Supporting Information

Electrochemical hydrocarboxylation of enol derivatives with CO_2: access to β-acetoxycarboxylic acids

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General materials and methods

^{1}H, ^{13}C, ^{19}F NMR spectra were recorded on Bruker AVneo-300 and Bruker Fourier 300 HD spectrometers (300.13, 75.48, 282.5 MHz, respectively) in $CDCl_3$ and DMSO-d_6. Chemical shifts were reported in parts per million (ppm), and the residual solvent peak was used as an internal reference: ^{1}H ($CDCl_3$ δ = 7.26 ppm), ^{13}C ($CDCl_3$ δ = 77.16 ppm), ^{1}H (DMSO-d_6 δ = 2.50 ppm), ^{13}C (DMSO-d_6 δ = 39.52 ppm). Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), quintet, m (multiplet), brs (broad singlet).[1] 1,4-dinitrobenzene has been used as a standard for determining ^{1}H NMR yields.

High resolution mass spectra (HR-MS) were measured on a Bruker micrOTOF II instrument using electrospray ionization (ESI).[2] The measurements were performed in a positive ion mode (interface capillary voltage – 4500 V); mass range from m/z 50 to m/z 3000 Da; external calibration with Electrospray Calibrant Solution (Fluka). A syringe injection was used for all acetonitrile solutions (flow rate 3 μL/min). Nitrogen was applied as a dry gas; interface temperature was set at 180 °C.

IR spectra were recorded on a «InfraLUM FT-08» by Lumex. Method of measurement is ATR.

Elemental analyzes were performed on a PerkinElmer 2400 CHN analyzer. Method of measurement is automatic.

Acetophenone, triethanolamine, Et_3N, K_2CO_3, pyridine, NaOH, monoethanolamine, imidazole, 1,3-bis(methylamino)propan-2-ol, N,N,N',N'-tetraethylmethanediamine, $LiClO_4$, *n*-Bu_4NClO_4, *n*-Bu_4NBF_4, *n*-Bu_4NBr, *n*-Bu_4NI, phenanthrene, 1-methylnaphthalene, anthracene, phenantroline, graphene, p-terphenyl, LiOH, PEG, o-phenylenediamine, methyl iodide were purchased from commercial sources and were used as is. All solvents were distilled according to standard procedures.[3]

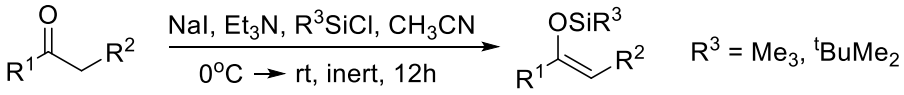
Electrochemical synthesis was carried out using the IKA ElectraSyn 2.0 cell. During the work we used electrodes from the ElectraSyn Starter Kit. At the end of the experiment, all electrodes were washed with water, acetone and then dried. In the case of carbon and platinum electrodes, they were placed in a 1M solution of HCl and allowed to soak for 2 hours, washed with water, acetone and dried in an oven overnight.

The TLC analysis was carried out on standard silica gel chromatography plates (DC-Fertigfolien ALUGRAMR Xtra SIL G/UV254). Column chromatography was performed using silica gel (0.060-0.200 mm, 60 A, CAS 7631-86-9, Acros).

Synthesis of the starting enol derivatives

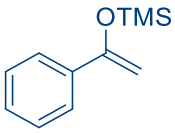
Silyl enol ethers.

Starting silyl enol ethers **S2, S3** were prepared according to literature.[4]



NaI (1.4 mmol, 210.0 mg, 1.4 eq.) was placed in a round bottom flask and dried under vacuum (15-20 mmHg) using a heat gun (gun temperature 100-150 °C) for 5 min. After cooling to room temperature, the flask was filled with argon. Then, CH_3CN (1 mL), ketone (1.0 mmol, 1.0 eq.), and Et_3N (1.5 mmol, 151.8 mg, 1.5 eq.) were successively added. The mixture was cooled with an ice/water bath, and R^3SiCl (1.3 mmol, 1.3 eq.) was added at 0 °C. The cooling bath was removed, and the mixture was stirred for 12 h at room temperature. Then, volatile components were evaporated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 40–45 °C). The solid residue was washed with hexane (3×15 mL) (the hexane layers were decanted and filtered through a cotton plug). The combined filtrates were concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20–25 °C), furnishing the silyl enol ether which was used without purification.

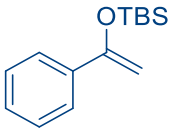
Trimethyl((1-phenylvinyl)oxy)silane, S2[4]



^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.74 – 7.70 (m, 2H), 7.46 – 7.37 (m, 3H), 5.05 – 5.03 (m, 1H), 4.57 – 4.55 (m, 1H), 0.40 (s, 9H).

$^{13}C\{^1H\}$ NMR (75.48 MHz, $CDCl_3$, δ): 155.8, 137.6, 128.2, 128.1 (2C), 125.3 (2C), 91.0, 0.1 (3C).

***Tert*-butyldimethyl((1-phenylvinyl)oxy)silane, S3**[5]

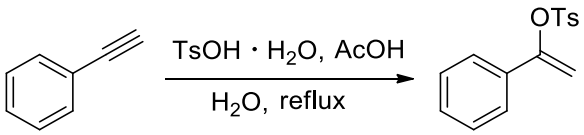


^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.68 – 7.59 (m, 2H), 7.42 – 7.27 (m, 3H), 4.91 (d, *J* = 1.7 Hz, 1H), 4.44 (d, *J* = 1.7 Hz, 1H), 1.03 (s, 9H), 0.23 (s, 6H).

$^{13}C\{^1H\}$ NMR (75.48 MHz, $CDCl_3$, δ): 156.1, 138.0, 128.3, 128.2 (2C), 125.4 (2C), 91.0, 26.1 (3C), 18.5, -4.5 (2C).

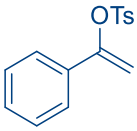
1-phenylvinyl 4-methylbenzenesulfonate.

Starting 1-phenylvinyl 4-methylbenzenesulfonate **S4** was prepared according to literature.[6]



The corresponding alkyne (19.6 mmol, 2.00 g) was added to a solution of *p*-toluenesulfonic acid monohydrate (19.6 mmol, 3.72 g), acetic acid (9.8 mL) and H_2O (3.9 mL). The reaction mixture was stirred and refluxed at 60 ^oC for 9 hours. The mixture was poured into saturated $NaHCO_3$ (70 mL), and extracted with CH_2Cl_2 (3 x 30 mL). The organic layer was dried over Na_2SO_4 and concentrated in vacuo. The crude mixture was purified by column chromatography on silica gel. (PE:EA, 20:1).

1-Phenylvinyl 4-methylbenzenesulfonate, S4[6]



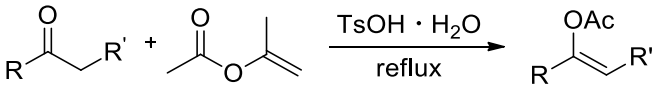
^1H NMR (300.13 MHz, $CDCl_3$, δ): 7.86 – 7.77 (m, 2H), 7.49 – 7.38 (m, 2H), 7.33 – 7.23 (m, 5H), 5.39 (d, *J* = 2.9 Hz, 1H), 5.10 (d, *J* = 2.9 Hz, 1H), 2.41 (s, 3H).

$^{13}C\{^1H\}$ NMR (75.48 MHz, $CDCl_3$, δ): 153.0, 145.3, 133.5, 133.3, 129.8 (2C), 129.4, 128.6 (2C), 128.5 (2C), 125.6 (2C), 103.1, 21.8.

Enol acetates.

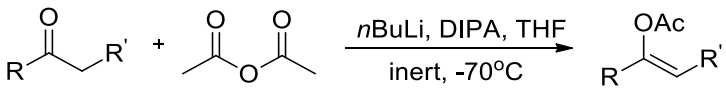
Starting enol acetates **1a-v** were prepared according to literature. [7-8]

Method 1.[7]



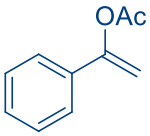
To a mixture of ketone (42.5 mmol) and 2-propenyl acetate (0.213 mol, 5 eq.) was added *p*-toluenesulfonic acid monohydrate (3.925 mmol, 0.09 eq.). The resulting mixture was refluxed in a 100 ml flask equipped with a condenser and a drying tube. The procedure of reaction was controlled by TLC. After achieving the optimal reaction conversion, reaction solution was cooled to room temperature, and the solvent was evaporated in vacuo. Ether was added (100 ml), and the resulting solution was washed with water (3 x 50 ml) and dried over Na_2SO_4. The solvent was evaporated in vacuo to give a dark orange/red oily residue. This residue was purified by column chromatography on silica gel to give target enol acetate.

Method 2. [8]



*n*Butyllithium (9.6 mmol) was added to a solution of diisopropylamine (9.6 mmol) in dried THF (40 mL) in a flame-dried round-bottom flask under inert atmosphere at −70 °C. The mixture was stirred for 30 min, and then ketone (8 mmol) was added. The resulting mixture was stirred for 45 min, and then acetic anhydride (16 mmol) was added. The reaction was stirred 30 min at −70 °C and another 30 min at room temperature. The mixture was poured into saturated $NaHCO_3$ (100 mL), and extracted thrice with EtOAc (60 mL). The combined organic layers were washed with 10mL brine and dried over $MgSO_4$, and the solvent was removed under reduced pressure. The crude mixture was purified by column chromatography on silica gel.

