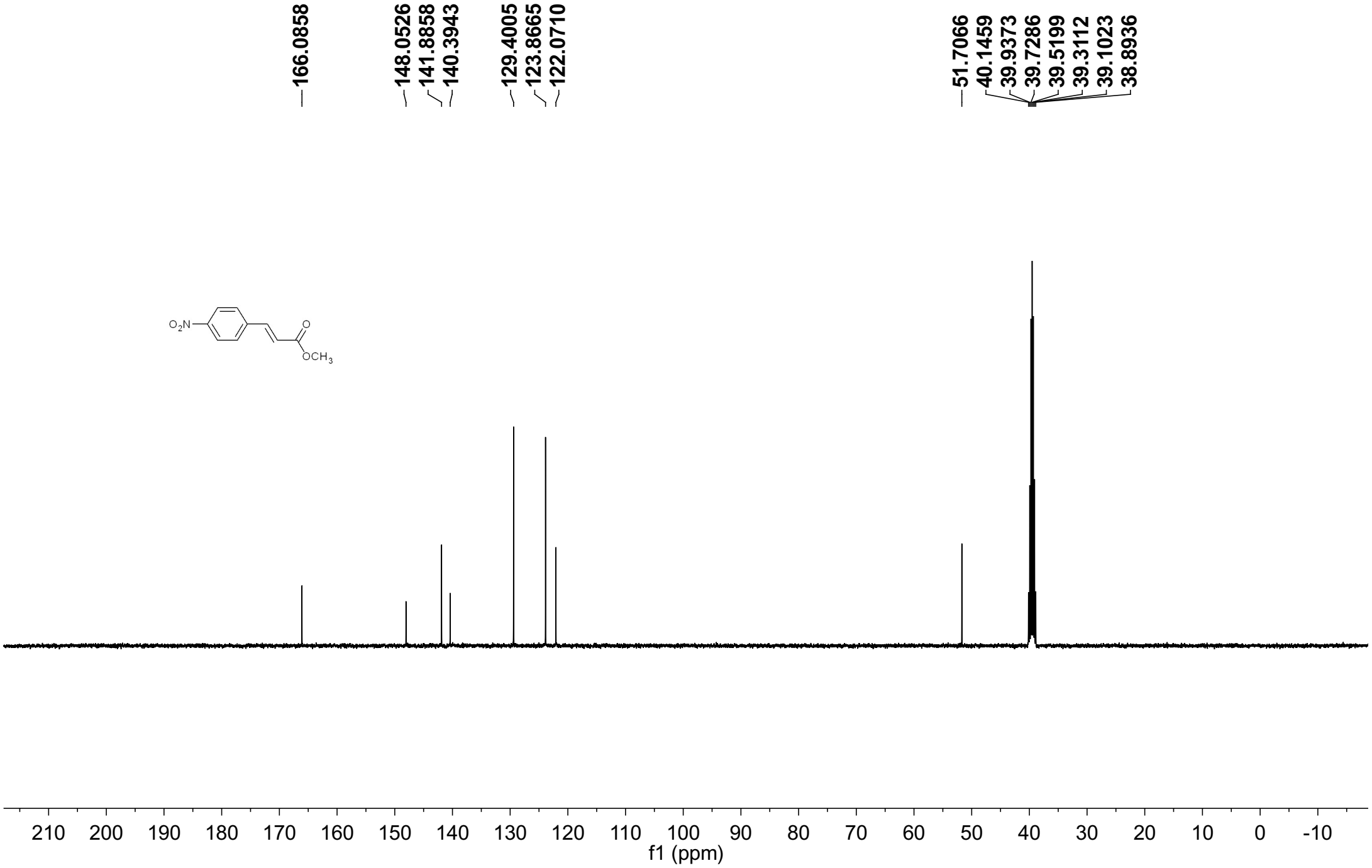
Compound 2q


166.5862
144.4612
133.9624
130.3933
128.8415
128.2709
117.7759
51.3690
40.1462
39.9377
39.7290
39.5202
39.3115
39.1028
38.8936
OCH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

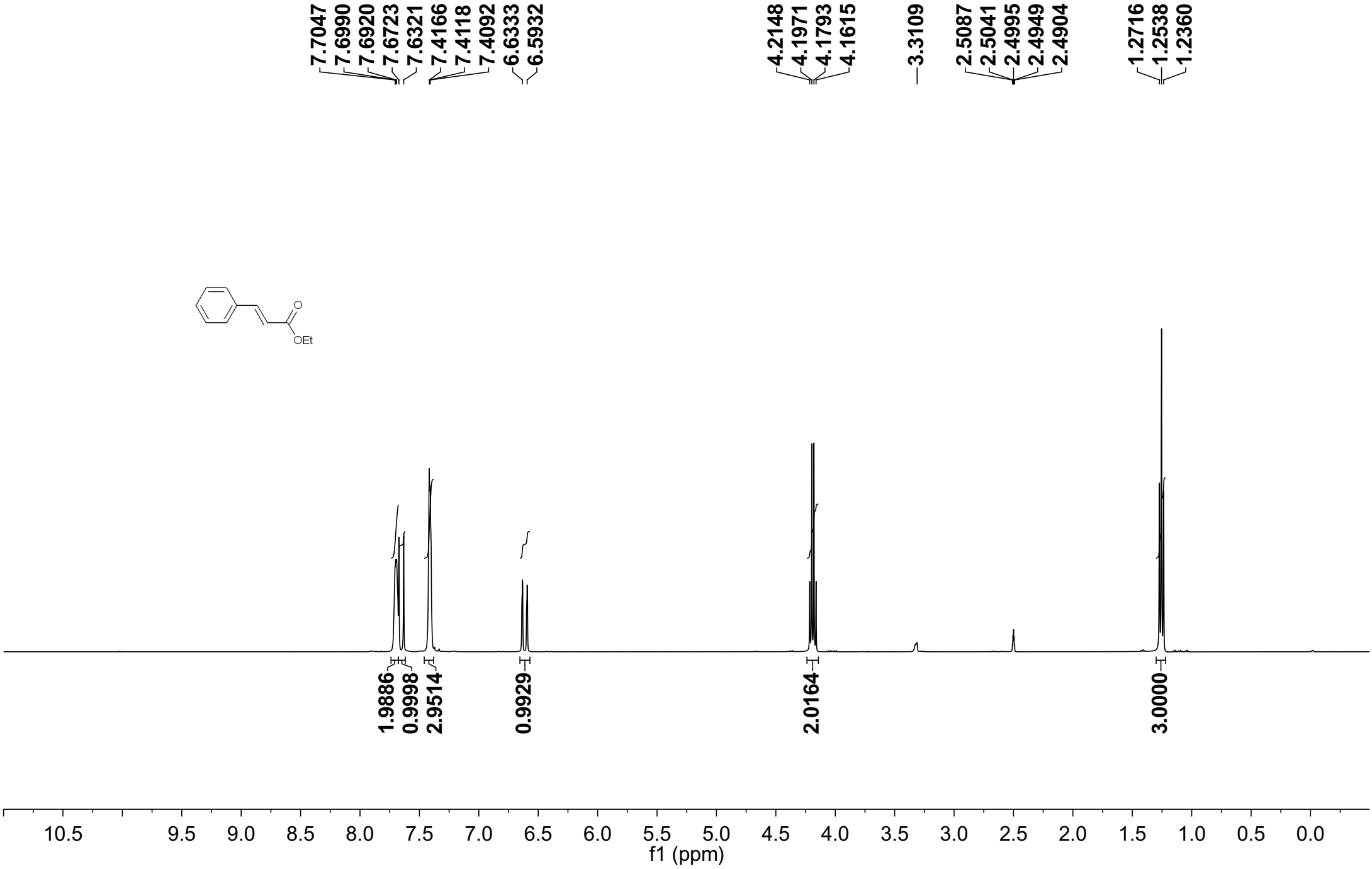
Compound 2r


8.2373
8.2154
8.0009
7.9789
7.7714
7.7311
6.8559
6.8157
3.7521
3.3146
2.5088
2.5047
2.5003
2.4959
O2N
O
OCH3
1.9827
2.0613
1.0063
1.0025
3.0000
10.5
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
f1 (ppm)

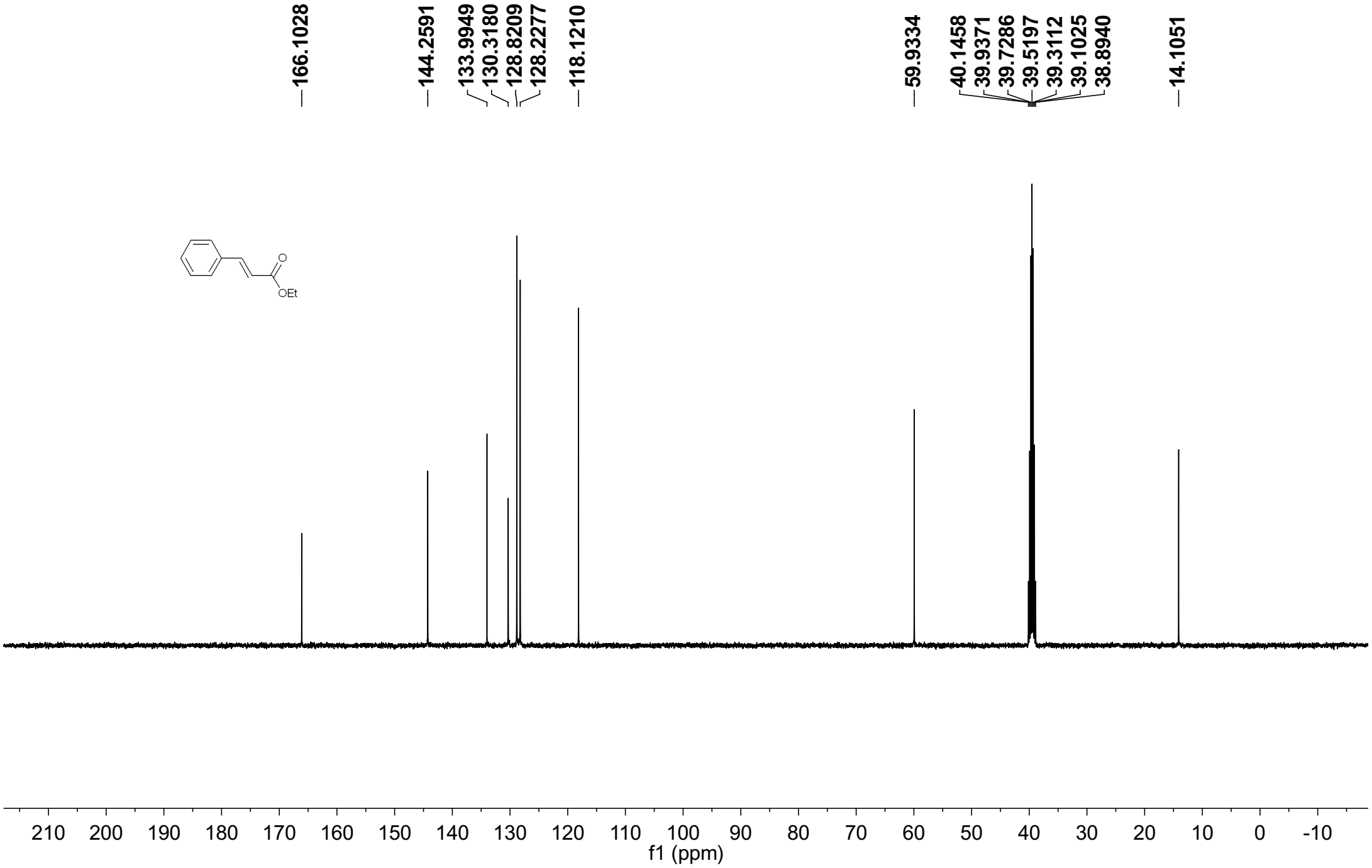
Compound 2r


166.0858
148.0526
141.8858
140.3943
129.4005
123.8665
122.0710
51.7066
40.1459
39.9373
39.7286
39.5199
39.3112
39.1023
38.8936
O2N
O
OCH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

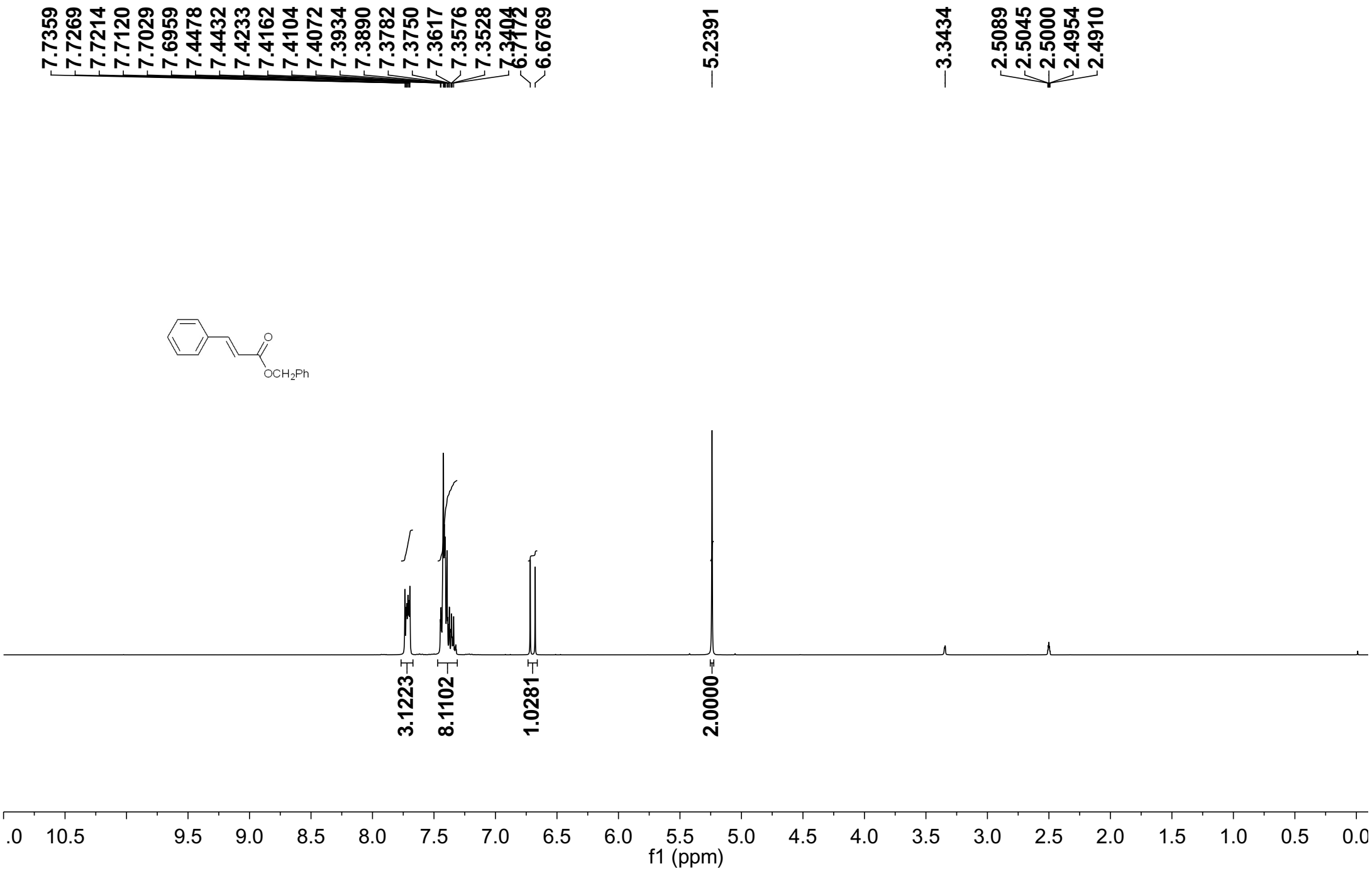
Compound 2s


7.7047
7.6990
7.6920
7.6723
7.6321
7.4166
7.4118
7.4092
6.6333
6.5932
4.2148
4.1971
4.1793
4.1615
3.3109
2.5087
2.5041
2.4995
2.4949
2.4904
1.2716
1.2538
1.2360
O
OEt
1.9886
0.9998
2.9514
0.9929
2.0164
3.0000
10.5
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)

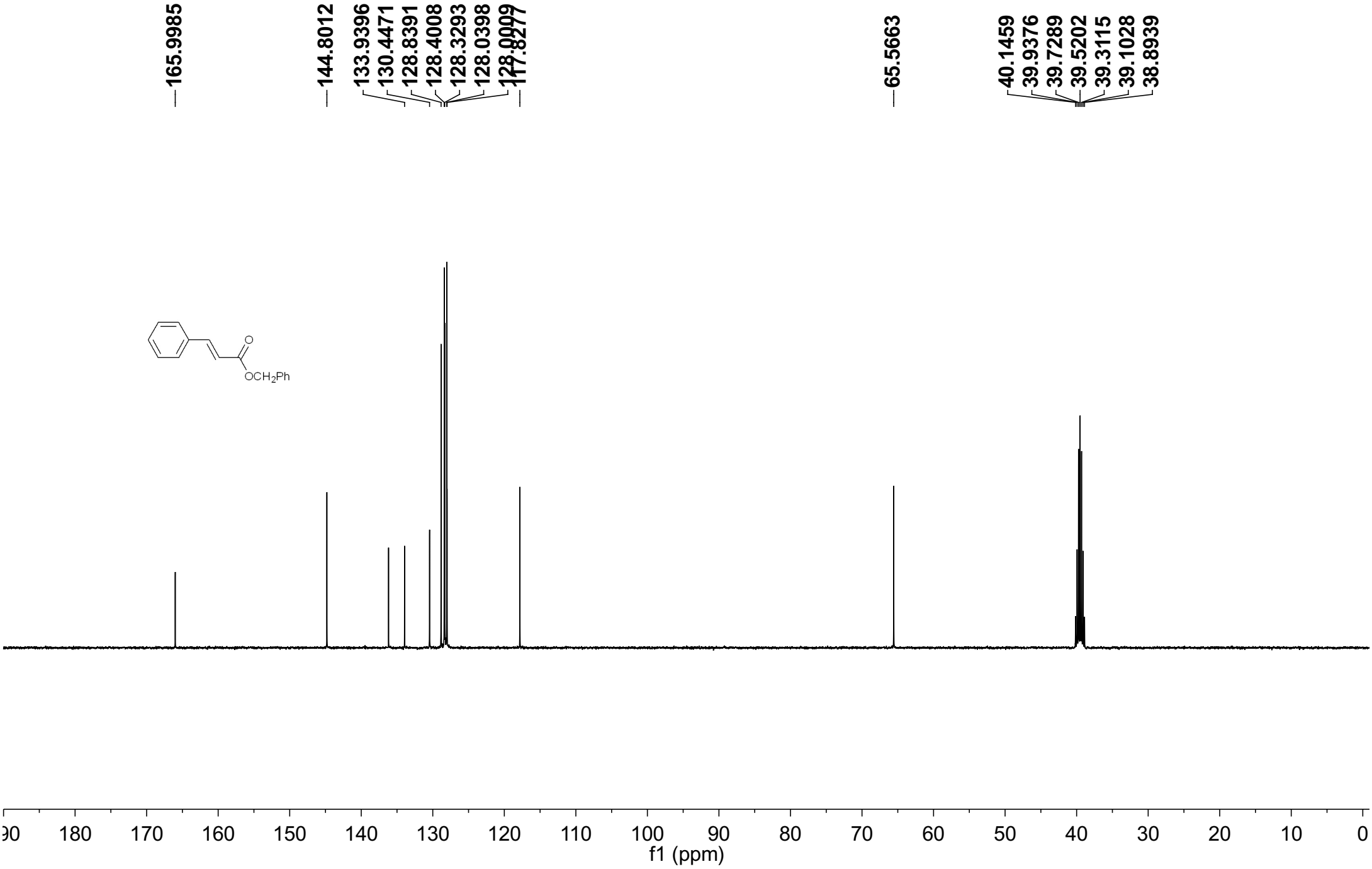
Compound 2s


166.1028
144.2591
133.9949
130.3180
128.8209
128.2277
118.1210
59.9334
40.1458
39.9371
39.7286
39.5197
39.3112
39.1025
38.8940
14.1051
OEt
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