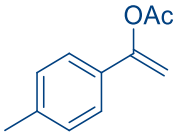
1-Phenylvinyl acetate, 1a [9]



^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.51 – 7.43 (m, 2H), 7.40 – 7.30 (m, 3H), 5.48 (d, *J* = 2.0 Hz, 1H), 5.02 (d, *J* = 2.0 Hz, 1H), 2.28 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 169.2, 153.1, 134.4, 129.1, 128.6 (2C), 125.0 (2C), 102.2, 21.1.

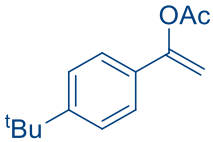
1-(*p*-Tolyl)vinyl acetate, 1b [10]



^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.39 – 7.34 (m, 2H), 7.18 – 7.13 (m, 3H), 5.48 (d, *J* = 2.2 Hz, 1H), 4.98 (d, *J* = 2.2 Hz, 1H), 2.35 (s, 3H), 2.27 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 169.2, 153.2, 139.1, 131.6, 129.3 (2C), 124.9 (2C), 101.3, 21.3, 21.1.

1-(4-(*Tert*-butyl)phenyl)vinyl acetate, 1c

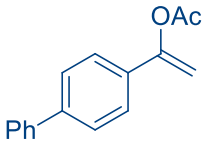


^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.43 – 7.35 (m, 4H), 5.43 (d, *J* = 2.2 Hz, 1H), 4.99 (d, *J* = 2.2 Hz, 1H), 2.28 (s, 3H), 1.32 (s, 9H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 169.3, 153.1, 152.2, 131.5, 125.6 (2C), 124.7 (2C), 101.5, 34.8, 31.3 (3C), 21.1.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{14}H_{18}O_2Na]^+$ 241.1199; Found 241.1203.

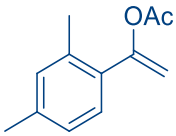
1-([1,1'-Biphenyl]-4-yl)vinyl acetate, 1d[11]



^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.62 – 7.51 (m, 6H), 7.49 – 7.35 (m, 3H), 5.53 (d, *J* = 2.3 Hz, 1H), 5.03 (d, *J* = 2.3 Hz, 1H), 2.31 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 169.3, 152.8, 141.9, 140.5, 133.3, 129.0 (2C), 127.7, 127.4 (2C), 127.2 (2C), 125.4 (2C), 102.3, 21.2.

1-(2,4-Dimethylphenyl)vinyl acetate, 1e

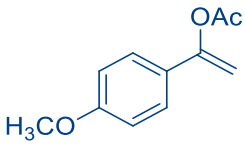


^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.29 – 7.24 (m, 1H), 7.02 – 6.95 (m, 2H), 5.15 (d, *J* = 1.6 Hz, 1H), 5.00 (d, *J* = 1.6 Hz, 1H), 2.38 (s, 3H), 2.31 (s, 3H), 2.13 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 168.9, 154.0, 138.8, 135.9, 132.7, 129.2, 126.5,126.5, 105.4, 21.3, 21.2, 20.4.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{12}H_{14}O_2NH_4]^+$ 208.1332; Found 208.1338.

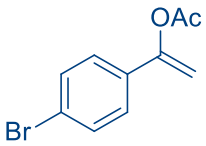
1-(4-Methoxyphenyl)vinyl acetate, 1f[9]



^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.43 – 7.36 (m, 2H), 6.90 – 6.84 (m, 2H), 5.35 (d, *J* = 2.2 Hz, 1H), 4.91 (d, *J* = 2.2 Hz, 1H), 3.81 (s, 3H), 2.26 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 169.3, 160.3, 152.9, 129.3, 126.4 (2C), 114.0 (2C), 100.4, 55.5, 21.1.

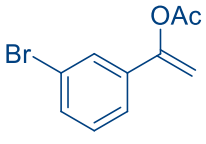
1-(4-Bromophenyl)vinyl acetate, 1r[12]



^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.51 – 7.44 (m, 2H), 7.36 – 7.29 (m, 2H), 5.45 (d, *J* = 2.4 Hz, 1H), 5.05 (d, *J* = 2.4 Hz, 1H), 2.27 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 169.0, 152.1, 133.4, 131.8 (2C), 126.6 (2C), 123.2, 102.9, 21.1.

1-(3-Bromophenyl)vinyl acetate, 1s

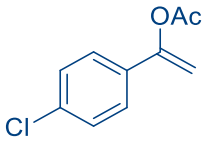


^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.61 – 7.57 (m, 1H), 7.48 – 7.36 (m, 2H), 7.25 – 7.17 (m, 1H), 5.47 (d, *J* = 2.4 Hz, 1H), 5.07 (d, *J* = 2.4 Hz, 1H), 2.28 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 169.0, 151.6, 136.6, 132.0, 130.2, 128.1, 123.7, 122.9, 103.6, 21.1.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{10}H_9BrO_2Na]^+$ 262.9678; Found 262.9683.

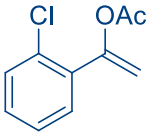
1-(4-Chlorophenyl)vinyl acetate, 1u[13]



^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.42 – 7.36 (m, 2H), 7.34 – 7.28 (m, 2H), 5.45 (d, *J* = 2.3 Hz, 1H), 5.05 (d, *J* = 2.3 Hz, 1H), 2.27 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 169.0, 152.1, 135.0, 133.0, 128.9 (2C), 126.3 (2C), 102.8, 21.1.

1-(2-Chlorophenyl)vinyl acetate, 1v

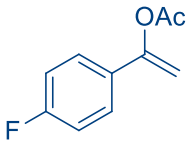


^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.48 – 7.43 (m, 1H), 7.40 – 7.35 (m,1H), 7.28 – 7.23 (m, 2H), 5.27 (d, *J* = 1.9 Hz, 1H), 5.23 (d, *J* = 1.9 Hz, 1H), 2.17 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ):168.9, 151.5, 134.6, 132.2, 130.9, 130.2, 130.0, 126.8, 107.2, 21.1.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{10}H_9ClO_2Na]^+$ 219.0183; Found 219.0188.

1-(4-Fluorophenyl)vinyl acetate, 1g[14]

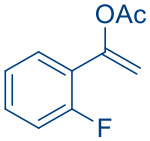


^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.48 – 7.40 (m, 2H), 7.07 – 6.98 (m, 2H), 5.40 (d, *J* = 2.2 Hz, 1H), 5.01 (d, *J* = 2.2 Hz, 1H), 2.27 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 169.1, 163.2 (d, $^{1}J_{CF}$ = 248.8 Hz), 152.2, 131.1 (d,$^{3}J_{CF}$ = 9.3 Hz),130.7 (d, $^{4}J_{CF}$ = 3.4 Hz), 126.9 (d, $^{3}J_{CF}$ = 8.3 Hz, 2C), 115.7 (d, $^{2}J_{CF}$ = 21.9 Hz, 2C), 102.1 (d, $^{6}J_{CF}$ = 1.6 Hz), 21.1.

^{19}F NMR (282.5 MHz, $CDCl_3$, δ): -112.35 (tt, *J* = 8.4, 5.2 Hz).

1-(2-Fluorophenyl)vinyl acetate, 1h



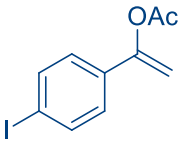
^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.41 – 7.24 (m, 2H), 7.16 – 7.02 (m, 2H), 7.39 (td, *J* = 7.7, 1.7 Hz, 1H), 7.32 (tdd, *J* = 7.6, 5.1, 1.8 Hz, 1H), 7.15 (td, *J* = 7.6. 1.2 Hz, 1H), 7.10 (ddd, *J* = 11.5, 8.1, 0.9 Hz, 1H), 5.56 (d, *J* = 1.9 Hz, 1H), 5.25 (t, *J* = 1.9 Hz, 1H), 2.24 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 169.1, 160.0 (d, $^{1}J_{CF}$ = 251.9 Hz), 148.0 (d, $^{3}J_{CF}$ = 3.3 Hz), 130.4 (d,$^{3}J_{CF}$ = 8.6 Hz), 128.1 (d, $^{3}J_{CF}$ = 2.7 Hz), 124.2 (d,$^{4}J_{CF}$ = 3.7Hz), 122.6 (d,$^{2}J_{CF}$ = 11.2 Hz), 116.5 (d, $^{2}J_{CF}$ = 22.7 Hz), 107.5 (d, $^{4}J_{CF}$ = 9.0 Hz), 21.0.

^{19}F NMR (282.5 MHz, $CDCl_3$, δ): -113.42 (dddd, *J* = 11.3, 7.2, 5.1, 1.7 Hz)

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{10}H_9FO_2Na]^+$ 203.0479; Found 203.0475.

1-(4-Iodophenyl)vinyl acetate, 1t

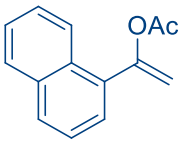


^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.71 – 7.64 (m, 2H), 7.23 – 7.15 (m,2H), 5.47 (d, *J* = 2.3 Hz, 1H), 5.04 (d, *J* = 2.3 Hz, 1H), 2.26 (s, 3H).

^{f13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 169.1, 152.2, 137.8 (2C), 134.0, 126.7 (2C), 103.0, 94.9, 21.1.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{10}H_9IO_2Na]^+$ 310.9539; Found 310.9534.

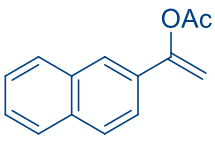
1-(Naphthalen-1-yl)vinyl acetate, 1i[15]



^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 8.30 – 8.20 (m, 1H), 7.91 – 7.79 (m, 2H), 7.65 – 7.59 (m, 1H), 7.57 – 7.41 (m, 3H), 5.36 (d, *J* = 1.5 Hz, 1H), 5.24 (d, *J* = 1.5 Hz, 1H), 2.13 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 169.1, 153.5, 133.7, 133.7, 130.9, 129.6, 128.5, 127.4, 126.6, 126.1, 125.6, 125.2, 107.0, 21.2.

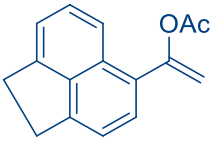
1-(Naphthalen-2-yl)vinyl acetate, 1j[9]



^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.93 – 7.77 (m, 4H), 7.66 – 7.56 (m, 1H), 7.54 – 7.45 (m, 2H), 5.62 (d, *J* = 2.3 Hz, 1H), 5.13 (d, *J* = 2.3 Hz, 1H), 2.35 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 169.3, 153.1, 133.6, 133.2, 131.7, 128.6, 128.5, 127.8, 126.8, 126.6, 124.2, 122.8, 102.8, 21.2.

1-(1,2-Dihydroacenaphthylen-5-yl)vinyl acetate, 1k

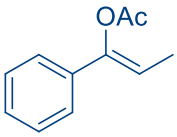


^{1}H NMR (300.13 MHz, DMSO-d_6, δ): 7.91 – 7.84 (m, 1H), 7.59 – 7.46 (m, 2H), 7.37 – 7.26 (m, 2H), 5.33 (d, *J* = 1.6 Hz, 1H), 5.27 (d, *J* = 1.6 Hz, 1H), 3.37 – 3.31 (m, 4H), 2.15 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 169.1, 153.5, 147.8, 146.4, 139.5, 129.1, 129.0, 128.6, 128.1, 120.7, 119.7, 118.9, 105.5, 30.6, 30.3, 21.2.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{16}H_{14}O_2Na]^+$ 261.0886; Found 261.0888.

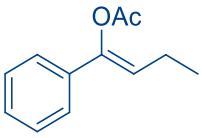
(*Z*)-1-Phenylprop-1-en-1-yl acetate, 1l[7]



^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.43 – 7.23 (m, 5H), 5.90 (q, *J* = 7.0 Hz, 1H), 2.31 (s, 3H), 1.73 (d, *J* = 7.0 Hz, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 168.7, 147.1, 135.1, 128.6 (2C), 128.1, 124.4 (2C), 112.8, 20.7, 11.7.

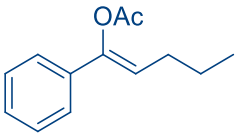
(*Z*)-1-Phenylbut-1-en-1-yl acetate, 1m[8]



^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.44 – 7.22 (m, 5H), 5.82 (t, *J* = 7.5 Hz, 1H), 2.29 (s, 3H), 2.15 (quintet, 2H), 1.08 (t, *J* = 7.5 Hz, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 168.9, 145.7, 135.1, 128.6 (2C), 128.2, 124.5 (2C), 119.9, 20.8, 19.8, 13.6.

(*Z*)-1-Phenylpent-1-en-1-yl acetate, 1n

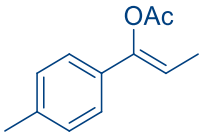


^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.44 – 7.23 (m, 5H), 5.83 (t, *J* = 7.4 Hz, 1H), 2.30 (s, 3H), 2.11 (q, *J* = 7.4 Hz, 2H), 1.50 (quintet, *J* = 7.4 Hz, 2H), 0.97 (t, *J* = 7.4 Hz, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 168.9, 146.3, 135.2, 128.6 (2C), 128.1, 124.5 (2C), 118.3, 28.4, 22.3, 20.7, 14.0.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{13}H_{16}O_2Na]^+$ 227.1043; Found 227.1042.

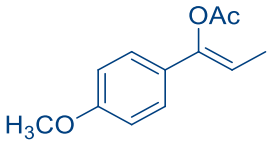
(*Z*)-1-(*p*-Tolyl)prop-1-en-1-yl acetate, 1o[8]



^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.31 – 7.24 (m, 2H), 7.17 – 7.05 (m, 2H), 5.84 (q, *J* = 7.0 Hz, 1H), 2.33 (s, 3H), 2.29 (s, 3H), 1.70 (d, *J* = 7.0 Hz, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 168.8, 147.2, 138.0, 132.4, 129.3 (2C), 124.3 (2C), 111.8, 21.3, 20.8, 11.6.

(*Z*)-1-(4-Methoxyphenyl)prop-1-en-1-yl acetate, 1p[8]



^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.43 – 7.28 (m, 2H), 6.90 – 6.79 (m, 2H), 5.76 (q, *J* = 7.0 Hz, 1H), 3.80 (s, 3H), 2.30 (s, 3H), 1.69 (d, *J* = 7.0 Hz, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 168.9, 159.6, 146.8, 127.9, 125.7 (2C), 114.0 (2C), 110.8, 55.4, 20.8, 11.6.

Cyclohex-1-en-1-yl acetate, 1q[16]

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 5.37 – 5.30 (m, 1H), 2.16 – 2.04 (m, 7H), 1.76 – 1.65 (m, 2H), 1.62 – 1.52 (m, 2H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 169.6, 148.5, 114.1, 26.9, 23.7, 22.7, 21.8, 21.2.

Reaction setup.

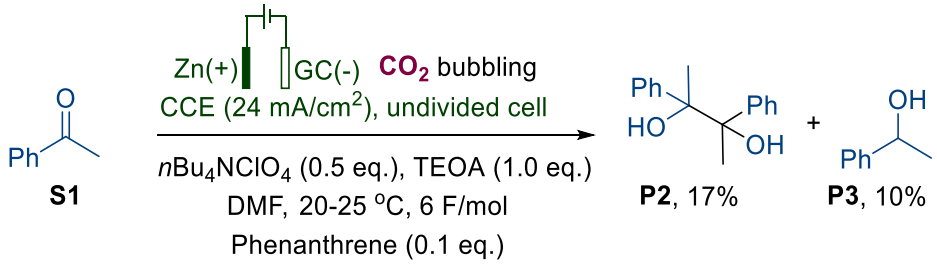
Picture S1. Electrochemical reaction setup

Picture S2. Typical mixture during a reaction

General Experimental Procedure for Scheme 2a.

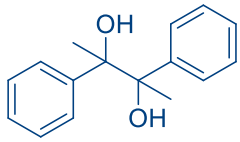
A 5 mL electrolysis cell IKA ElectraSyn 2.0 containing a glassy carbon cathode and a zinc anode (30 mm × 7 mm × 0.1 mm) was charged with a solution of enol derivative **S** (1.0 mmol), nBu$_4$NClO$_4$ (0.5 mmol), TEOA (1.0 mmol) and phenanthrene (0.1 eq.) in DMF (3 mL). The resulting mixture was bubbled with CO_2 (at a flow rate of 2 mL/min) and electrolysed at a constant current of 50 mA (j = 24 mA/cm^2) with stirring at 20-25°C for 194 min. Then, aq. HCl (5 M, 4 mL) was added to the reaction mixture. Extraction with PE/EtOAc (1:1, 5 × 5 mL) afforded the organic phase, which was washed with 10 mL brine and dried over Na_2SO_4, filtered and concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20–25 °C). The crude mixture was purified by column chromatography on silica gel.

Electrolysis of S1



Products **P2** (17%, 40 mg, 0.17 mmol) and **P3** (10%, 12 mg, 0.10 mmol) were isolated by chromatography on SiO_2 (PE:EA = from 8:1 to 1:1).

2,3-Diphenylbutane-2,3-diol, P2[17]



White powder, m.p. = 128-130 ˚C (lit. mp = 127-130 ˚C).[17]

The mixture of DL and meso isomers (ratio = 2/1).

DL isomer[17]:

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.34 – 7.13 (m, 10H), 2.61 (s, 2H), 1.54 (s, 6H).

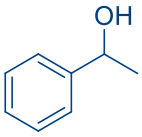
^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 143.5 (2C), 127.5 (4C), 127.3 (4C), 127.2 (2C), 79.0 (2C), 25.2 (2C).

Meso isomer[17]:

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.34 – 7.13 (m, 10H), 2.31 (s, 2H), 1.59 (s, 6H)

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 143.9 (2C), 127.4 (4C), 127.1 (4C), 127.0 (2C), 78.7 (2C), 25.2 (2C).