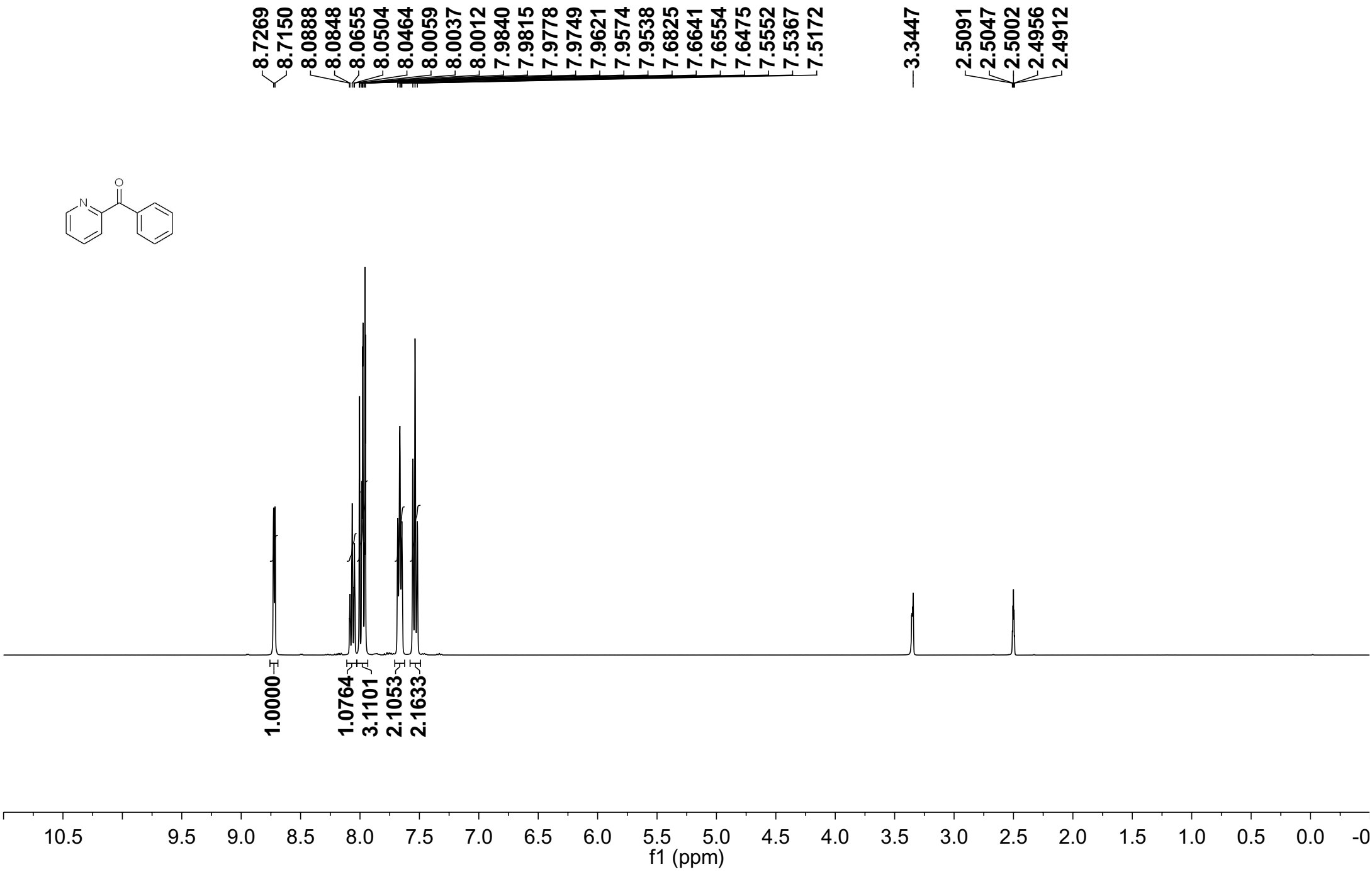
Compound 2t


7.7359
7.7269
7.7214
7.7120
7.7029
7.6959
7.4478
7.4432
7.4233
7.4162
7.4104
7.4072
7.3934
7.3890
7.3782
7.3750
7.3617
7.3576
7.3528
7.3404
6.7172
6.6769
5.2391
3.3434
2.5089
2.5045
2.5000
2.4954
2.4910
OCH2Ph
3.1223
8.1102
1.0281
2.0000
f1 (ppm)

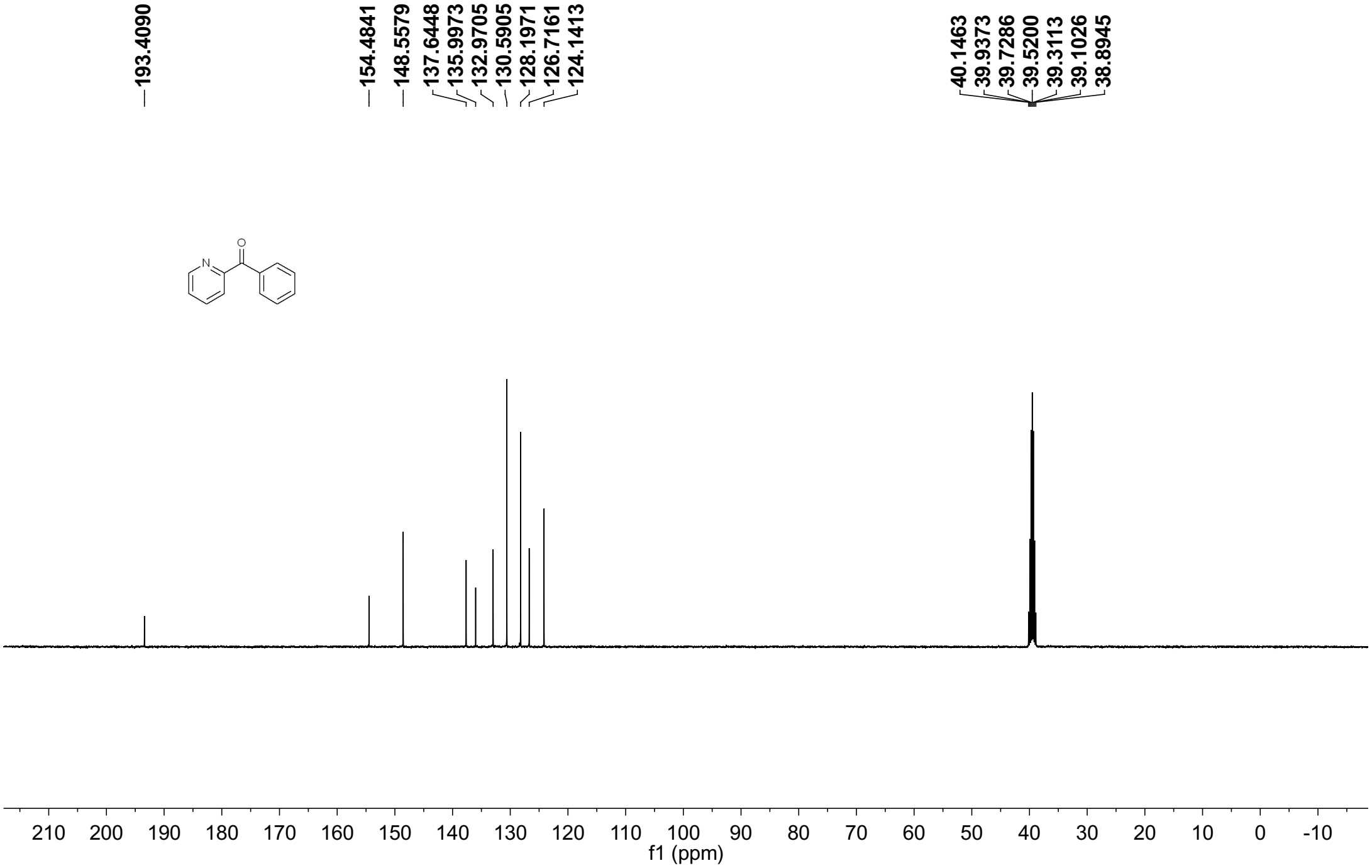
Compound 2t


165.9985
144.8012
133.9396
130.4471
128.8391
128.4008
128.3293
128.0398
128.0009
117.8277
65.5663
40.1459
39.9376
39.7289
39.5202
39.3115
39.1028
38.8939
OCH2Ph
O
f1 (ppm)

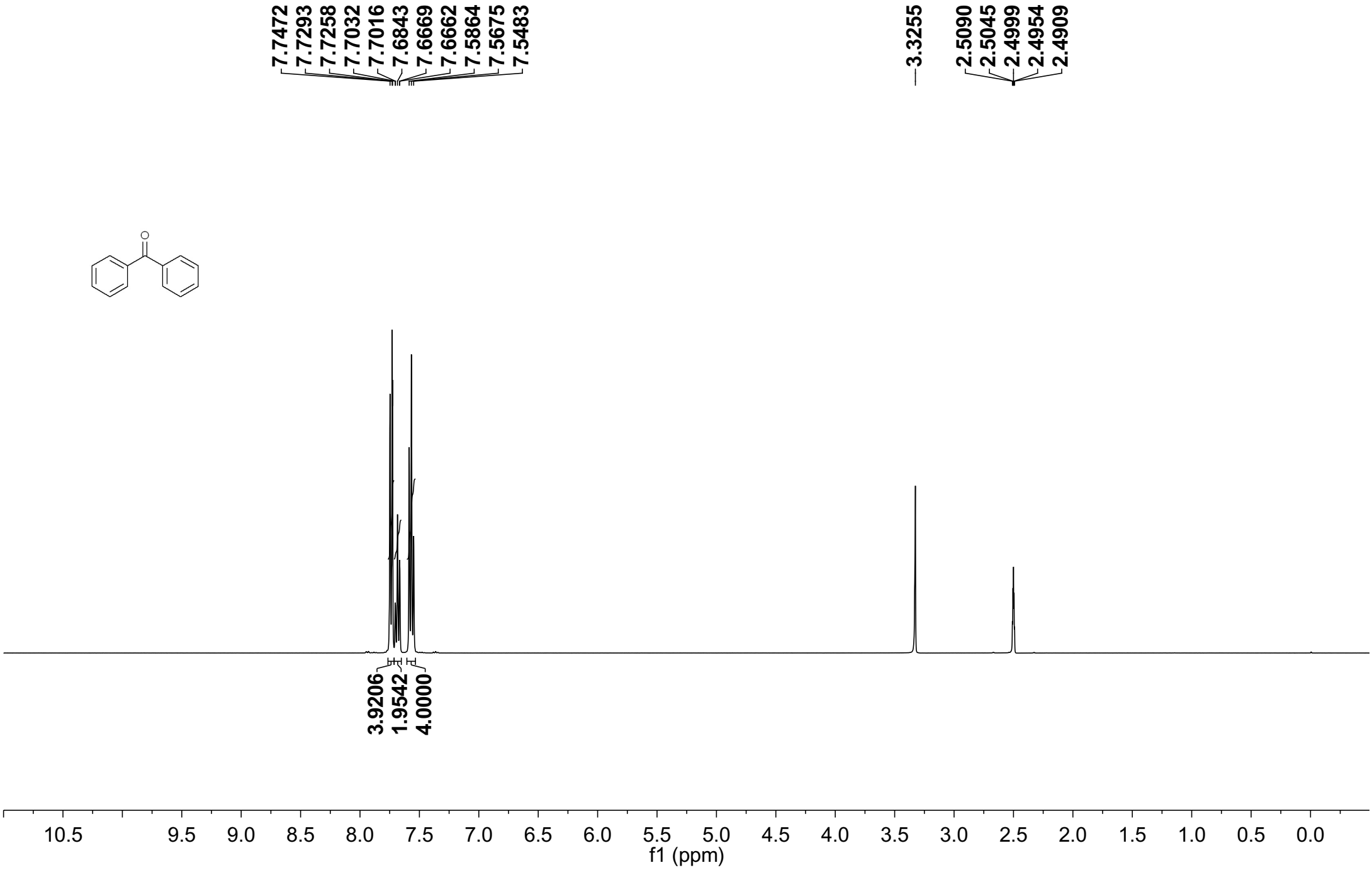
Compound 4a


8.7269
8.7150
8.0888
8.0848
8.0655
8.0504
8.0464
8.0059
8.0037
8.0012
7.9840
7.9815
7.9778
7.9749
7.9621
7.9574
7.9538
7.6825
7.6641
7.6554
7.6475
7.5552
7.5367
7.5172
3.3447
2.5091
2.5047
2.5002
2.4956
2.4912
1.0000
1.0764
3.1101
2.1053
2.1633
10.5
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)

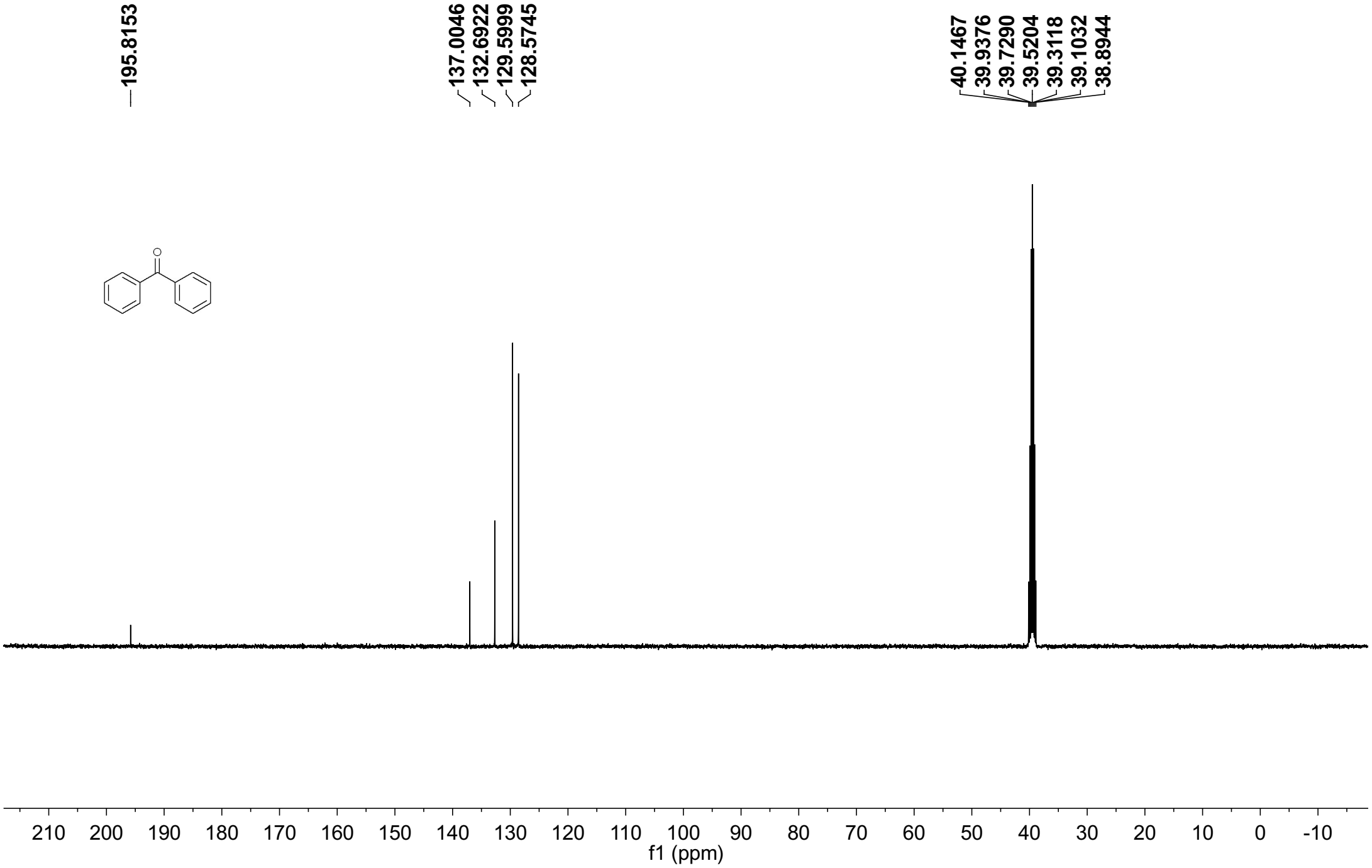
Compound 4a


193.4090
154.4841
148.5579
137.6448
135.9973
132.9705
130.5905
128.1971
126.7161
124.1413
40.1463
39.9373
39.7286
39.5200
39.3113
39.1026
38.8945
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

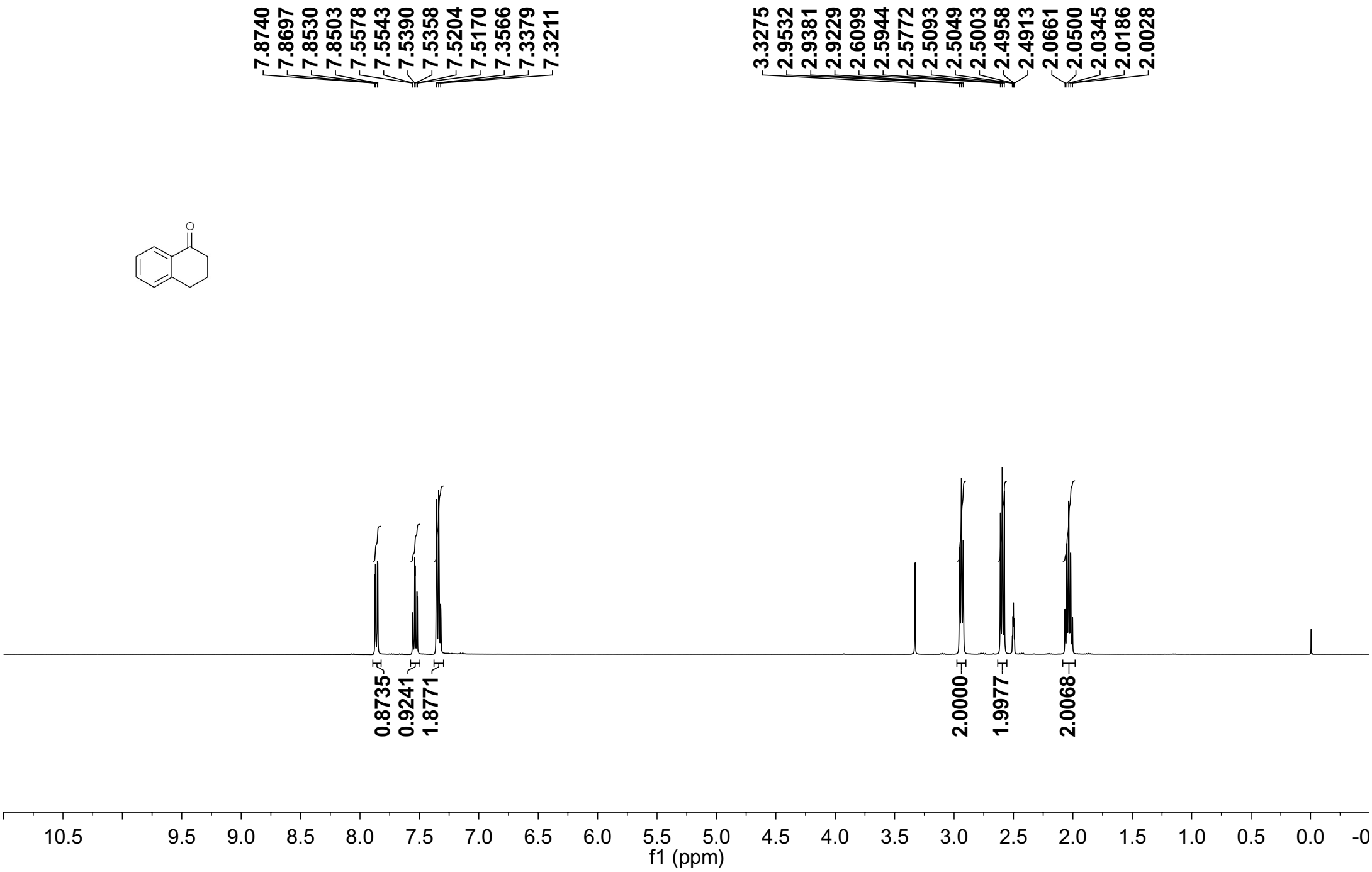
Compound 4b


7.7472
7.7293
7.7258
7.7032
7.7016
7.6843
7.6669
7.6662
7.5864
7.5675
7.5483
3.3255
2.5090
2.5045
2.4999
2.4954
2.4909
3.9206
1.9542
4.0000
10.5
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)