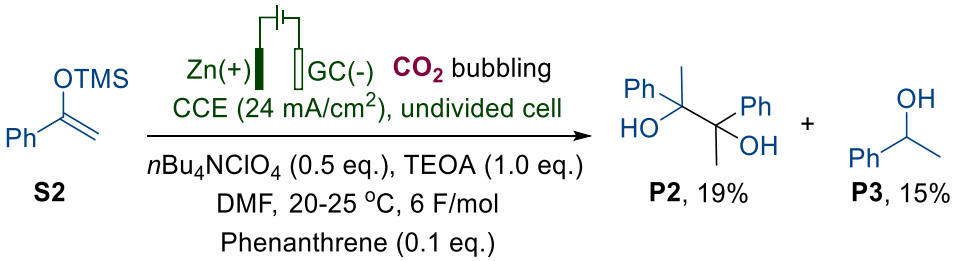
1-Phenylethanol, P3[18]



^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.43 – 7.20 (m, 5H), 4.87 (q, *J* = 6.4 Hz, 1H), 1.49 (d, *J* = 6.4 Hz, 3H).

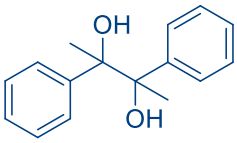
^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 145.9, 128.6 (2C), 127.5, 125.5 (2C), 70.4, 25.2.

Electrolysis of S2



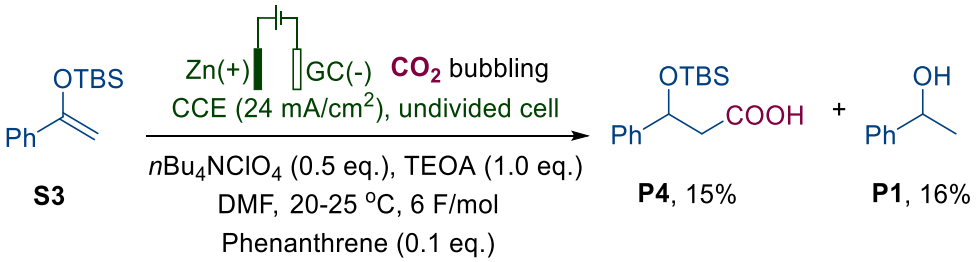
Products **P2** (19%, 45 mg, 0.19 mmol) and **P3** (15%, 18 mg, 0.15 mmol) were isolated by chromatography on SiO_2 (PE:EA = from 5:1 to 1:1).

2,3-Diphenylbutane-2,3-diol, P2[17]



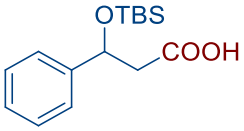
The mixture of DL and meso isomers (ratio = 2/1).

Electrolysis of S3



Products **P4** (15%, 41 mg, 0.15 mmol) and **P1** (16%, 20 mg, 0.16 mmol) were isolated by chromatography on SiO_2 (PE:EA = from 4:1 to 1:1).

3-((*Tert*-butyldimethylsilyl)oxy)-3-phenylpropanoic acid, P4[19]



Colorless oil.

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 10.68 (brs, 1H), 7.40 – 7.22 (m, 5H), 5.15 (dd, *J* = 8.8, 4.2 Hz, 1H), 2.77 (dd, *J* = 15.0, 8.8 Hz, 1H), 2.64 (dd, *J* = 15.0, 4.2 Hz, 1H), 0.86 (s, 9H), 0.04 (s, 3H), -0.16 (s, 3H).

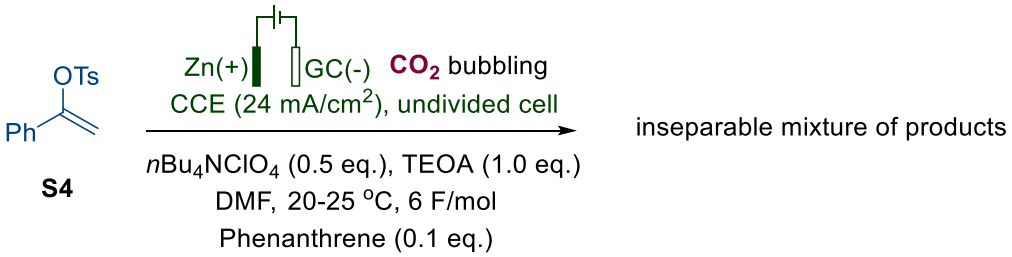
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$, δ): 176.4, 143.6, 128.5 (2C), 127.9, 125.9 (2C), 72.1, 46.0, 25.8 (3C), 18.2, -4.6, -5.2.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{15}H_{24}O_3SiNa]^+$ 303.1387; Found 303.1387.

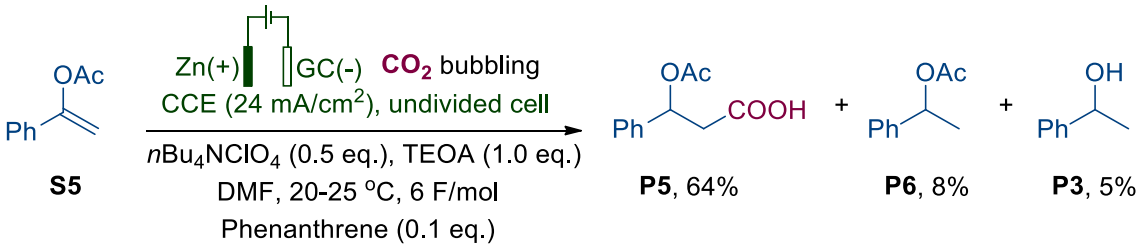
Anal. Calcd for $C_{15}H_{24}SiO_3$: C, 64.24; H, 8.63. Found: C, 64.54; H, 8.59.

IR (ATR): 2955, 2928, 2896, 2857, 1708, 1251, 1091, 828, 776, 698 cm^{-1}.

Electrolysis of S4



Electrolysis of S5

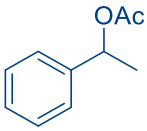


Products **P5** (64%, 133.1 mg, 0.65 mmol), **P3** (8%, 13,8 mg, 0.08 mmol), and **P6** (5%, 6 mg, 0.05 mmol) were isolated by chromatography on SiO_2 (PE:EA = from 5:1 to 1:1).

Further in the text, **S5** is **1a**, **P5** is **2a**.

The characteristic data of the acids **P5 (2a)** is given below.

1-Phenylethyl acetate, P6 [20]



Yellow liquid.

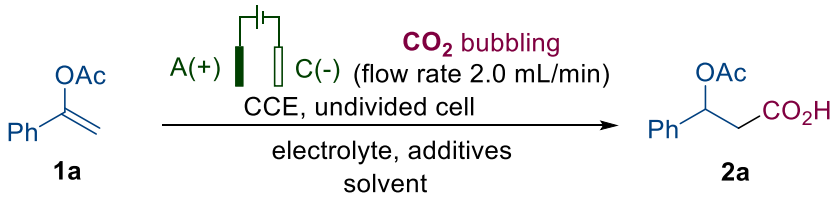
^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.41 – 7.26 (m, 5H), 5.89 (q, *J* = 6.6 Hz, 1H), 2.08 (s, 3H), 1.54 (d, *J* = 6.6 Hz, 3H).

$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$, δ): 170.5, 141.8, 128.6 (2C), 128.0, 126.2 (2C), 72.4, 22.3, 21.5.

Experimental procedure for Table 1.

A 5 mL electrolysis cell IKA ElectraSyn 2.0 containing a cathode (Zn, Pt, GC) and an Zn anode (30 mm × 7 mm × 0.1 mm) was charged with a solution of enol acetate **1a** (1.0 mmol), electrolyte (0.5 mmol), amine (TEOA, DABCO, Et_3N, $HOCH_2CH_2NH_2$) (1.0 mmol), and redox mediator (phenanthrene, *p*-terphenyl) (0.1 eq.) in DMF (3 mL). The resulting mixture was bubbled with CO_2 (at a flow rate of 2 mL/min) and electrolyzed at a constant current of 30-70 mA (j = 14-33 mA/cm^2) or without electricity with stirring at 20-25°C for 103-242 min. Then, aq. HCl (5M, 4 mL) was added to the reaction mixture. Extraction with PE/EtOAc (1:1, 5 × 5 mL) afforded the organic phase, which was washed with 10mL brine and dried over Na_2SO_4, filtered and concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 20–25 °C). The crude mixture was purified by column chromatography on silica gel or analyzed by ^{1}H NMR spectroscopy.

Table S1. The detailed optimization of electrochemical hydrocarboxylation of enol acetate 1a.



Entry	Anode/Cathode	Electrolyte (0.5 mmol)	Additives (base/amine 1.0 mmol, mediator 0.1 mmol)	Solvent (3 mL)	Current density, mA/cm^2	Electricity passed per **1a**, F/mol	Yield **2a**, %
1	C/C	^{n}Bu$_4$NI	TEOA, -	DMF	**7**	**4**	traces [b]
2	C/C	^{n}Bu$_4$NI	TEOA, -	DMF	**48**	**4**	11[b]
3	C/C	^{n}Bu$_4$NI	TEOA, -	**CH$_3$CN**	24	6	traces [b]
4	C/C	^{n}Bu$_4$NI	TEOA, -	**DMSO**	24	6	19[a]
5	C/C	^{n}Bu$_4$NI	TEOA, -	**THF**	24	6	traces [b]
6	C/C	**^{n}Bu$_4$NBr**	TEOA, -	DMF	24	6	14[a]
7	C/C	**^{n}Bu$_4$NBF$_4$**	TEOA, -	DMF	24	6	33[a]
8	C/C	**^{n}Me$_4$NBr**	TEOA, -	DMF	24	6	n.d.[a]
9	C/C	**^{n}Bu$_4$NClO$_4$**	TEOA, -	DMF	24	6	36[b]
10	C/C	**LiClO$_4$**	TEOA, -	DMF	24	6	9[a]
11	C/C	**KI**	TEOA, -	DMF	24	6	traces [b]
12	**C/C**	^{n}Bu$_4$NI	TEOA, -	DMF	24	6	21[a]
13	**C/Ni**	^{n}Bu$_4$NI	TEOA, -	DMF	24	6	6[b]
14	**C/SS**	^{n}Bu$_4$NI	TEOA, -	DMF	24	6	26[a]
15	**C/Zn**	^{n}Bu$_4$NI	TEOA, -	DMF	24	6	40[a]
16	**C/Mg**	^{n}Bu$_4$NI	TEOA, -	DMF	24	6	traces [a]
17	**C/RVC$_{foam}$**	^{n}Bu$_4$NI	TEOA, -	DMF	24	6	15[a]
18	**C/Ni$_{foam}$**	^{n}Bu$_4$NI	TEOA, -	DMF	24	6	traces [a]
19	**C/Cu**	^{n}Bu$_4$NI	TEOA, -	DMF	24	6	traces [b]
20	**Zn/C**	^{n}Bu$_4$NI	TEOA, -	DMF	24	6	50[b]
21	**Mg/C**	^{n}Bu$_4$NI	TEOA, -	DMF	24	6	45[a]
22	**SS/Zn**	^{n}Bu$_4$NI	TEOA, -	DMF	24	6	37[a]
23	**Zn/Zn**	^{n}Bu$_4$NI	TEOA, -	DMF	24	6	48[a]

24	Zn/Zn	$^{n}Bu_4NI$	-, -	DMF	24	6	33[a]
25	Zn/Zn	**$^{n}Bu_4NPF_6$**	TEOA, -	DMF	24	6	34[a]
26	Zn/Zn	**nBu_4NBF4**	TEOA, -	DMF	24	6	51[a]
27	Zn/Zn	**$LiClO_4$**	TEOA, -	DMF	24	6	traces [a]
28	Zn/Zn	**$^{n}Bu_4NBr$**	TEOA, -	DMF	24	6	55[a]
29	Zn/Zn	**$[PyH]ClO_4$**	TEOA, -	DMF	24	6	traces [a]
30	Zn/Zn	**$^{n}Bu_4NClO_4$**	TEOA, -	DMF	24	6	57[a]
31	Zn/Zn	**$^{n}Bu_4NClO_4$**	TEOA, phenanthrene	DMF	24	6	63[a]
32	Zn/Zn	$^{n}Bu_4NClO_4$	**DABCO**, -	DMF	24	6	31[a]
33	Zn/Zn	$^{n}Bu_4NClO_4$	**Et_3N**, -	DMF	24	6	24[a]
34	Zn/Zn	$^{n}Bu_4NClO_4$	**K_2CO_3**, -	DMF	24	6	27[a]
35	Zn/Zn	$^{n}Bu_4NClO_4$	**Py**, -	DMF	24	6	22[a]
36	Zn/Zn	$^{n}Bu_4NClO_4$	**NaOH**, -	DMF	24	6	19[a]
37	Zn/Zn	$^{n}Bu_4NClO_4$	**H_2O**, -	DMF	24	6	25[a]
38	Zn/Zn	$^{n}Bu_4NClO_4$	**$HOCH_2CH_2NH_2$**, -	DMF	24	6	0[a]
39	Zn/Zn	$^{n}Bu_4NClO_4$	**Imidazole**, -	DMF	24	6	30[a]
40	Zn/Zn	$^{n}Bu_4NClO_4$	**1,3-bis(methylamino)propan-2-ol**, -	DMF	24	6	20[a]
41	Zn/Zn	$^{n}Bu_4NClO_4$	**N,N,N',N'-tetraethylmethanediamine**, -	DMF	24	6	12[a]
42	Zn/Zn	$^{n}Bu_4NClO_4$	**TEOA, anthracene**	DMF	24	6	48[a]
43	Zn/Zn	$^{n}Bu_4NClO_4$	**TEOA, phenantroline**	DMF	24	6	44[a]
44	Zn/Zn	$^{n}Bu_4NClO_4$	**TEOA, graphene**	DMF	24	6	44[a]
45	Zn/Zn	$^{n}Bu_4NClO_4$	**TEOA, 1-methylnaphthalene**	DMF	24	6	50[a]
46	Zn/Zn	$^{n}Bu_4NClO_4$	**TEOA, *p*-terphenyl**	DMF	24	6	53[b]
47	Zn/Zn	$^{n}Bu_4NClO_4$	TEOA, phenanthrene	DMF	24	**4**	38[b]

48 [c]	Zn/Zn	$^{n}Bu_4NClO_4$	TEOA, phenanthrene	DMF	24	6	37[b]
49 [d]	Zn/Zn	$^{n}Bu_4NClO_4$	TEOA, phenanthrene	DMF	24	6	6[b]
50 [e]	Zn/Zn	$^{n}Bu_4NClO_4$	TEOA, phenanthrene	DMF	24	6	48[b]
51	**Pt/Pt**	$^{n}Bu_4NClO_4$	TEOA, phenanthrene	DMF	**24**	**6**	11[a]
52	**Zn/Pt**	$^{n}Bu_4NClO_4$	TEOA, phenanthrene	DMF	**24**	**6**	46[a]
53	**Zn/GC**	$^{n}Bu_4NClO_4$	TEOA, phenanthrene	DMF	**24**	**6**	64[a]
54	**Zn/GC**	$^{n}Bu_4NClO_4$	TEOA, phenanthrene	DMF	**24**	**4**	63[a]
55	**Zn/GC**	$^{n}Bu_4NClO_4$	TEOA, phenanthrene	DMF	**24**	**2**	14[b]
56	**Zn/GC**	$^{n}Bu_4NClO_4$	TEOA, phenanthrene	DMF	**14**	**4**	35[a]
57	**Zn/GC**	$^{n}Bu_4NClO_4$	TEOA, phenanthrene	DMF	**33**	**4**	61[a]

[a] isolated **2a** yield.
[b] yield **2a** by ^{1}H NMR
[c] at 0°C
[d] under 5 bar
[e] CO_2 flow rate of 0.5 mL/min

General Experimental Procedure for Table S1.

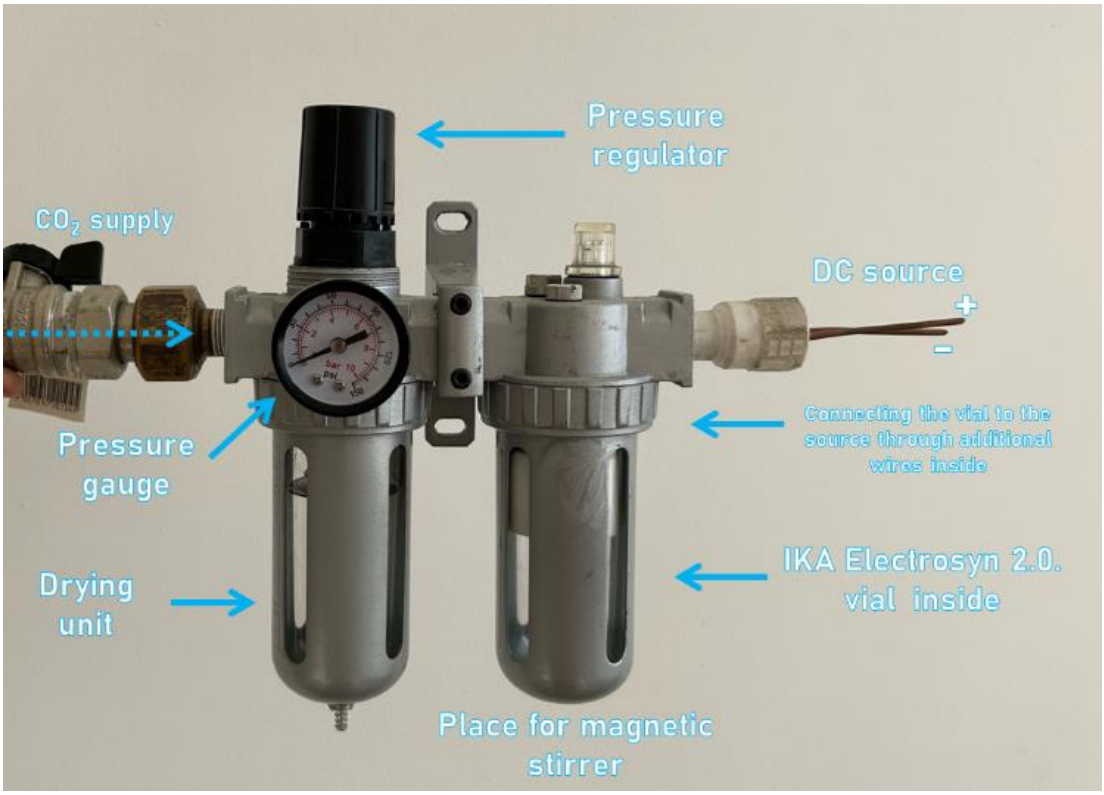
Isolation method A

A 5 mL electrolysis cell IKA ElectraSyn 2.0 containing a plate cathode and plate anode (30 mm × 7 mm × 0.1 mm) was charged with a solution of enol acetate **1a** (1.0 mmol), supporting electrolyte (0.5 mmol) and additives (base/amine 1.0 mmol; mediator 0.1 mmol) in solvent (3 mL). The resulting mixture was bubbled with CO_2 (at a flow rate of 2 mL/min) and electrolyzed at a constant current of 15 – 100 mA (j = 7 - 48 mA/cm^2) with stirring at 20-25 °C for 64-428 min. Then, aq. HCl (5M, 4 mL) was added to the crude reaction mixture. Extraction with Et_2O (1:1, 5×5 mL) afforded the organic phase, which was washed with 10 mL brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 25–30 °C).