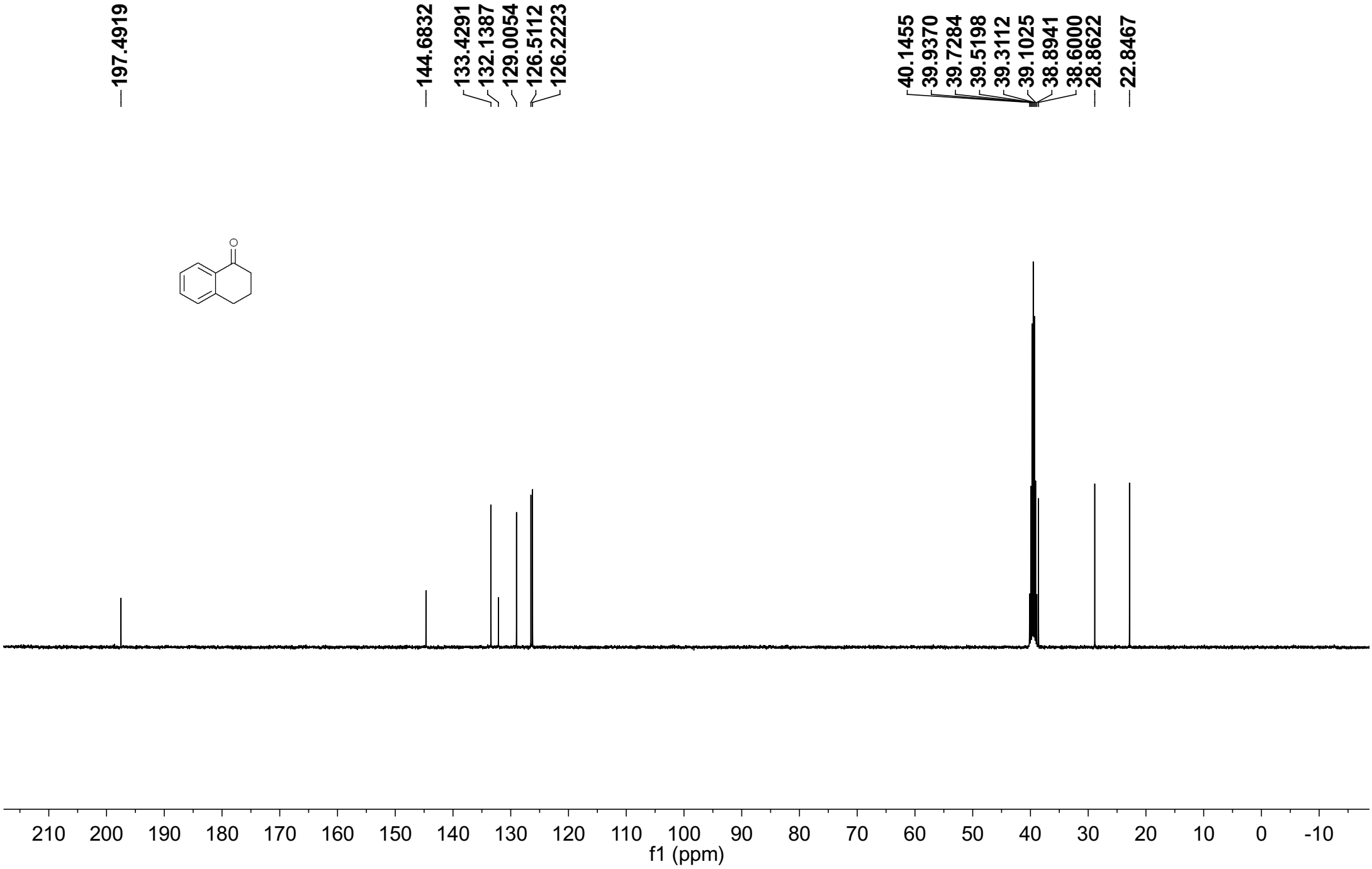
Compound 4b


195.8153
137.0046
132.6922
129.5999
128.5745
40.1467
39.9376
39.7290
39.5204
39.3118
39.1032
38.8944
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

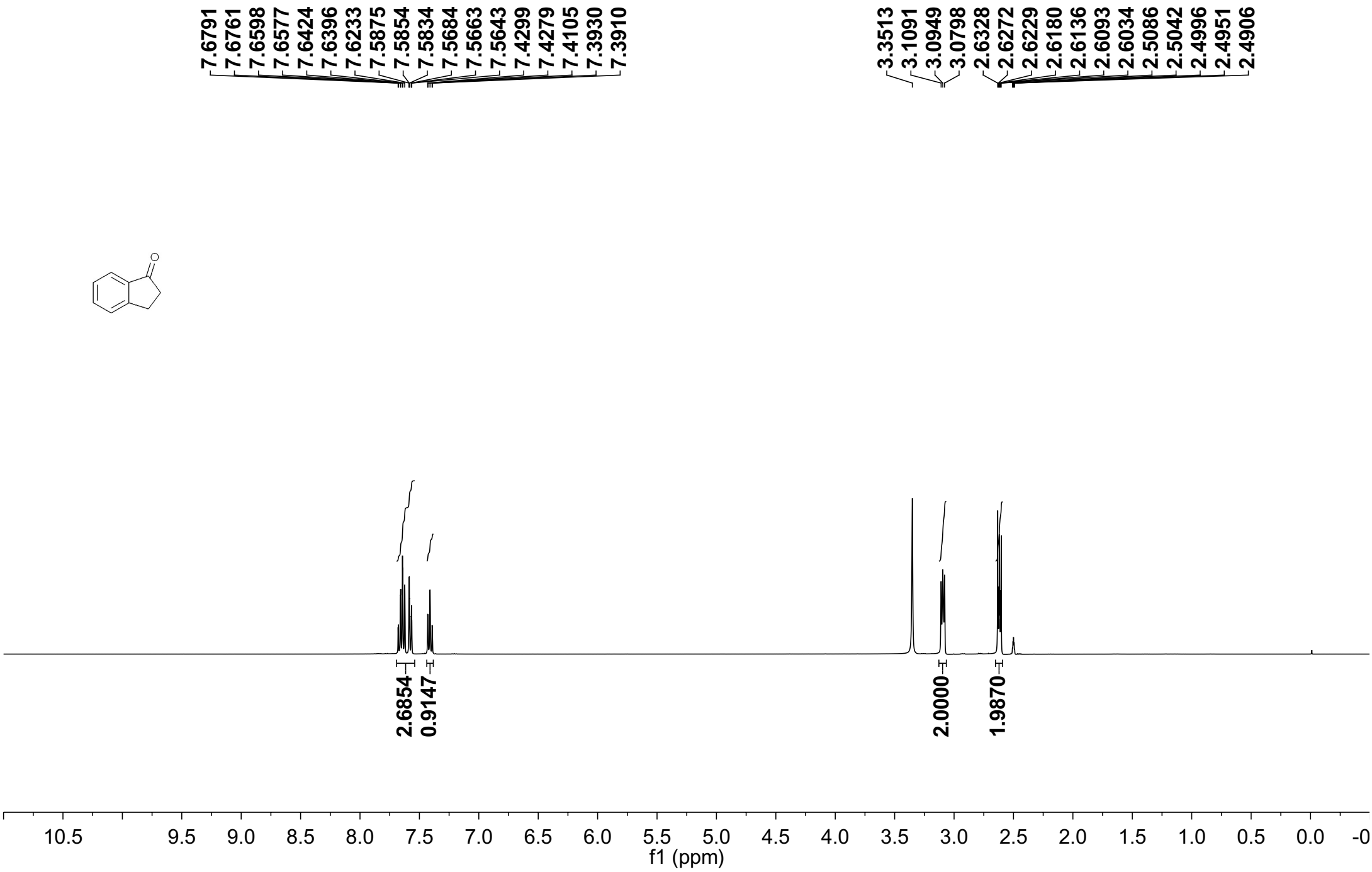
Compound 4c


7.8740
7.8697
7.8530
7.8503
7.5578
7.5543
7.5390
7.5358
7.5204
7.5170
7.3566
7.3379
7.3211
3.3275
2.9532
2.9381
2.9229
2.6099
2.5944
2.5772
2.5093
2.5049
2.5003
2.4958
2.4913
2.0661
2.0500
2.0345
2.0186
2.0028
0.8735
0.9241
1.8771
2.0000
1.9977
2.0068
10.5
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
f1 (ppm)

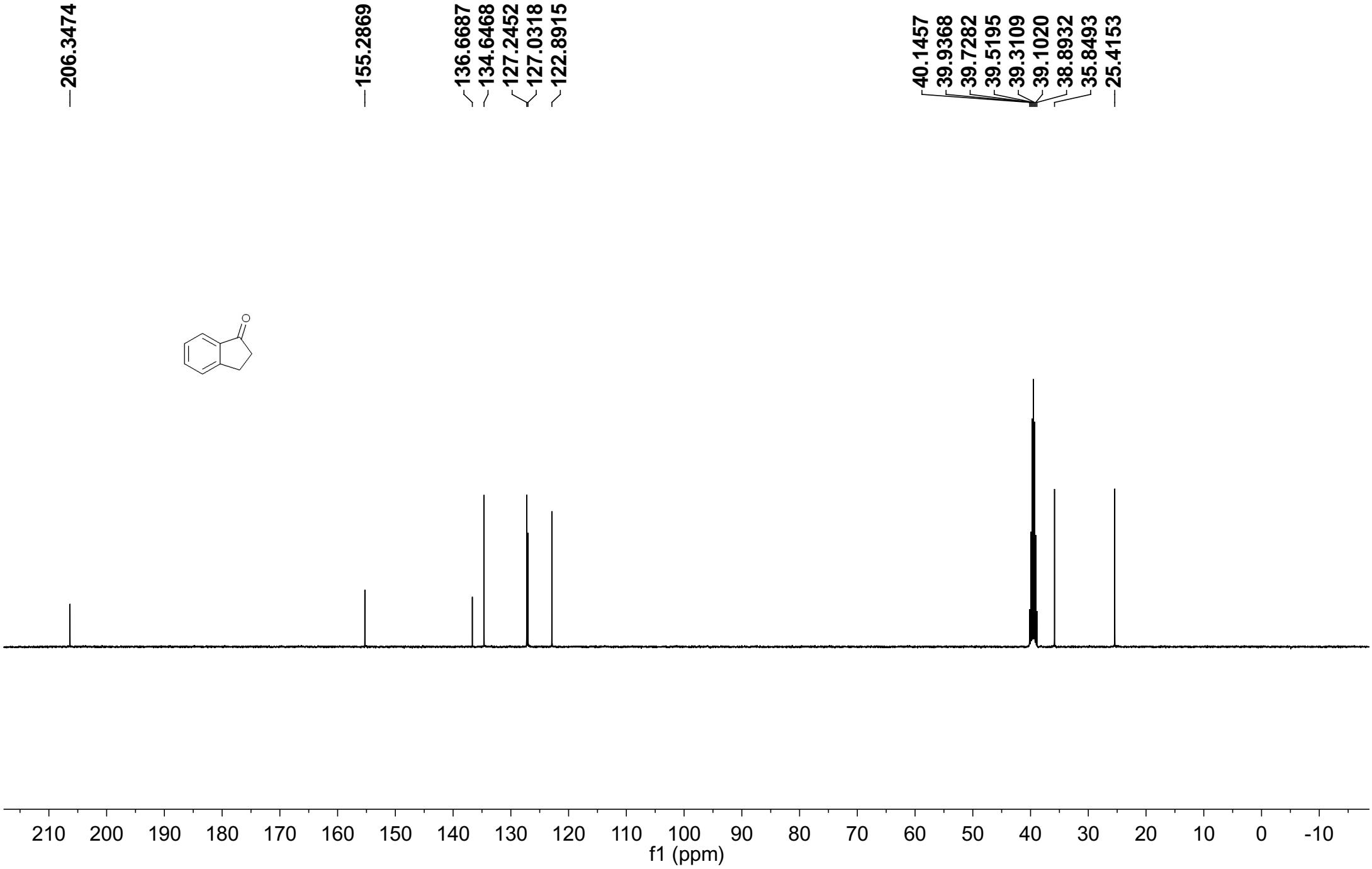
Compound 4c


197.4919
144.6832
133.4291
132.1387
129.0054
126.5112
126.2223
40.1455
39.9370
39.7284
39.5198
39.3112
39.1025
38.8941
38.6000
28.8622
22.8467
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

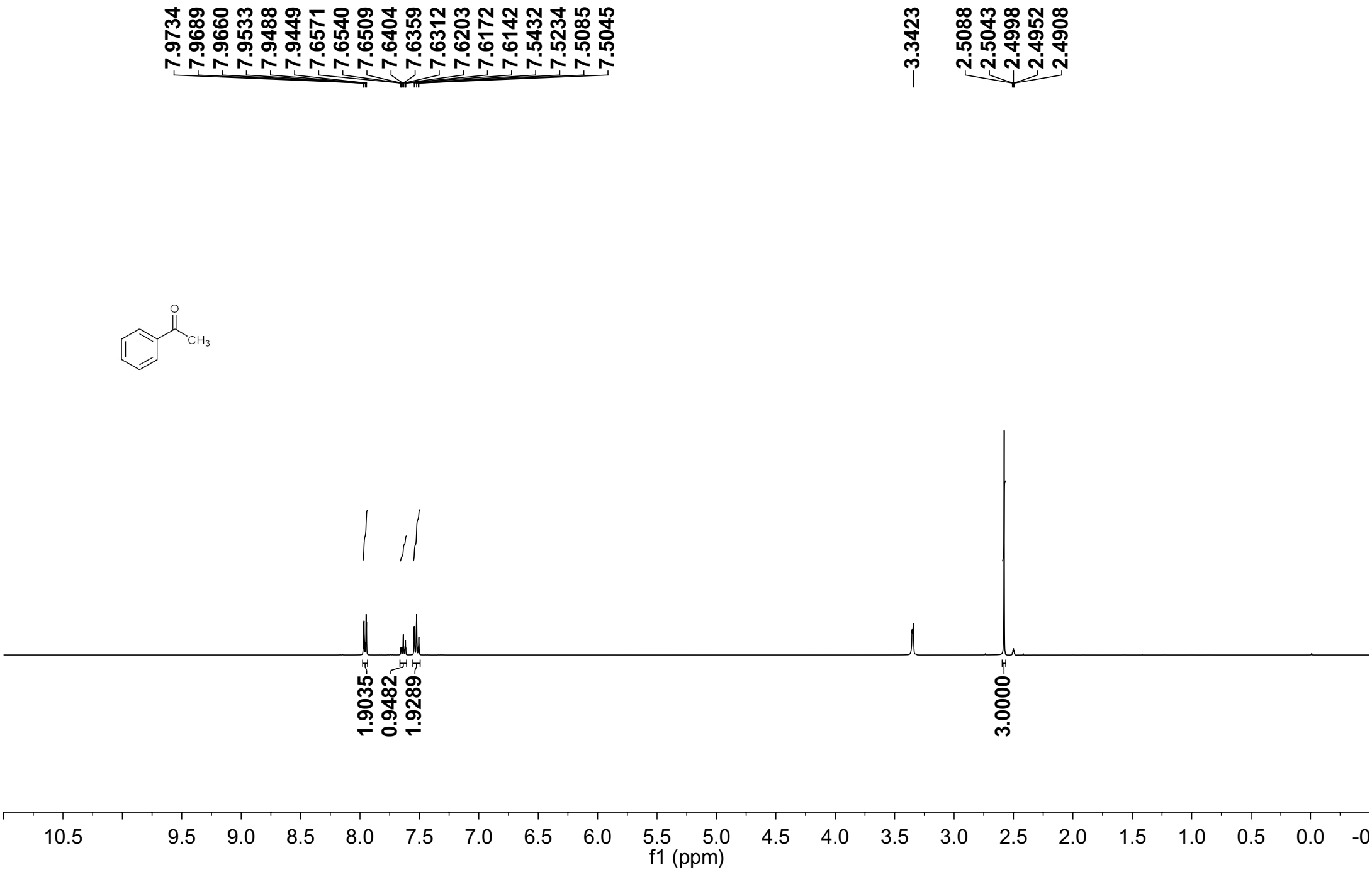
Compound 4d


7.6791
7.6761
7.6598
7.6577
7.6424
7.6396
7.6233
7.5875
7.5854
7.5834
7.5684
7.5663
7.5643
7.4299
7.4279
7.4105
7.3930
7.3910
3.3513
3.1091
3.0949
3.0798
2.6328
2.6272
2.6229
2.6180
2.6136
2.6093
2.6034
2.5086
2.5042
2.4996
2.4951
2.4906
2.6854
0.9147
2.0000
1.9870
10.5
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
f1 (ppm)

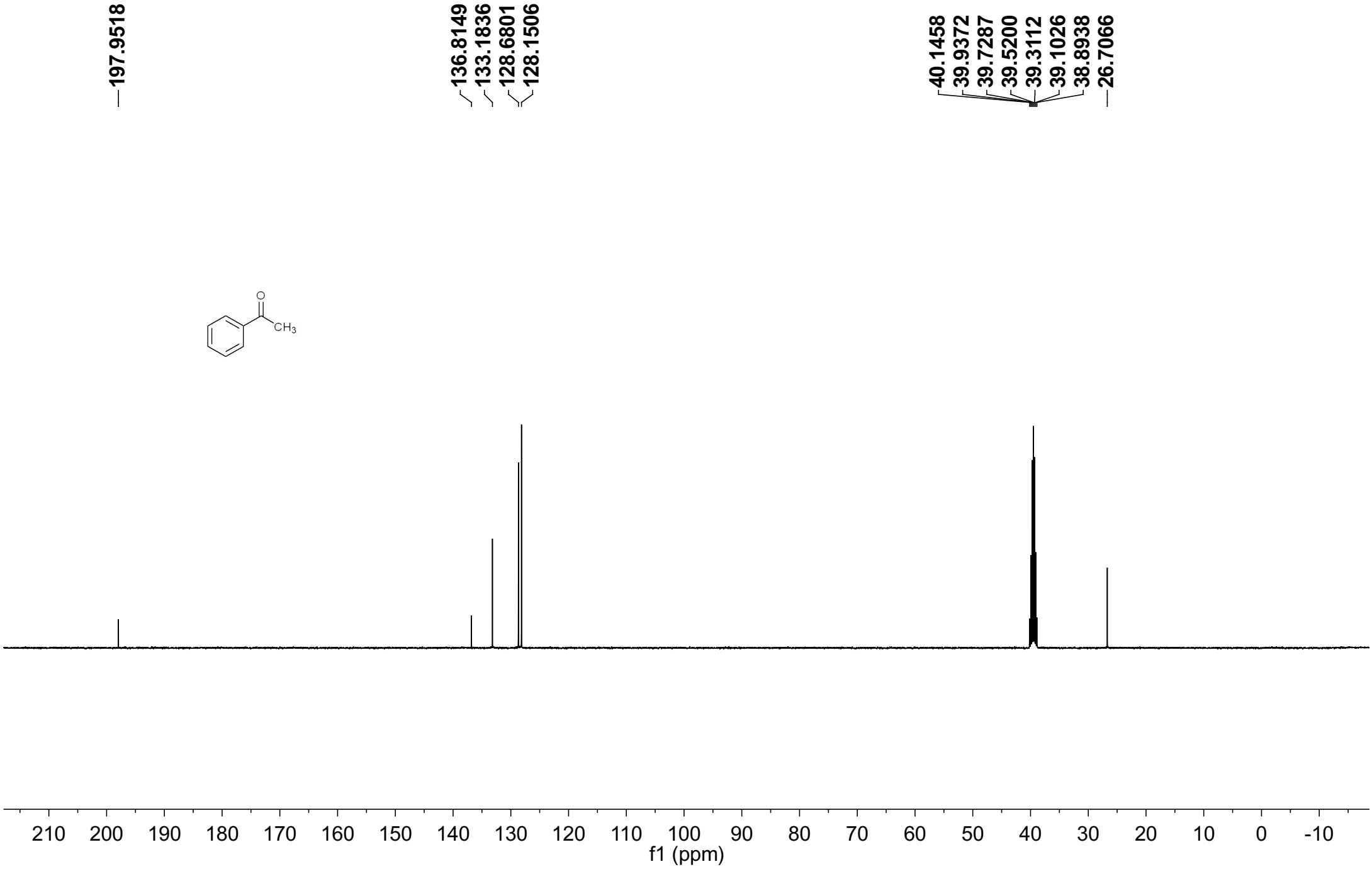
Compound 4d


206.3474
155.2869
136.6687
134.6468
127.2452
127.0318
122.8915
40.1457
39.9368
39.7282
39.5195
39.3109
39.1020
38.8932
35.8493
25.4153
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

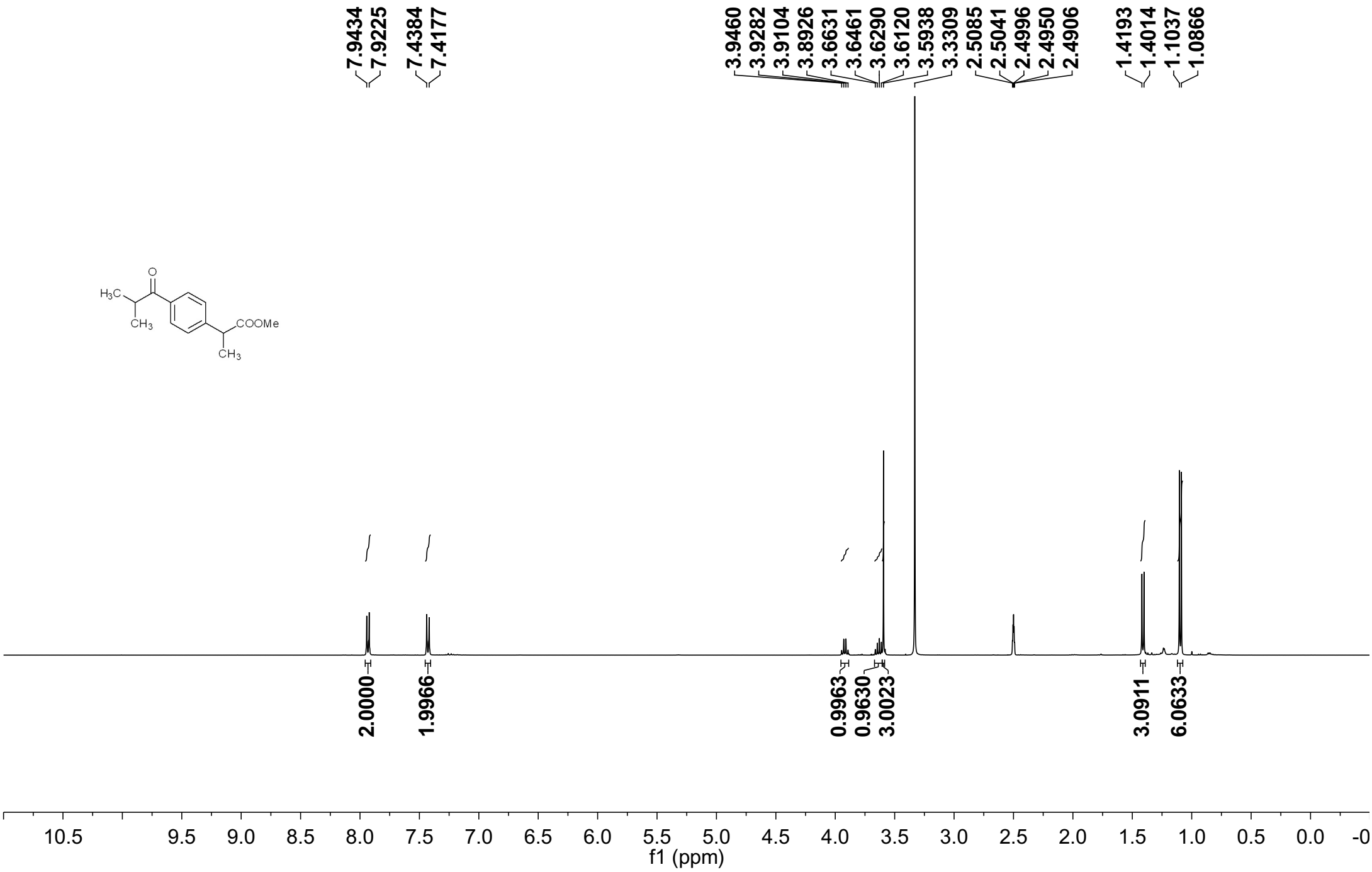
Compound 4e


7.9734
7.9689
7.9660
7.9533
7.9488
7.9449
7.6571
7.6540
7.6509
7.6404
7.6359
7.6312
7.6203
7.6172
7.6142
7.5432
7.5234
7.5085
7.5045
3.3423
2.5088
2.5043
2.4998
2.4952
2.4908
O
CH3
1.9035
0.9482
1.9289
3.0000
10.5
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
f1 (ppm)

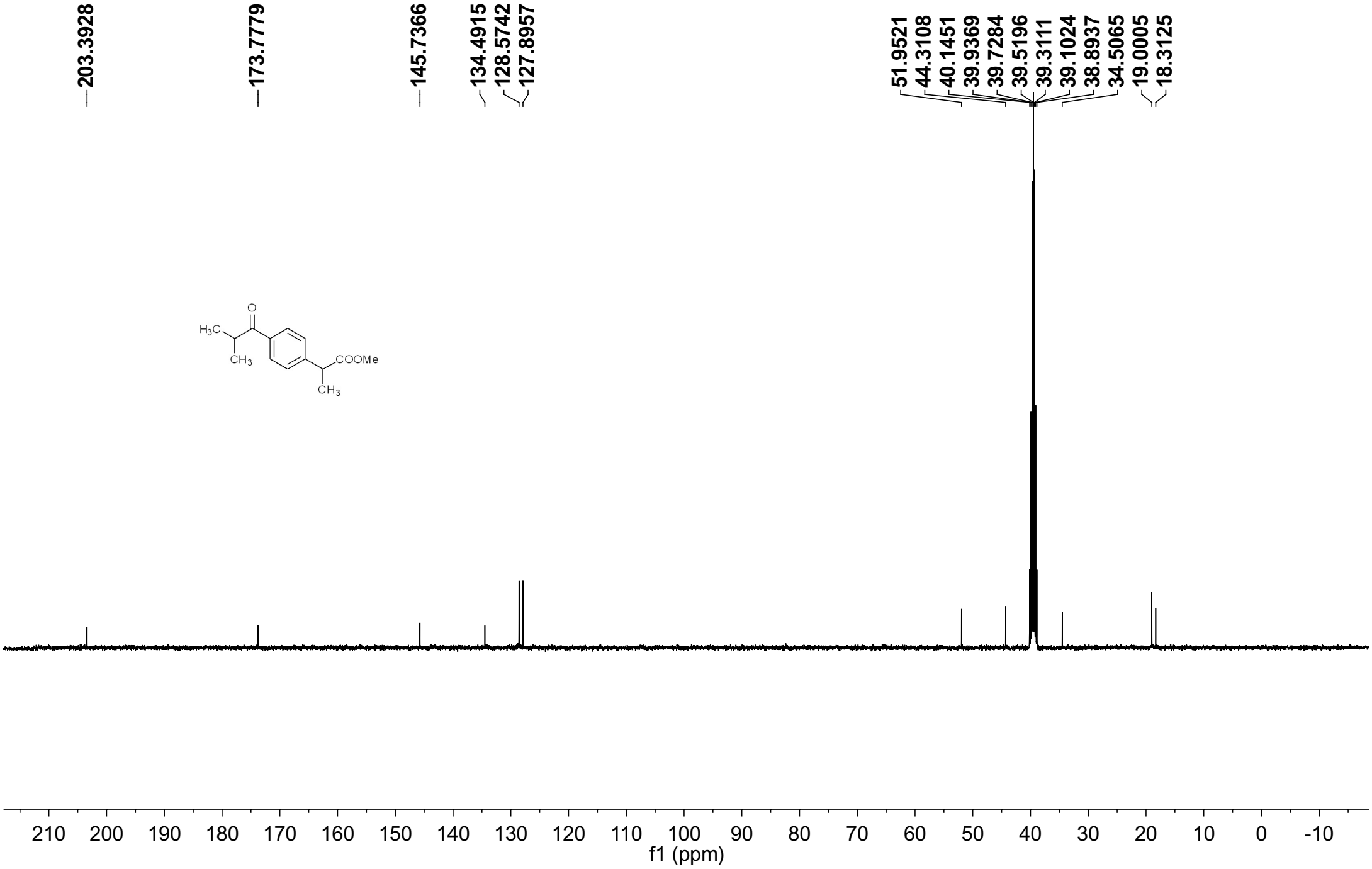
Compound 4e


197.9518
136.8149
133.1836
128.6801
128.1506
40.1458
39.9372
39.7287
39.5200
39.3112
39.1026
38.8938
26.7066
O
CH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

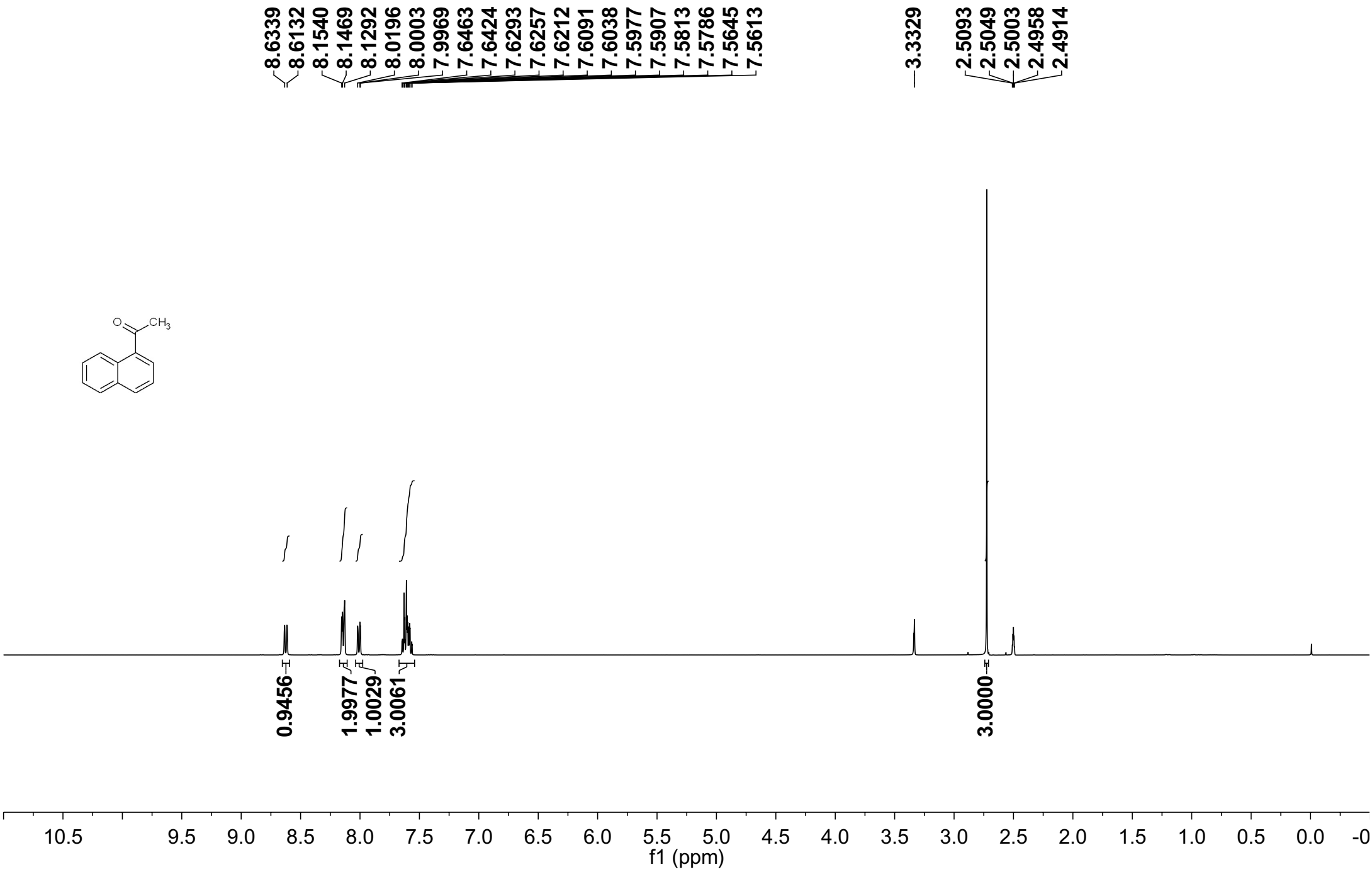
Compound 4f


7.9434
7.9225
7.4384
7.4177
3.9460
3.9282
3.9104
3.8926
3.6631
3.6461
3.6290
3.6120
3.5938
3.3309
2.5085
2.5041
2.4996
2.4950
2.4906
1.4193
1.4014
1.1037
1.0866
2.0000
1.9966
0.9963
0.9630
3.0023
3.0911
6.0633
O
H3C
CH3
COOMe
CH3
10.5
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
f1 (ppm)

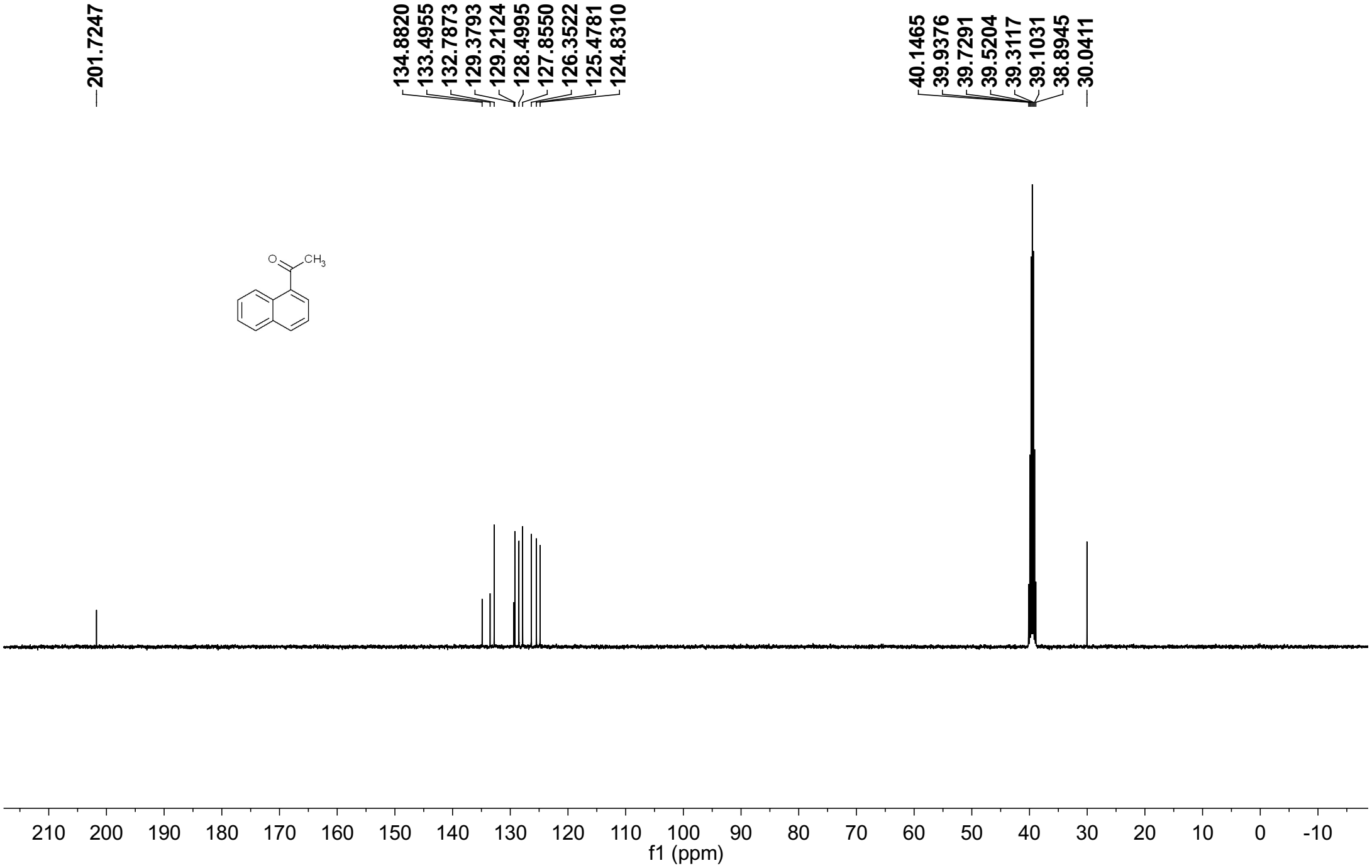
Compound 4f


203.3928
173.7779
145.7366
134.4915
128.5742
127.8957
51.9521
44.3108
40.1451
39.9369
39.7284
39.5196
39.3111
39.1024
38.8937
34.5065
19.0005
18.3125
O
H3C
CH3
COOMe
CH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