In [a] cases, the crude mixture was purified by column chromatography on silica gel (PE:EA = from 5:1 to 1:1) to give the target product.

In [b] cases, the **2a** yield was determined by ^{1}H NMR from the crude reaction mixture.

entries 3,5: CH_3CN and THF are volatile compounds, during the reaction the solvent was carried away by the CO_2 flow. Therefore, as the volume of solvent was reduced, extra portion solvent was added to the reaction (about 20 mL in total for each entry).



Picture S3. Electrochemical reaction setup for the experiment 52 (Table S1).

General Experimental Procedure for Scheme 3.

A 5 mL electrolysis cell IKA ElectraSyn 2.0 containing zinc cathode and glassy carbon anode (30 mm × 7 mm × 0.1 mm) was charged with a solution of enol acetate **1** (1.0 mmol), *n*Bu_4NClO_4 (0.5 mmol, 171 mg), TEOA (1.0 mmol, 149 mg) and phenanthrene (0.1 mmol, 17.8 mg) in DMF (3 mL). The resulting mixture was bubbled with CO_2 (at a flow rate of 2 mL/min) and electrolyzed at a constant current of 50 mA (j = 24 mA/cm^2) with stirring at 20-25 °C for 194 min (for **1e** – 478 min, **1o** – 487 min, for **1p** – 362 min.). Then, aq. HCl (5M, 4 mL) was added to the crude reaction mixture. Extraction with PE/EtOAc (1:1, 5×5 mL) afforded the organic phase, which was washed with 10mL brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 25–30 °C). The crude mixture was purified by column chromatography on silica gel (PE:EA = from 5:1 to 1:1) to give the target product **2**.

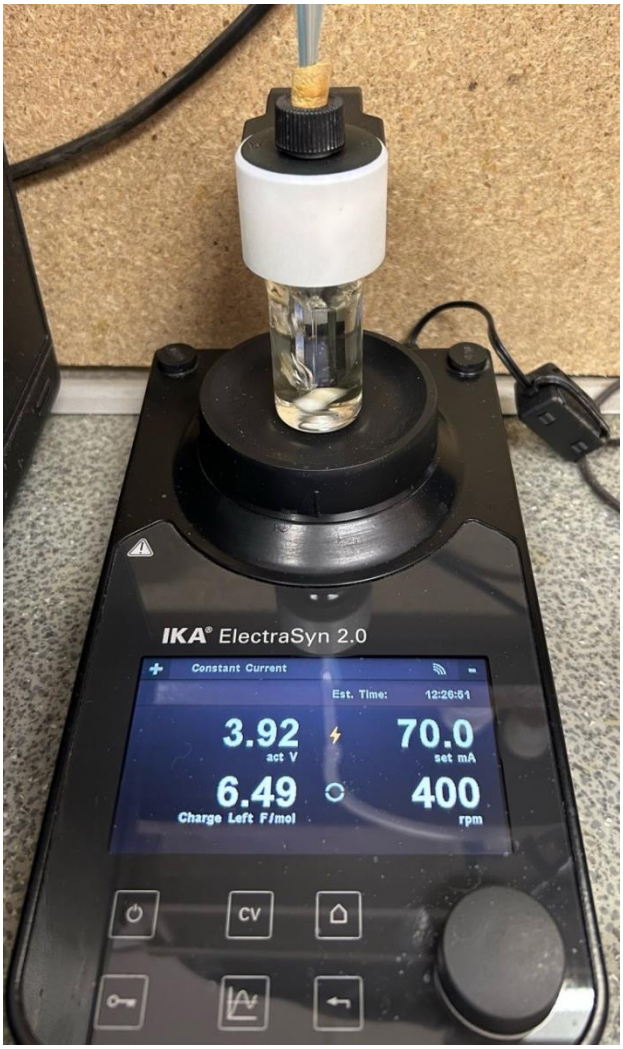
If column chromatography is unsuccessful, pure acids can be isolated by the following procedure:

$NaHCO_3$ (20 mmol), H_2O (10 mL), EtOAc (10 mL) was added to the crude reaction mixture and the resulting mixture were stirred for 4 hours at room temperature. After that water (15 mL) was added and aqueous layer was washed with EtOAc (4×5 mL). The resulting aqueous layer was washed with 0.7M solution of HCOOH in Et_2O (3×15 mL). Combined organic layers were dried over Na_2SO_4, filtered and concentrated under reduced pressure using rotary evaporator (15-20 mmHg), (bath temperature, ca. 40–45 °C) to give the target product **2**.

Gram-scale experiment

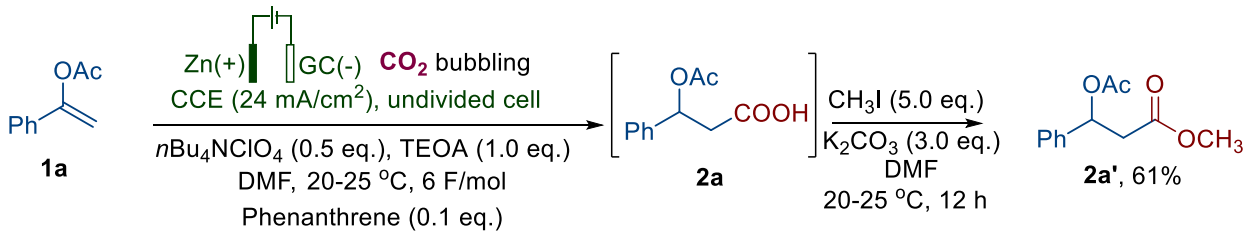
A 20 mL electrolysis cell IKA ElectraSyn 2.0 containing a glassy carbon cathode and zinc anode (30 mm × 7 mm × 0.1 mm) was charged with a solution of enol acetate **1a** (5.0 mmol, 810 mg), *n*Bu_4NClO_4 (2.5 mmol, 855 mg), TEOA (5.0 mmol, 745 mg) and phenanthrene (0.5 mmol, 89 mg) in DMF (15 mL). The resulting mixture was bubbled with CO_2 (at a flow rate of 4 mL/min) and electrolyzed at a constant current of 70 mA (j = 33 mA/cm^2) with stirring at 20-25° for 804 min. Then, aq. HCl (5M, 10 mL) was added to the crude reaction mixture. Extraction with PE/EtOAc (1:1, 5×30 mL) afforded the organic phase, which was washed with 2×30mL brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 25–30 °C). Then the crude reaction mixture was added $NaHCO_3$ (50 mmol), H_2O (70 mL), EtOAc (70 mL) and stirred for 8 hours at room temperature. After that H_2O (15 mL) was added and aqueous layer was washed EtOAc (4×10 mL). The resulting aqueous layer was washed 0.7M solution of HCOOH in Et_2O (3×50 mL). Combined organic layers

were dried over Na_2SO_4, filtered and concentrated under reduced pressure using rotary evaporator (15-20 mmHg), (bath temperature, ca. 40–45 °C) to give the target product **2a**.



Picture S4. Electrochemical reaction setup for Gram-scale experiment.

*The synthesis of methyl ester **2a'***

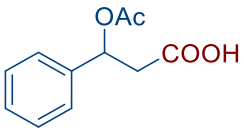


A 5 mL electrolysis cell IKA ElectraSyn 2.0 containing zinc cathode and glassy carbon anode (30 mm × 7 mm × 0.1 mm) was charged with a solution of enol acetate **1a** (1.0 mmol), *n*Bu_4NClO_4 (0.5 mmol, 171 mg), TEOA (1.0 mmol, 149 mg) and phenanthrene (0.1 mmol, 17.8 mg) in DMF (3 mL). The resulting mixture was bubbled with CO_2 (at a flow rate of 2 mL/min) and electrolyzed at a constant current of 50 mA (j = 24 mA/cm^2) with stirring at 20-25 °C for 194 min.

Later, DMF (2.5 mL), K_2CO_3 (3.0 mmol, 414 mg), methyl iodide (5.0 mmol, 709.5 mg) were added to the reaction mixture. The mixture was stirred at 20-25 °C for 12 h. Then, water (10 mL) was added to the crude reaction mixture. Extraction with PE/EtOAc (1:1, 5×10 mL) afforded the organic phase, which was washed with 10mL brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 25–30 °C). The crude mixture was purified by column chromatography on silica gel (PE:EtOAc = from 10:1 to 5:1) to give the target product **2a'**.

The characterization data of the synthesized products.

3-Acetoxy-3-phenylpropanoic acid, 2a[21]



Yield 64% (133.1 mg, 0.64 mmol). For gram-scale experiment yield was 66% (0.690 g, 3.31 mmol)

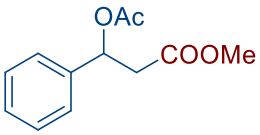
Brown crystals, m.p. = 99-100 ˚C (lit. mp = 100-101 ˚C).[24]

^{1}H NMR (300.13 MHz, $CDCl_3$, δ):11.20 (brs, 1H), 7.50 – 7.27 (m, 5H), 6.17 (dd, *J* = 9.0, 5.0 Hz, 1H), 3.03 (dd, *J* = 16.3, 9.0 Hz, 1H), 2.81 (dd, *J* = 16.3, 5.0 Hz, 1H), 2.07 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 176.0, 170.1, 139.1, 128.8 (2C), 128.6, 126.6 (2C), 71.9, 41.1, 21.1.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{11}H_{12}O_4Na]^+$ 231.0628; Found 231.0623.

Methyl 3-acetoxy-3-phenylpropanoate, 2a' [22]



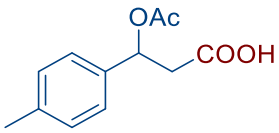
Yield 61% (135.2 mg, 0.61 mmol). Colorless oil.

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.42 – 7.27 (m, 5H), 6.17 (dd, *J* = 8.9, 5.2 Hz, 1H), 3.67 (s, 3H), 2.98 (dd, *J* = 15.8, 8.9 Hz, 1H), 2.76 (dd, *J* = 15.8, 5.2 Hz, 1H), 2.05 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 170.3, 169.9, 139.4, 128.8 (2C), 128.5, 126.6 (2C), 72.2, 52.0, 41.4, 21.2.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{12}H_{14}O_4NH_4]^+$ 240.1230; Found 240.1232.

3-Acetoxy-3-(p-tolyl)propanoic acid, 2b



Yield 55% (122.0 mg, 0.55 mmol). Brownish powder, m.p. = 122-124˚C.

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 10.65 (brs, 1H), 7.35 – 7.08 (m, 4H), 6.14 (dd, *J* = 8.9, 5.1 Hz, 1H), 3.03 (dd, *J* = 16.2, 8.9 Hz, 1H), 2.80 (dd, *J* = 16.2, 5.1 Hz, 1H), 2.35 (s, 3H), 2.05 (s, 3H).

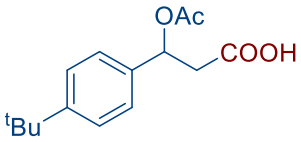
^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 176.1, 170.1, 138.5, 136.1, 129.5 (2C), 126.7 (2C), 71.8, 41.1, 21.3, 21.2.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{12}H_{14}O_4Na]^+$ 245.0784; Found 245.0773.

Anal. Calcd for $C_{12}H_{14}O_4$: C, 64.85; H, 6.35. Found: C, 65.12; H, 6.45.

IR (ATR): 3184, 3050, 2971, 2952, 2873, 1734, 1701, 1519, 1437, 1220, 1208, 1181, 1013, 942 cm^{-1}.

3-Acetoxy-3-(4-(*tert*-butyl)phenyl)propanoic acid, 2c[21]



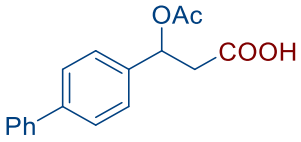
Yield 52% (145.3 mg, 0.52 mmol). Yellow oil.

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 9.72 (brs, 1H), 7.42 – 7.24 (m, 4H), 6.16 (dd, *J* = 9.2, 4.9 Hz, 1H), 3.03 (dd, *J* = 16.3, 9.2 Hz, 1H), 2.80 (dd, *J* = 16.3, 4.9 Hz, 1H), 2.05 (s, 3H), 1.31 (s, 9H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 176.1, 170.2, 151.6, 136.0, 126.4 (2C), 125.7 (2C), 71.7, 41.1, 34.7, 31.4 (3C), 21.2.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{15}H_{20}O_4Na]^+$ 287.1254; Found 287.1260.

3-([1,1'-Biphenyl]-4-yl)-3-acetoxypropanoic acid, 2d[21]



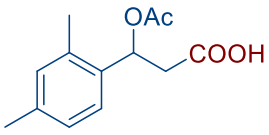
Yield 53% (157.8 mg, 0.53 mmol). White powder, m.p. = 136-138 °C.

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 10.30 (brs, 1H), 7.63 – 7.54 (m, 4H), 7.49 – 7.30 (m, 5H), 6.22 (dd, *J* = 9.0, 5.1 Hz, 1H), 3.08 (dd, *J* = 16.3, 9.0 Hz, 1H), 2.86 (dd, *J* = 16.3, 5.1 Hz, 1H), 2.09 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 175.7, 170.1, 141.6, 140.7, 138.0, 128.9 (2C), 127.6, 127.6 (2C), 127.3 (2C), 127.2 (2C), 71.7, 41.0, 21.2.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{17}H_{16}O_4Na]^+$ 307.0941; Found 307.0938.

3-Acetoxy-3-(2,4-dimethylphenyl)propanoic acid, 2e



Yield 45% (106.1 mg, 0.45 mmol). Yellow powder, m.p. = 80-82 °C.

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 10.30 (brs, 1H), 7.28 – 7.19 (m, 1H), 7.06– 6.96 (m, 2H),6.33 (dd, *J* = 9.2, 4.7 Hz, 1H), 2.97 (dd, *J* = 16.3, 9.2 Hz, 1H), 2.75 (dd, *J* = 16.3, 4.7 Hz, 1H), 2.42 (s, 3H), 2.30 (s, 3H), 2.05 (s, 3H).

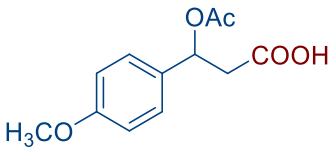
^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 175.9, 170.0, 138.1, 135.3, 134.7, 131.5, 127.2, 125.9, 68.9, 40.7, 21.2, 19.1.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{13}H_{16}O_4Na]^+$ 259.0941; Found 259.0942.

Anal. Calcd for $C_{13}H_{16}O_4$: C, 66.09; H, 6.83. Found: C, 66.38; H, 6.97.

IR (ATR):3040, 2949, 2923, 2858, 1746, 1707, 1435, 1371, 1225, 1212, 1187 cm^{-1}.

3-Acetoxy-3-(4-methoxyphenyl)propanoic acid, 2f



Yield 25% (60.1 mg, 0.25 mmol). Yellow powder, 114-116 °C.

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 9.86 (brs, 1H), 7.34 – 7.27 (m, 2H), 6.90– 6.84 (m, 2H), 6.11 (dd, *J* = 8.8, 5.3 Hz, 1H), 3.80 (s, 4H), 3.03 (dd, *J* = 16.2, 8.8 Hz, 1H), 2.79 (dd, *J* = 16.2, 5.3 Hz, 1H), 2.04 (s, 3H).

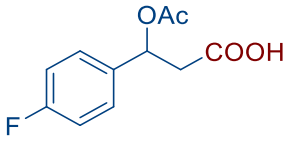
^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 175.6, 170.1, 159.8, 131.2, 128.2 (2C), 114.2 (2C), 71.6, 55.4, 40.9, 21.3.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{12}H_{14}O_5Na]^+$ 261.0733; Found 261.0742.

Anal. Calcd for $C_{12}H_{14}O_5$: C, 60.50; H, 5.92. Found: C, 60.79; H, 6.00.

IR (ATR): 3041, 2955, 2925, 2837, 1733, 1706, 1240, 1206, 1174, 1023, 822 cm^{-1}.

3-Acetoxy-3-(4-fluorophenyl)propanoic acid, 2g



Yield 49% (110.2 mg, 0.49 mmol). Yellow powder, m.p. = 72-74 °C.

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.38 – 7.29 (m, 2H), 7.08– 6.94 (m, 2H), 6.12 (dd, *J* = 8.8, 5.3 Hz, 1H), 3.02 (dd, *J* = 16.3, 8.8 Hz, 1H), 2.78 (dd, *J* = 16.3, 5.3 Hz, 1H), 2.05 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 175.6, 170.0, 162.8 (d, $^1J_{CF}$ = 247.4 Hz), 134.9 (d, $^4J_{CF}$ = 3.2 Hz), 128.6 (d, $^3J_{CF}$ = 8.2 Hz, 2C), 115.8 (d, $^2J_{CF}$ = 21.8 Hz, 2C), 71.2, 41.0, 21.2.