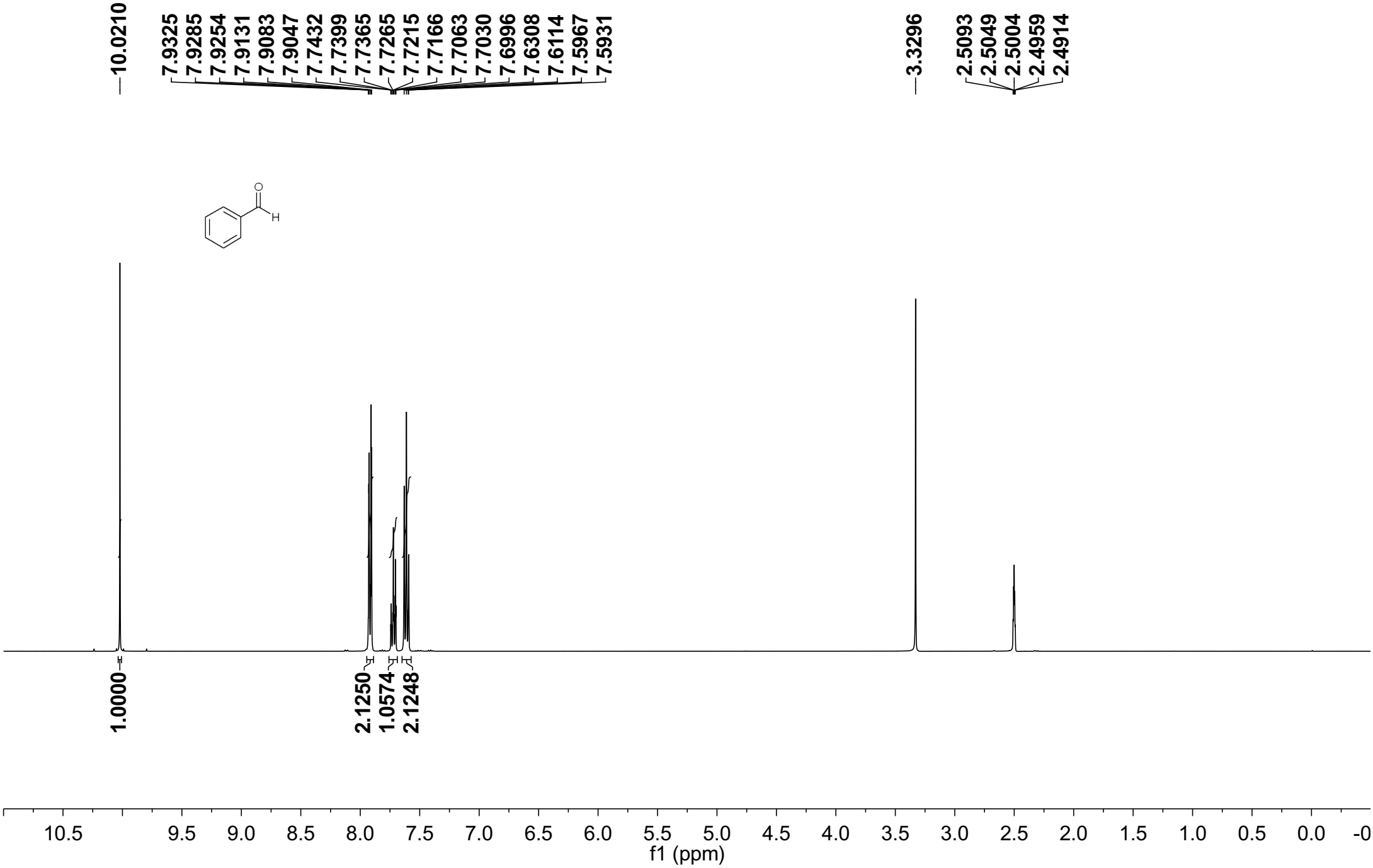
Compound 4g


8.6339
8.6132
8.1540
8.1469
8.1292
8.0196
8.0003
7.9969
7.6463
7.6424
7.6293
7.6257
7.6212
7.6091
7.6038
7.5977
7.5907
7.5813
7.5786
7.5645
7.5613
3.3329
2.5093
2.5049
2.5003
2.4958
2.4914
O
CH3
0.9456
1.9977
1.0029
3.0061
3.0000
10.5
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
f1 (ppm)

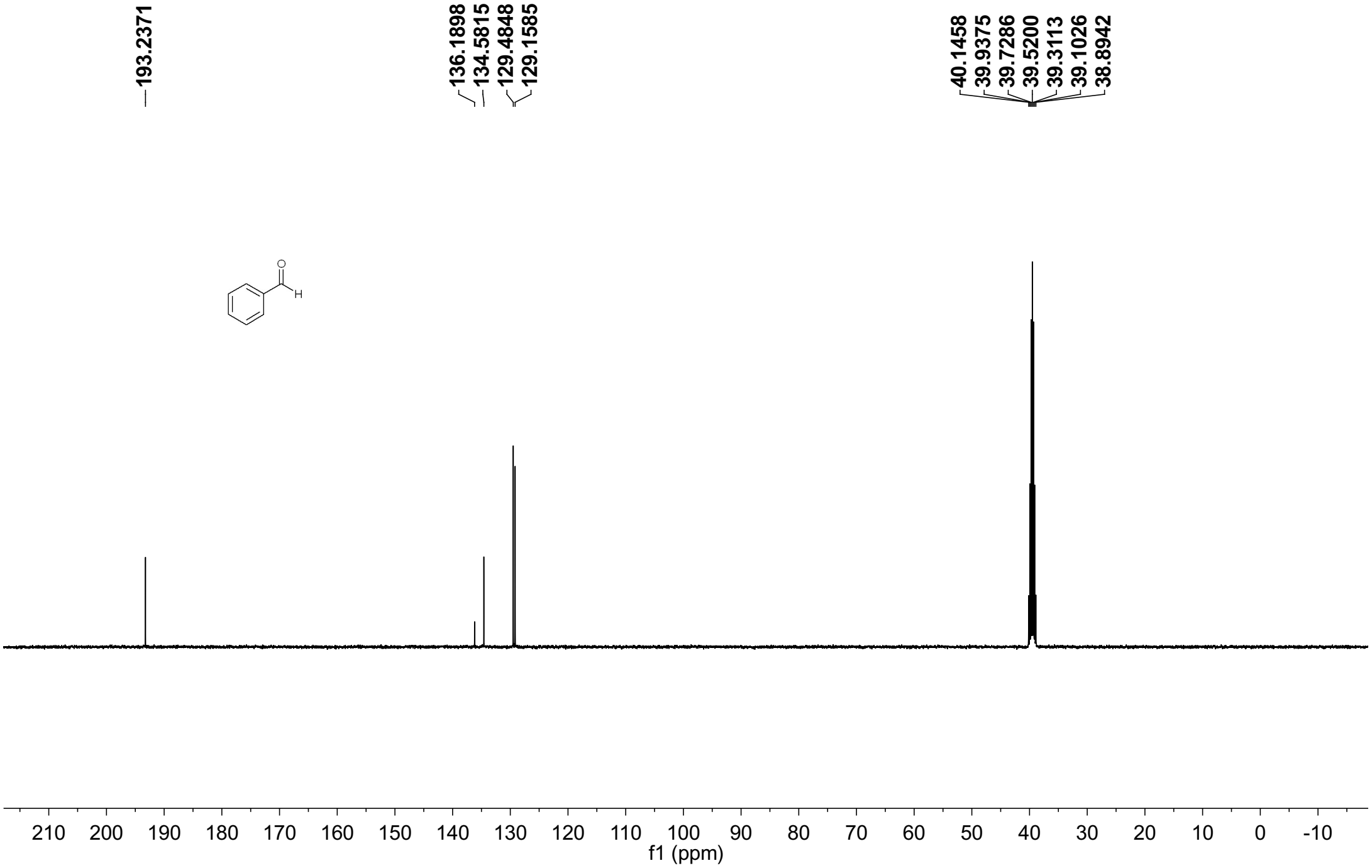
Compound 4g


201.7247
134.8820
133.4955
132.7873
129.3793
129.2124
128.4995
127.8550
126.3522
125.4781
124.8310
40.1465
39.9376
39.7291
39.5204
39.3117
39.1031
38.8945
30.0411
O
CH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

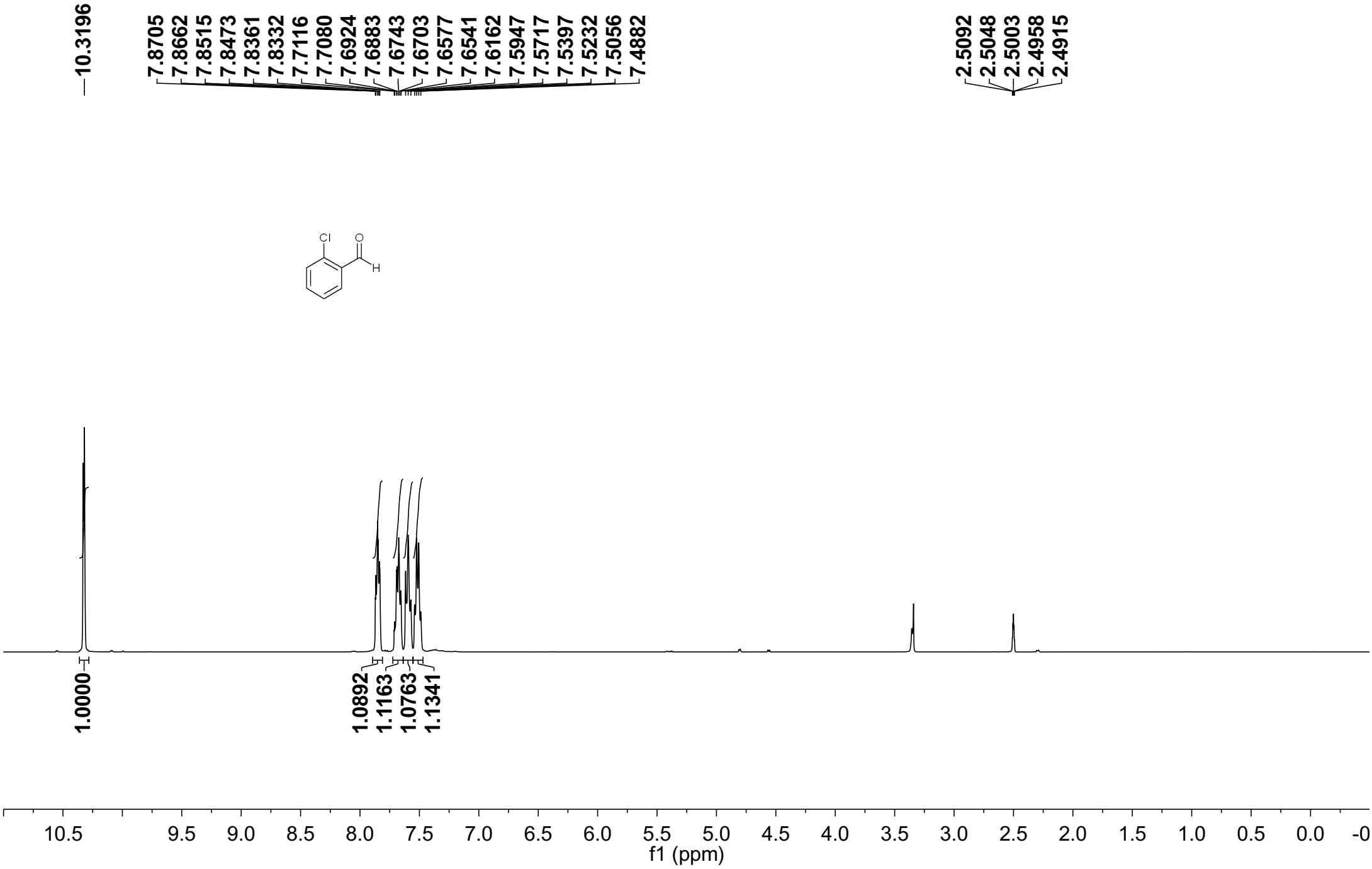
Compound 6a


10.0210
7.9325
7.9285
7.9254
7.9131
7.9083
7.9047
7.7432
7.7399
7.7365
7.7265
7.7215
7.7166
7.7063
7.7030
7.6996
7.6308
7.6114
7.5967
7.5931
3.3296
2.5093
2.5049
2.5004
2.4959
2.4914
O
H
1.0000
2.1250
1.0574
2.1248
10.5
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
f1 (ppm)

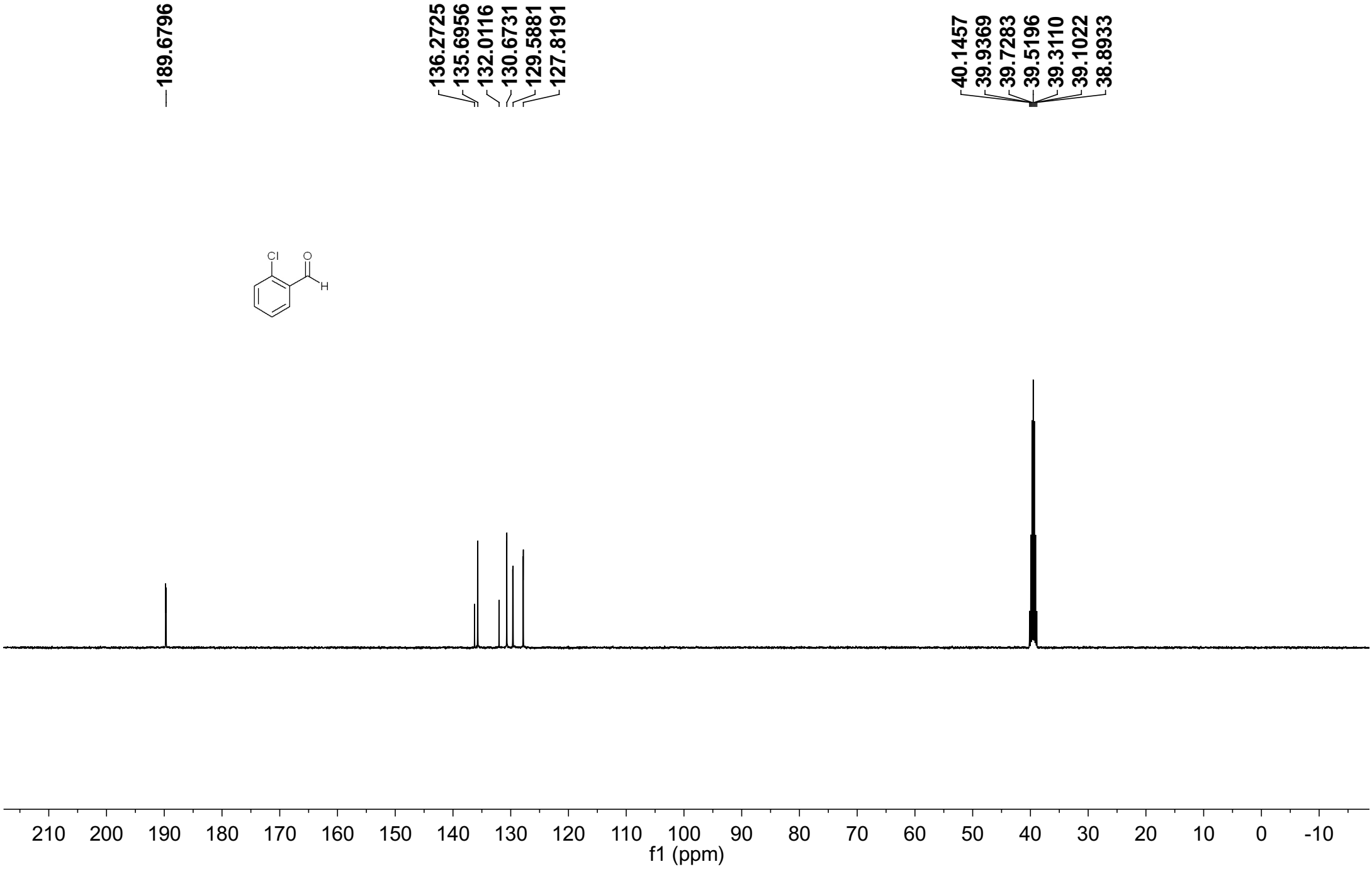
Compound 6a


193.2371
136.1898
134.5815
129.4848
129.1585
40.1458
39.9375
39.7286
39.5200
39.3113
39.1026
38.8942
O
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

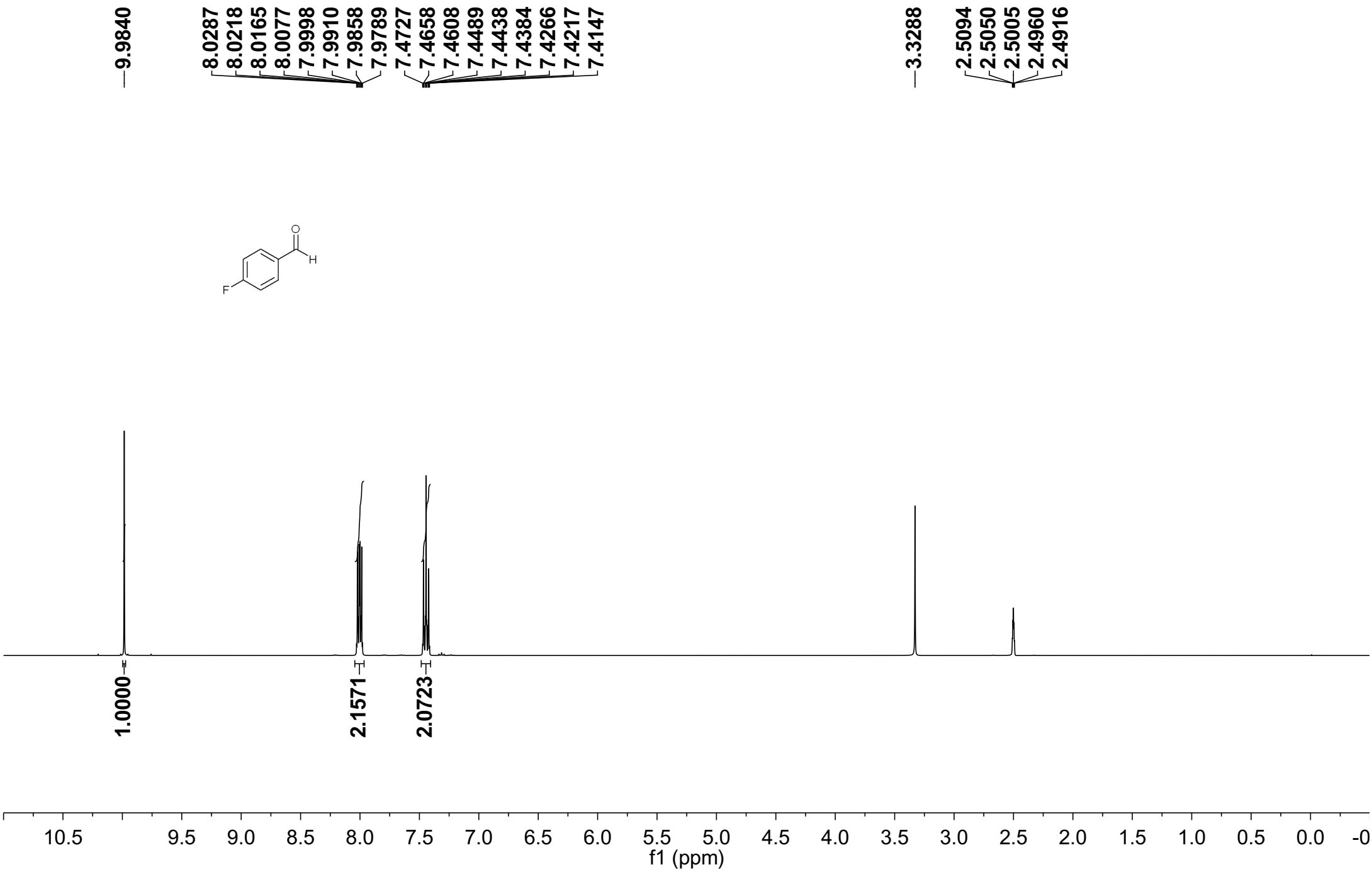
Compound 6b


10.3196
7.8705
7.8662
7.8515
7.8473
7.8361
7.8332
7.7116
7.7080
7.6924
7.6883
7.6743
7.6703
7.6577
7.6541
7.6162
7.5947
7.5717
7.5397
7.5232
7.5056
7.4882
2.5092
2.5048
2.5003
2.4958
2.4915
Cl
O
H
1.0000
1.0892
1.1163
1.0763
1.1341
10.5
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
f1 (ppm)

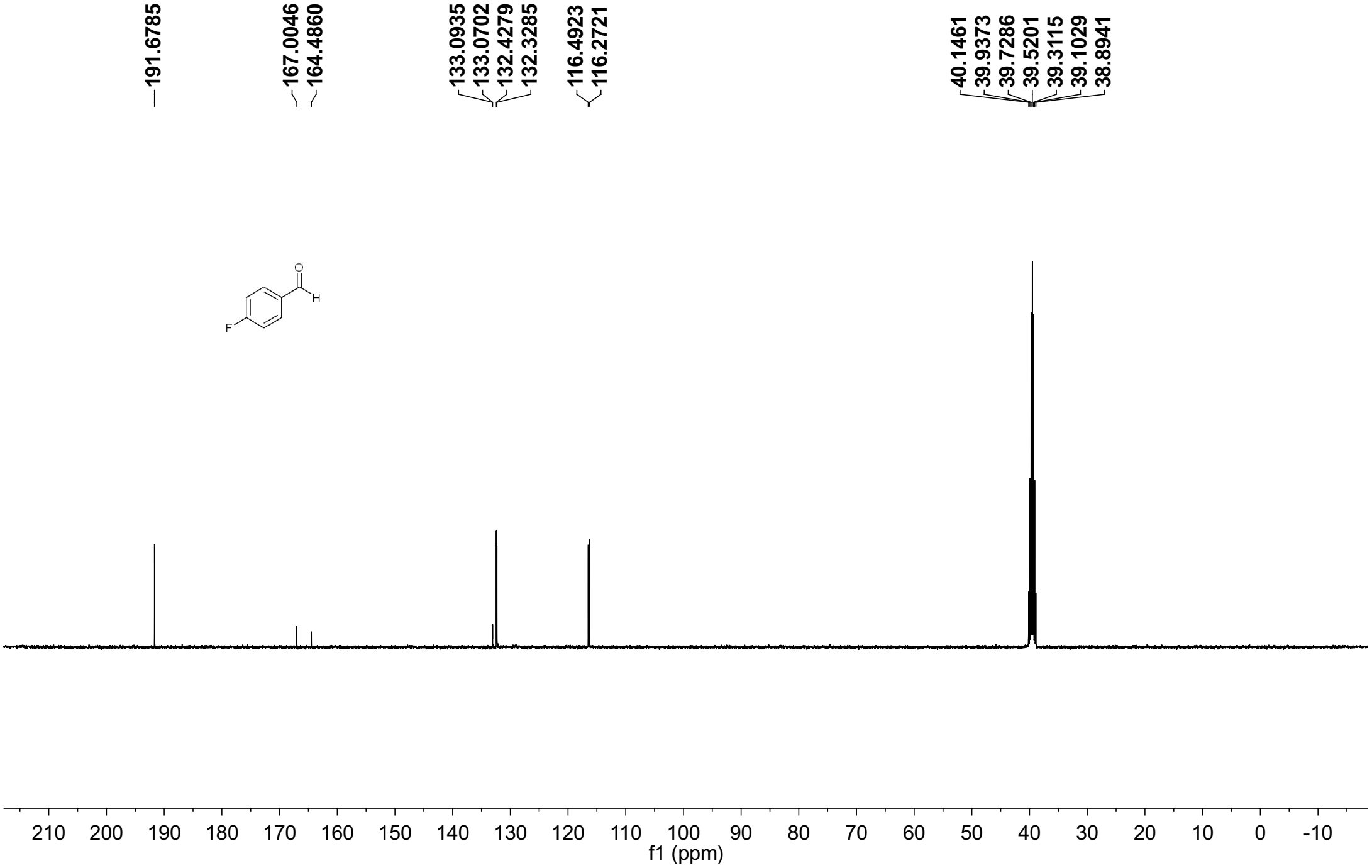
Compound 6b


189.6796
136.2725
135.6956
132.0116
130.6731
129.5881
127.8191
40.1457
39.9369
39.7283
39.5196
39.3110
39.1022
38.8933
Cl
O
H
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

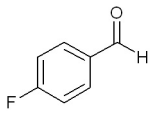
Compound 6c


9.9840
8.0287
8.0218
8.0165
8.0077
7.9998
7.9910
7.9858
7.9789
7.4727
7.4658
7.4608
7.4489
7.4438
7.4384
7.4266
7.4217
7.4147
3.3288
2.5094
2.5050
2.5005
2.4960
2.4916
O
H
F
1.0000
2.1571
2.0723
10.5
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
f1 (ppm)

Compound 6c

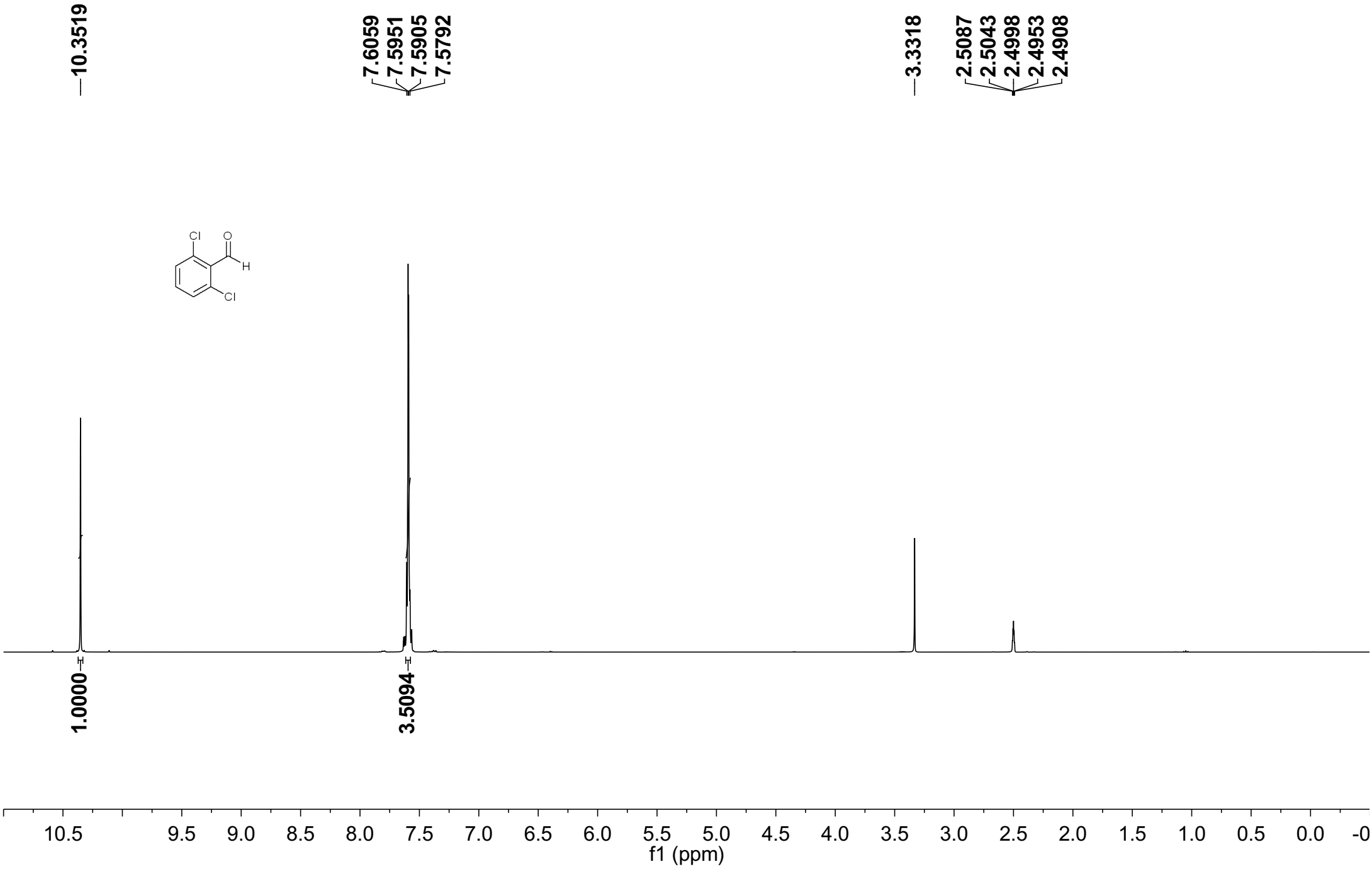

191.6785
167.0046
164.4860
133.0935
133.0702
132.4279
132.3285
116.4923
116.2721
40.1461
39.9373
39.7286
39.5201
39.3115
39.1029
38.8941
O
H
F
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Compound 6c

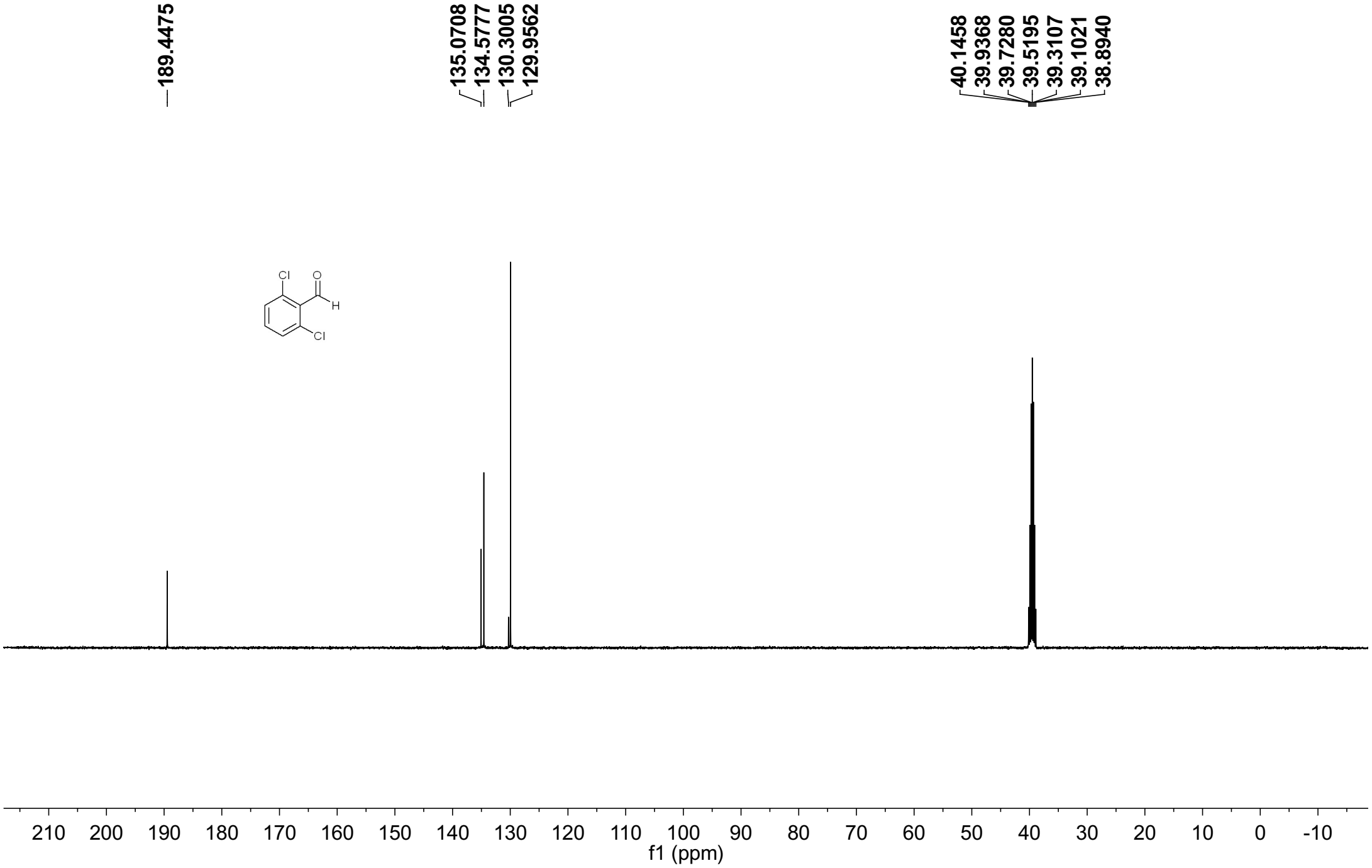

O
H
F

-103.5708
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
-210
f1 (ppm)

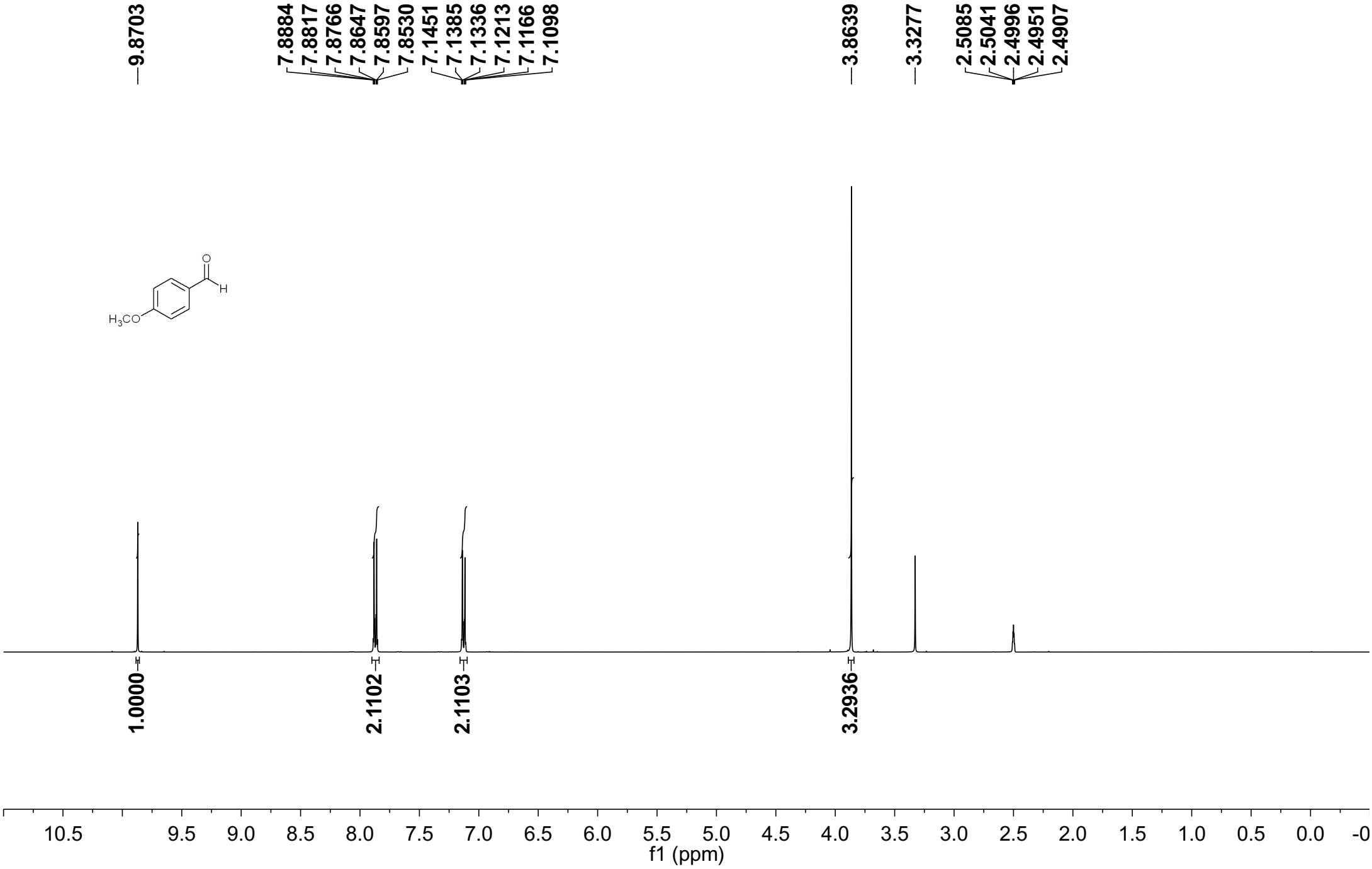
Compound 6d


10.3519
7.6059
7.5951
7.5905
7.5792
3.3318
2.5087
2.5043
2.4998
2.4953
2.4908
Cl
O
H
Cl
1.0000
3.5094
10.5
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
f1 (ppm)

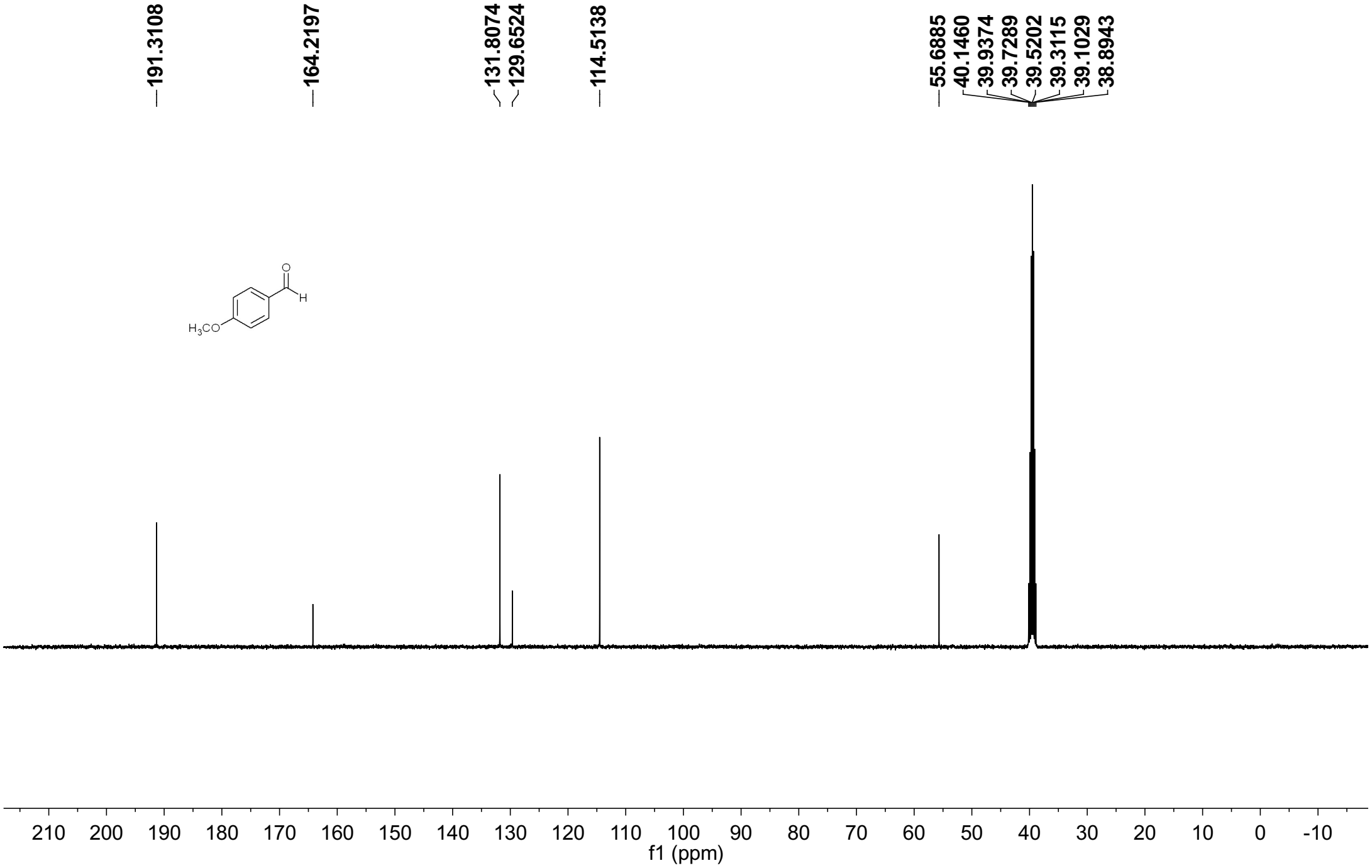
Compound 6d


189.4475
135.0708
134.5777
130.3005
129.9562
40.1458
39.9368
39.7280
39.5195
39.3107
39.1021
38.8940
Cl
O
H
Cl
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

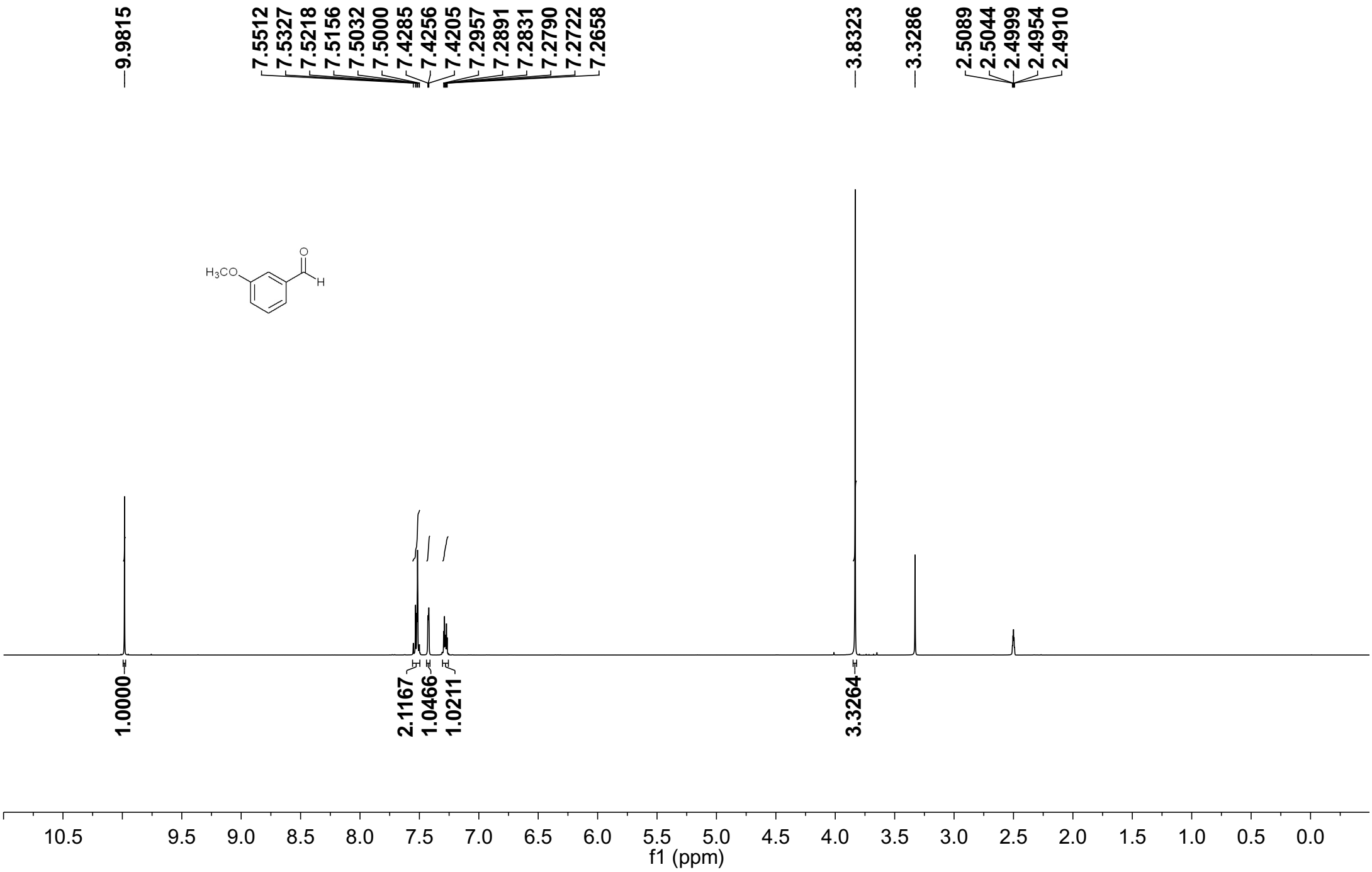
Compound 6e


9.8703
7.8884
7.8817
7.8766
7.8647
7.8597
7.8530
7.1451
7.1385
7.1336
7.1213
7.1166
7.1098
3.8639
3.3277
2.5085
2.5041
2.4996
2.4951
2.4907
O
H
H3CO
1.0000
2.1102
2.1103
3.2936
10.5
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
f1 (ppm)

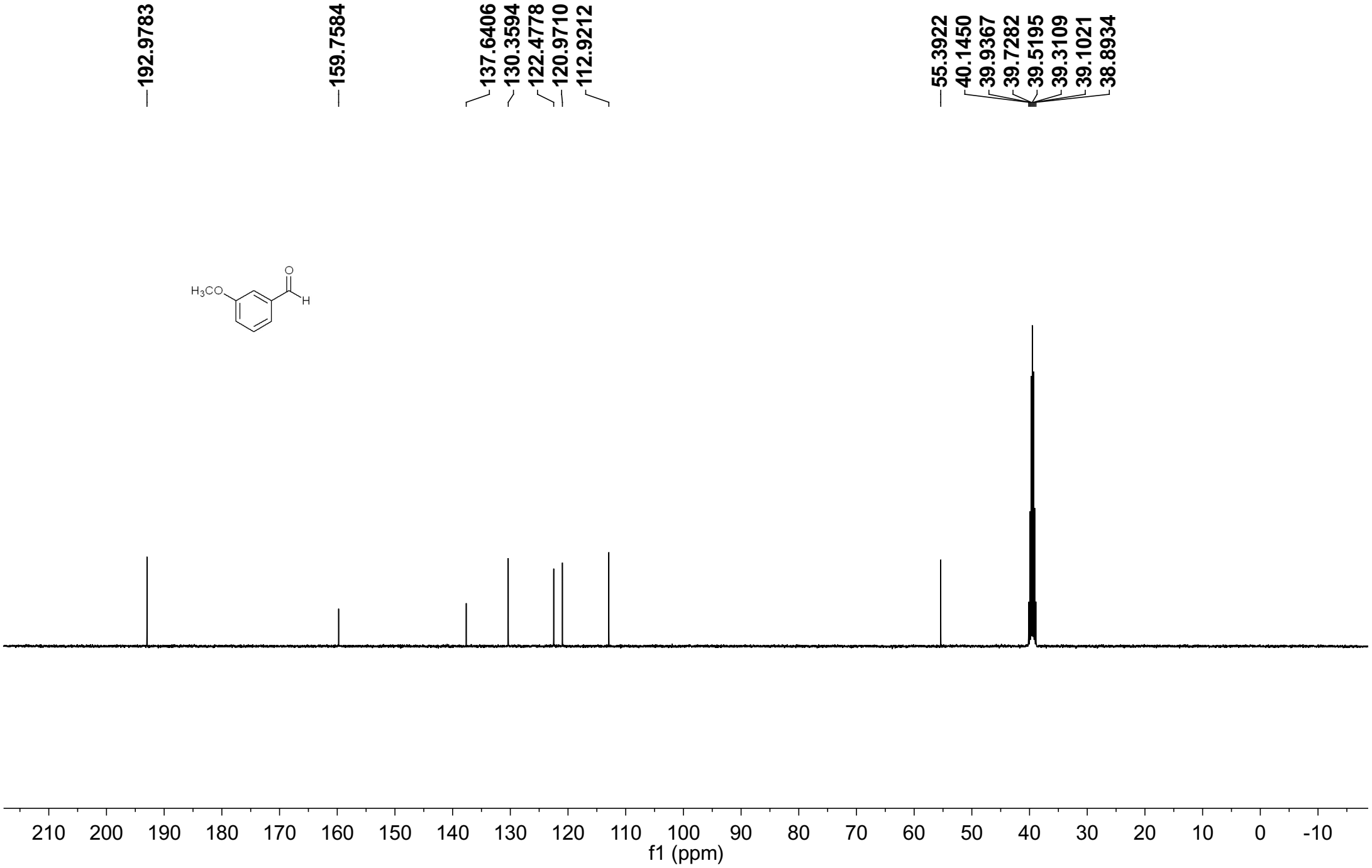
Compound 6e


191.3108
164.2197
131.8074
129.6524
114.5138
55.6885
40.1460
39.9374
39.7289
39.5202
39.3115
39.1029
38.8943
O
H
H3CO
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

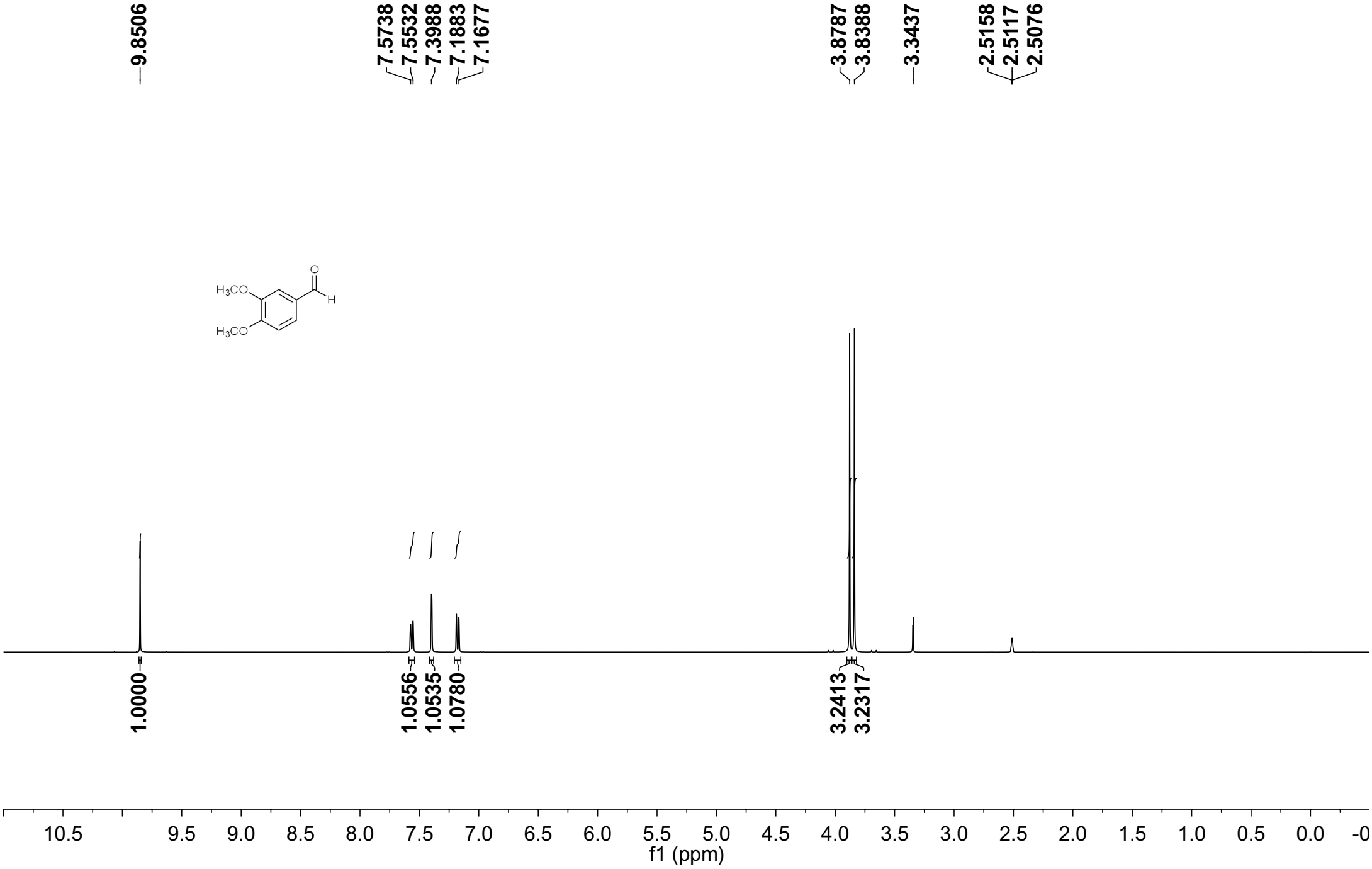
Compound 6f


9.9815
7.5512
7.5327
7.5218
7.5156
7.5032
7.5000
7.4285
7.4256
7.4205
7.2957
7.2891
7.2831
7.2790
7.2722
7.2658
3.8323
3.3286
2.5089
2.5044
2.4999
2.4954
2.4910
H3CO
O
H
1.0000
2.1167
1.0466
1.0211
3.3264
10.5
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)

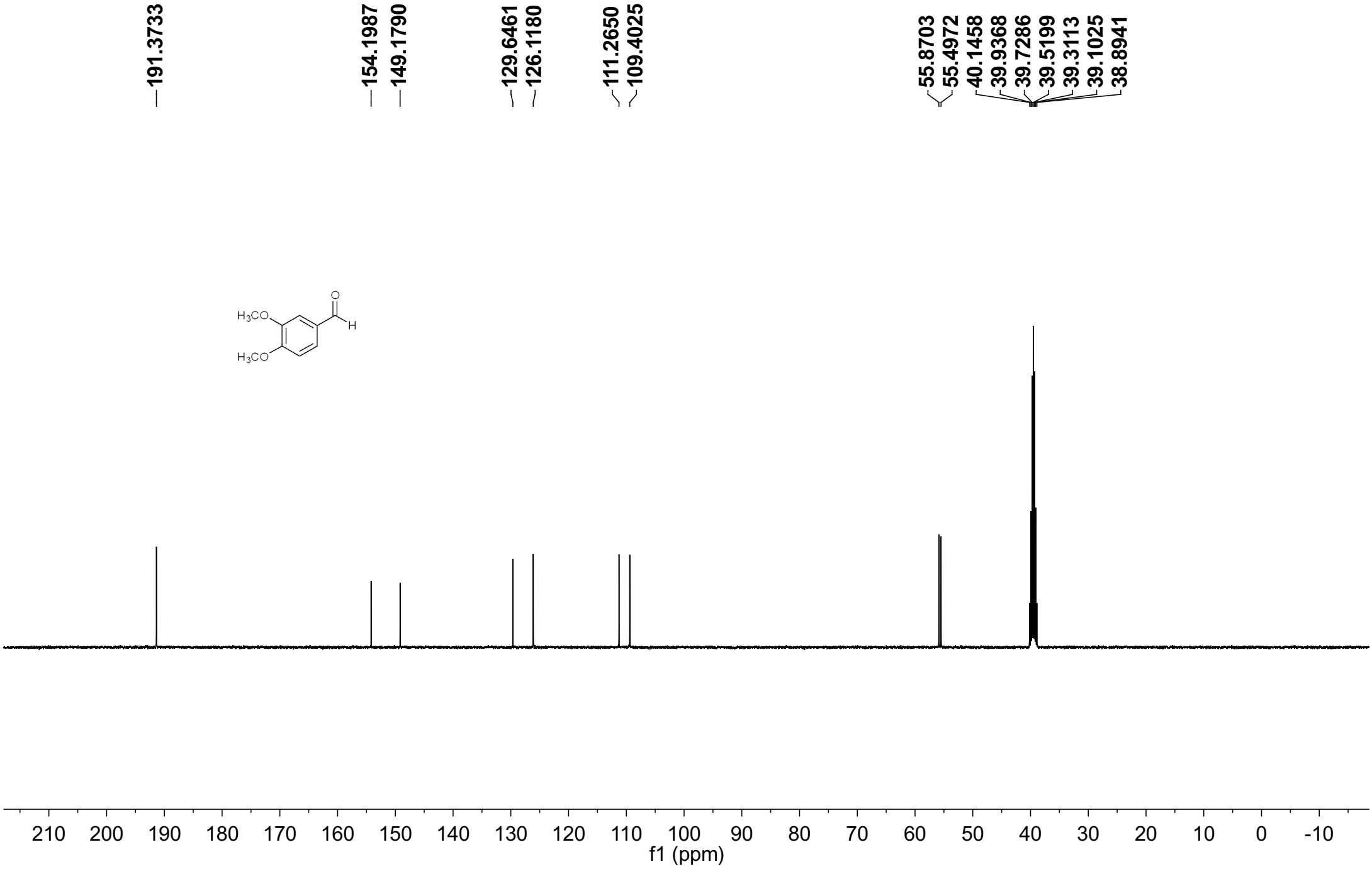
Compound 6f


192.9783
159.7584
137.6406
130.3594
122.4778
120.9710
112.9212
55.3922
40.1450
39.9367
39.7282
39.5195
39.3109
39.1021
38.8934
H3CO
O
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

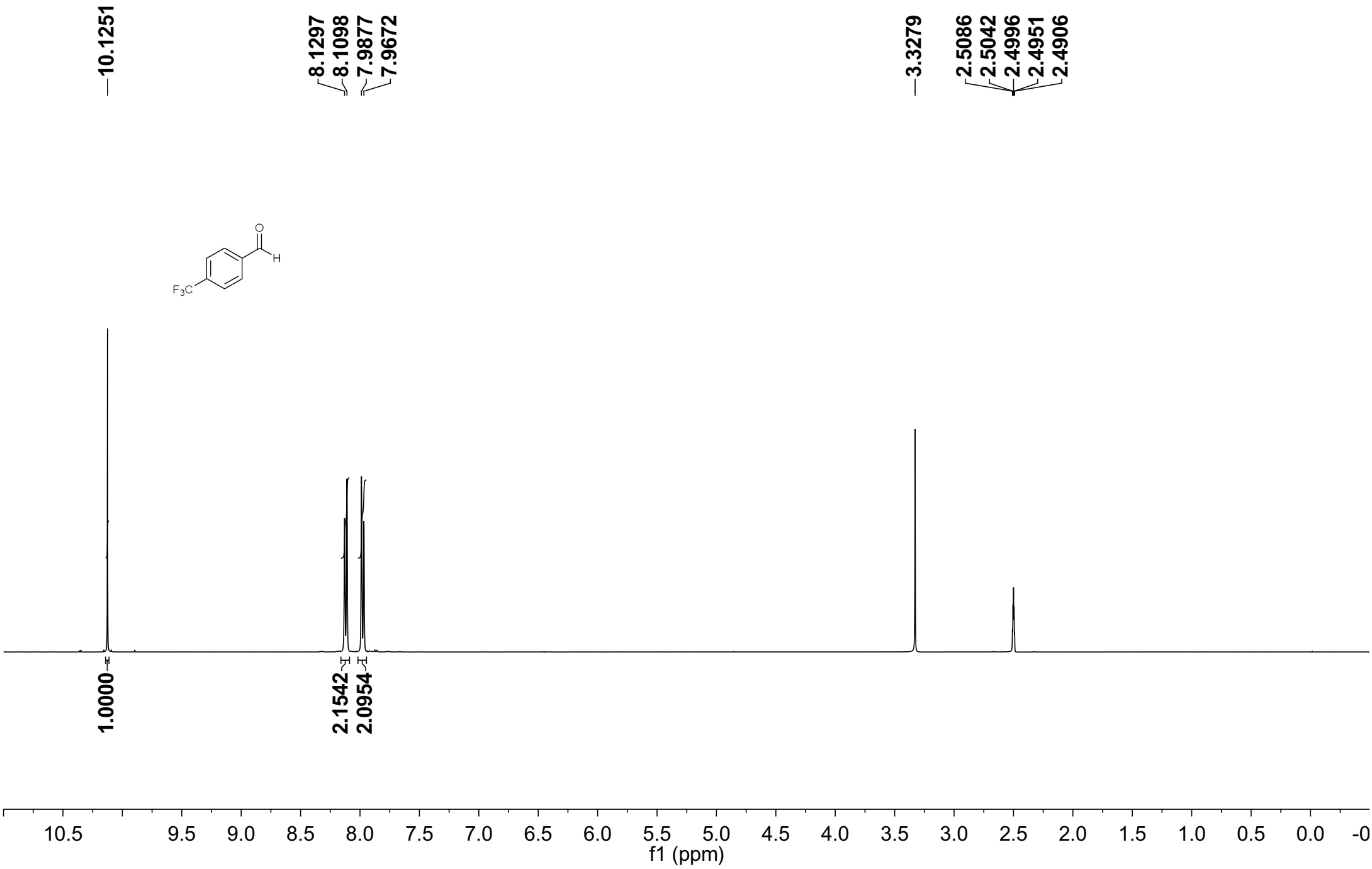
Compound 6g


9.8506
7.5738
7.5532
7.3988
7.1883
7.1677
3.8787
3.8388
3.3437
2.5158
2.5117
2.5076
H3CO
H3CO
O
H
1.0000
1.0556
1.0535
1.0780
3.2413
3.2317
10.5
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
f1 (ppm)

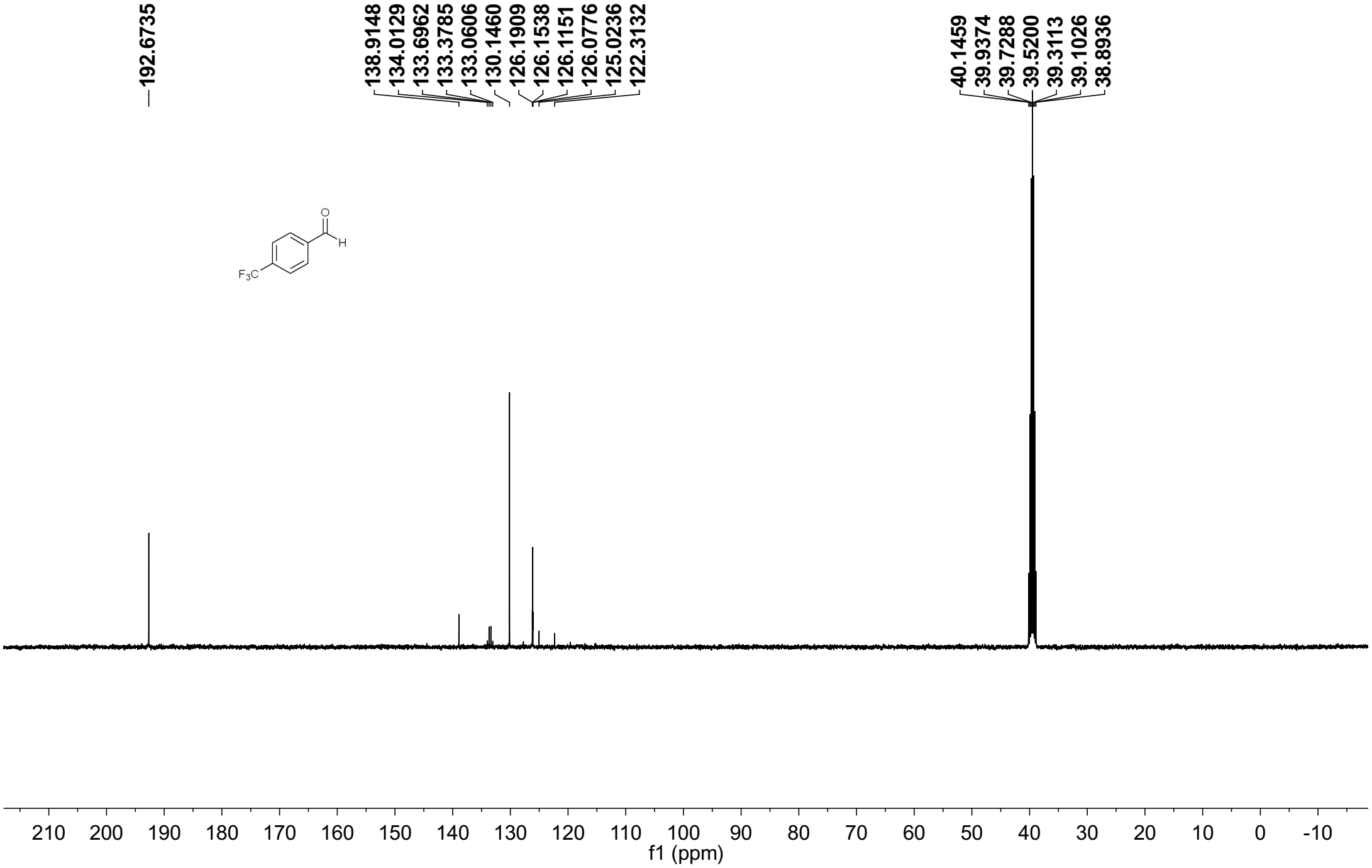
Compound 6g


191.3733
154.1987
149.1790
129.6461
126.1180
111.2650
109.4025
55.8703
55.4972
40.1458
39.9368
39.7286
39.5199
39.3113
39.1025
38.8941
H3CO
H3CO
O
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

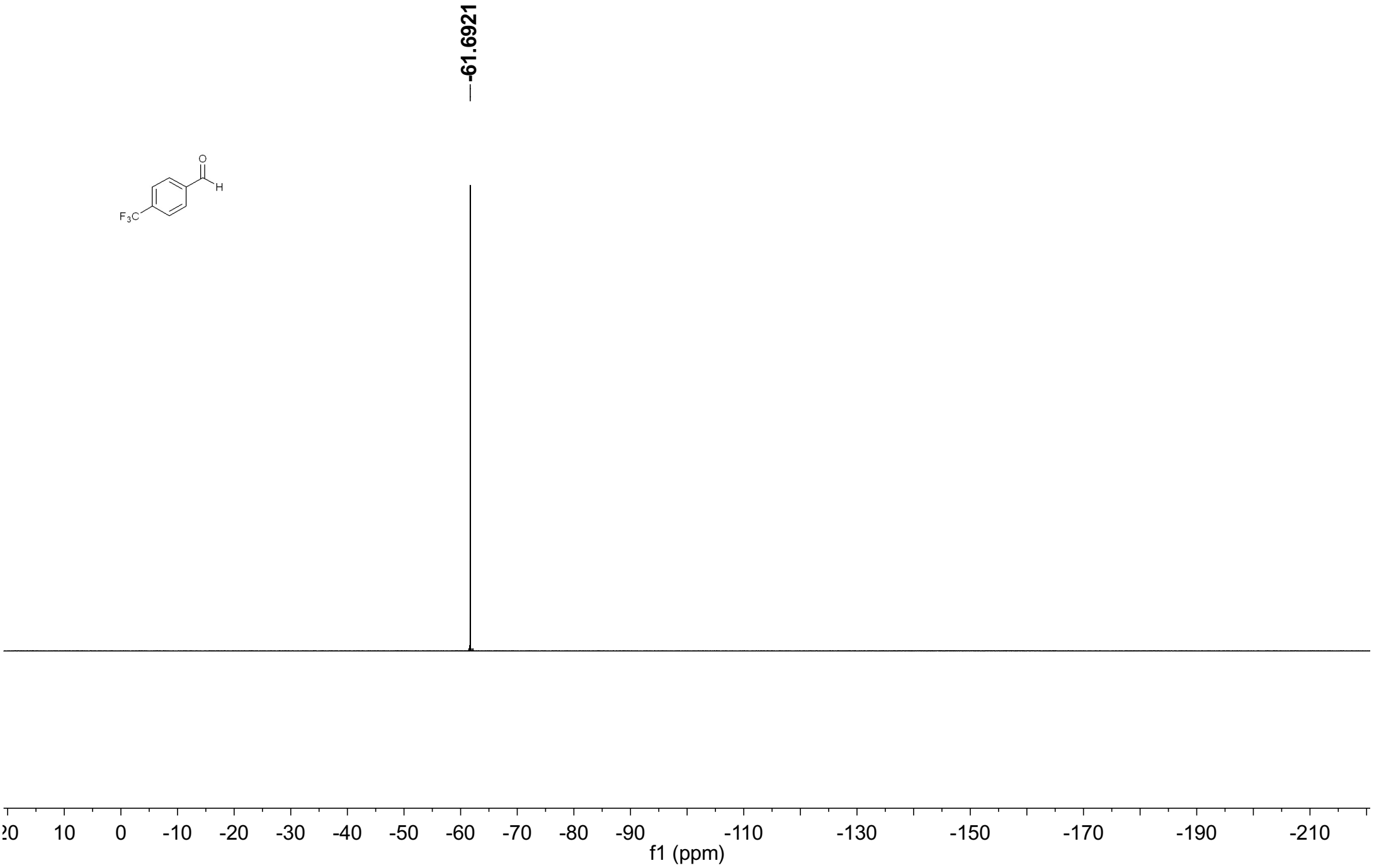
Compound 6h


10.1251
8.1297
8.1098
7.9877
7.9672
3.3279
2.5086
2.5042
2.4996
2.4951
2.4906
O
H
F3C
1.0000
2.1542
2.0954
10.5
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
f1 (ppm)

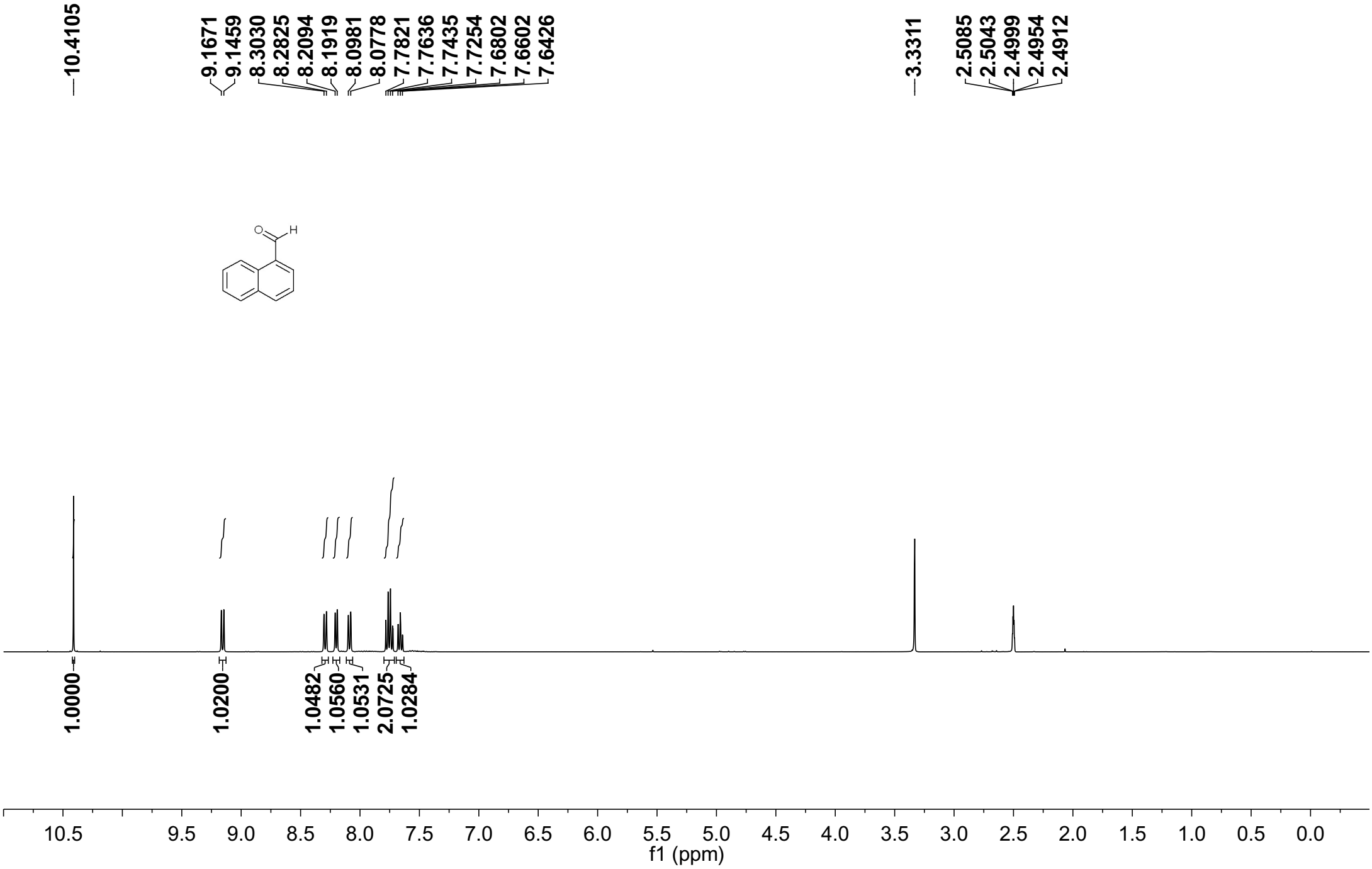
Compound 6h


192.6735
138.9148
134.0129
133.6962
133.3785
133.0606
130.1460
126.1909
126.1538
126.1151
126.0776
125.0236
122.3132
40.1459
39.9374
39.7288
39.5200
39.3113
39.1026
38.8936
O
H
F3C
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

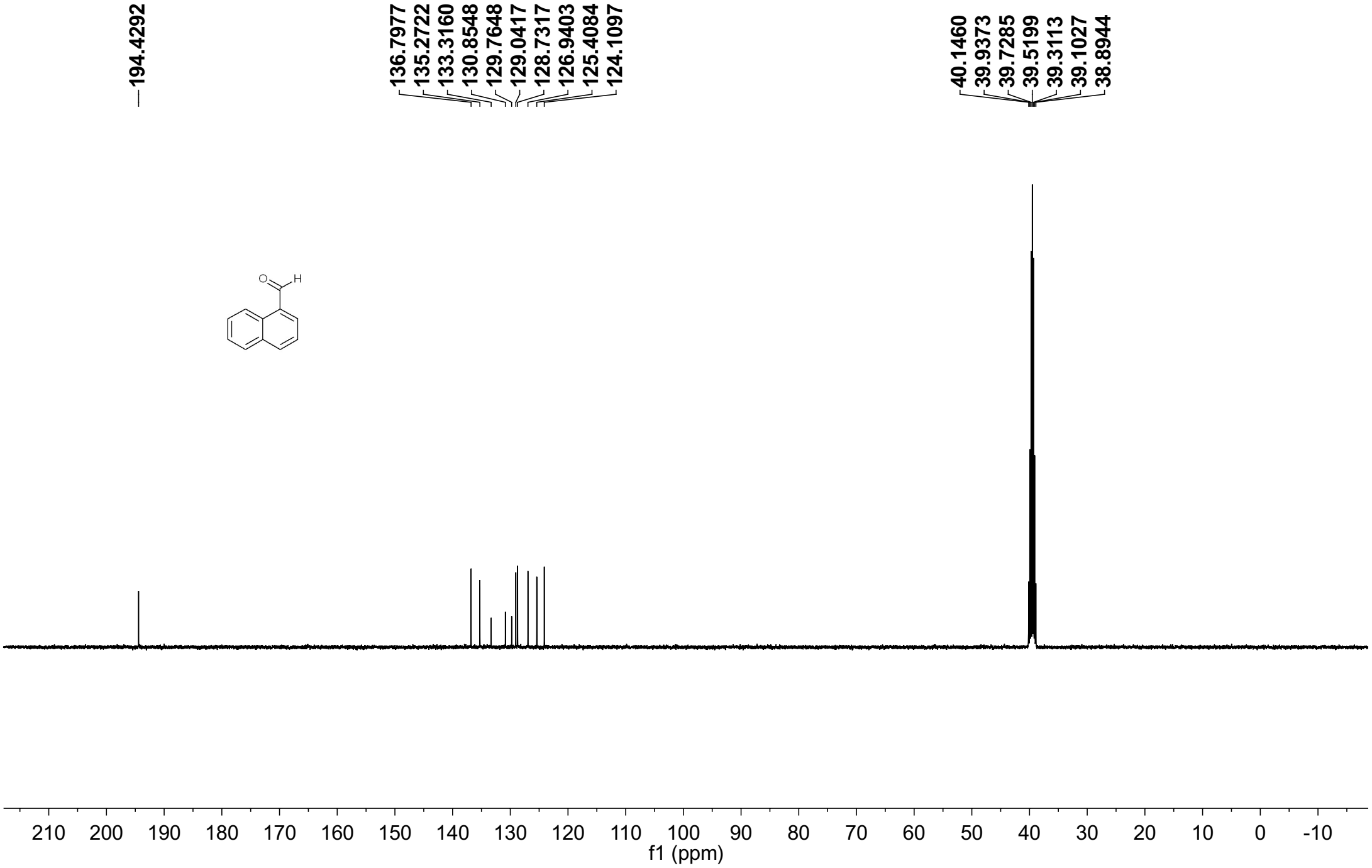
Compound 6h


-61.6921
O
H
F3C
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-110
-130
-150
-170
-190
-210
f1 (ppm)

Compound 6i


10.4105
9.1671
9.1459
8.3030
8.2825
8.2094
8.1919
8.0981
8.0778
7.7821
7.7636
7.7435
7.7254
7.6802
7.6602
7.6426
3.3311
2.5085
2.5043
2.4999
2.4954
2.4912
O
H
1.0000
1.0200
1.0482
1.0560
1.0531
2.0725
1.0284
10.5
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)

Compound 6i


194.4292
136.7977
135.2722
133.3160
130.8548
129.7648
129.0417
128.7317
126.9403
125.4084
124.1097
40.1460
39.9373
39.7285
39.5199
39.3113
39.1027
38.8944
O
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)