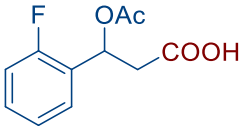
^{19}F NMR (282 MHz, $CDCl_3$, δ): -113.13 (tt, *J* = 8.6, 5.2 Hz).

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{11}H_{11}FO_4Na]^+$ 249.0534; Found 249.0542.

Anal. Calcd for $C_{11}H_{11}FO_4$: C, 58.41; H, 4.90. Found: C, 58.74; H, 4.87.

IR (ATR): 3130, 3070, 2934, 2858, 2798, 1734, 1698, 1604, 1511, 1267, 1222, 1175, 1159, 883 cm^{-1}.

3-Acetoxy-3-(2-fluorophenyl)propanoic acid, 2h



Yield 52% (117.3 mg, 0.52 mmol). Brownish powder, m.p. = 66-68 °C.

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.39 (td, *J* = 7.5, 1.8 Hz, 1H), 7.31 (tdd, *J* = 7.3, 5.3, 1.9, 1H) , 7.16 (td, *J* = 7.5, 1.2 Hz, 1H), 7.07 (ddd, *J* = 10.5, 8.2, 1.2 Hz, 1H), 6.39 (dd, *J* = 9.2, 4.6 Hz, 1H), 3.04 (dd, *J* = 16.5, 9.2 Hz, 1H), 2.88 (dd, *J* = 16.5, 4.6 Hz, 1H), 2.08 (s, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 175.8, 170.0,159.9 (d, $^{1}J_{CF}$ = 248.0 Hz), 130.2 (d, $^{3}J_{CF}$ = 8.4 Hz), 128.0 (d, $^{3}J_{CF}$ = 3.8 Hz), 126.2 (d, $^{2}J_{CF}$ = 13.1 Hz), 124.4 (d, $^{4}J_{CF}$ = 3.8 Hz), 115.9 (d, $^{2}J_{CF}$ = 21.5 Hz), 66.9 (d, $^{3}J_{CF}$ = 2.5 Hz), 39.7 (d, $^{4}J_{CF}$ = 1.9 Hz), 20.9.

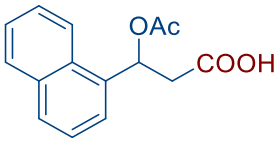
^{19}F NMR (282 MHz, $CDCl_3$, δ): -117.23 (ddd, *J* = 10.5, 7.3, 5.3 Hz).

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{11}H_{11}FO_4Na]^+$ 249.0534; Found 249.0534.

Anal. Calcd for $C_{11}H_{11}FO_4$: C, 58.41; H, 4.90. Found: C, 58.21; H, 4.83.

IR (ATR): 3110, 3052, 2924, 2696, 1738, 1702, 1491, 1434, 1376, 1247, 1233, 1211, 1196, 1032, 765 cm^{-1}.

3-Acetoxy-3-(naphthalen-1-yl)propanoic acid, 2i



Yield 62% (159.1 mg, 0.62 mmol). Yellow powder, m.p. = 96-98 ˚C.

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 9.53 (brs, 1H), 8.19 – 8.13 (m, 1H), 7.90 – 7.80 (m, 2H), 7.61 – 7.42 (m, 4H), 6.94 (dd, *J* = 9.2, 4.2 Hz, 1H), 3.12 (dd, *J* = 16.4, 9.2 Hz, 1H), 3.01 (dd, *J* = 16.4, 4.2 Hz, 1H), 2.14 (s, 3H).

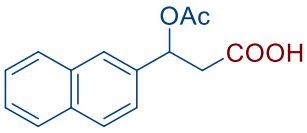
^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 175.9, 170.0, 135.1, 134.0, 130.0, 129.2, 129.2, 126.9, 126.1, 125.4, 123.9, 123.0, 69.6, 41.0, 21.2.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{15}H_{14}O_4Na]^+$ 281.0784; Found 281.0779.

Anal. Calcd for $C_{15}H_{14}O_4$: C, 69.76; H, 5.46. Found: C, 69.74; H, 5.42.

IR (ATR):3060, 2924, 1729, 1693, 1512, 1377, 1255, 1239, 1159, 1021, 778 cm^{-1}.

3-Acetoxy-3-(naphthalen-2-yl)propanoic acid, 2j



Yield 64% (165.3 mg, 0.64 mmol). White powder, m.p. = 124-126 ˚C.

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.87 – 7.79 (m, 3H), 7.53 – 7.44 (m, 4H), 6.33 (dd, *J* = 9.0, 5.1 Hz, 1H), 3.13 (dd, *J* = 16.3, 9.0 Hz, 1H), 2.90 (dd, *J* = 16.3, 5.1 Hz, 1H), 2.08 (s, 3H).

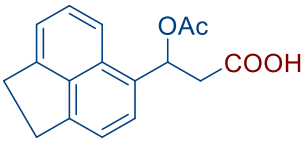
^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 175.7, 170.1, 136.4, 133.4, 133.2, 128.8, 128.3, 127.8, 126.6, 126.1, 124.1, 72.1, 41.1, 21.2.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{15}H_{14}O_4Na]^+$ 281.0784; Found 281.0792.

Anal. Calcd for $C_{15}H_{14}O_4$: C, 69.76; H, 5.46. Found: C, 69.66; H, 5.66.

IR (ATR): 3031, 2967, 2917, 2683, 1736, 1698, 1426, 1375, 1241, 1222, 1186, 1033, 833, 751 cm^{-1}.

3-Acetoxy-3-(1,2-dihydroacenaphthylen-5-yl)propanoic acid, 2k



Yield 51% (144.8 mg, 0.51 mmol). White powder, m.p. = 126-128 ˚C.

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.90 – 7.83 (m, 1H), 7.54 – 7.47 (m, 2H), 7.35 – 7.22 (m, 2H), 6.78 (dd, *J* = 9.2, 4.7 Hz, 1H), 3.44 – 3.25 (m, 4H), 3.19 (dd, *J* = 16.3, 9.2 Hz, 1H), 2.99 (dd, *J* = 16.3, 4.7 Hz, 1H), 2.09 (s, 3H).

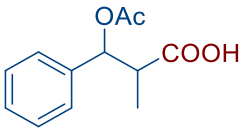
^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 175.8, 170.1, 147.2, 146.9, 139.8, 130.7, 128.7, 128.6, 126.1, 119.7, 119.1, 119.0, 70.1, 40.7, 30.6, 30.1, 21.2.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{17}H_{16}O_4Na]^+$ 307.0941; Found 307.0933.

Anal. Calcd for $C_{17}H_{16}O_4$: C, 71.82; H, 5.67. Found: C, 71.80; H, 5.68.

IR (ATR): 3046, 2960, 2945, 2908, 2825, 1751, 1707, 1418, 1364, 1250, 1222, 1194, 841, 769 cm^{-1}.

3-Acetoxy-2-methyl-3-phenylpropanoic acid, 2l [23]



Yield of two diastereomers was 52% (115.4 mg, 0.52 mmol). The diastereomer ratio was 5:2. Yellow oil.

HRMS (ESI/TOF) m/z: $[M + K]^+$ Calcd for $[C_{12}H_{14}O_4K]^+$ 261.0524; Found 261.0529.

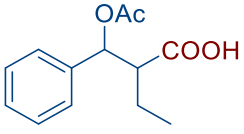
Major: ^{1}H NMR (300.23 MHz, $CDCl_3$, δ): 9.80 (brs, 1H), 7.36 – 7.26 (m, 5H), 5.82 (d, *J* = 10.1 Hz, 1H), 3.07 – 2.91 (m, 1H), 2.01 (s, 3H), 1.01 (d, *J* = 7.2 Hz, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 179.1, 169.9, 138.3, 128.5 (2C), 128.2, 126.7 (2C), 75.5, 45.5, 21.0, 12.2.

Minor: ^{1}H NMR (300.23 MHz, $CDCl_3$, δ): 9.80 (brs, 1H), 7.36 – 7.26 (m, 5H), 6.10 (d, *J* = 6.4 Hz, 1H), 3.07 – 2.91 (m, 1H), 2.10 (s, 3H), 1.24 (d, *J* = 7.1 Hz, 3H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): 180.2, 169.9, 137.7, 128.7, 128.7 (2C), 127.6 (2C), 77.2, 45.3, 21.1, 14.0.

2-(Acetoxy(phenyl)methyl)butanoic acid, 2m



Yield of mixture was 49% (115.63 mg, 0.49 mmol). The diastereomer ratio was 10:7. Yellow oil. HRMS (ESI/TOF) m/z: $[M + K]^+$ Calcd for $[C_{13}H_{16}O_4K]^+$ 275.0675; Found 275.0680.

Anal. Calcd for $C_{13}H_{16}O_4$: C, 66.09; H, 6.83. Found: C, 65.95; H, 6.73.

IR (ATR): 3035, 2974, 2937, 2880, 1740, 1707, 1378, 1358, 1227, 1023, 869, 699 cm^{-1}.

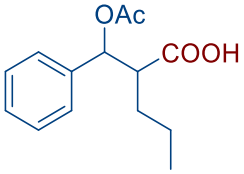
Major: ^1H NMR (300.23 MHz, $CDCl_3$, δ):10.19 (brs, 1H), 7.40 – 7.26 (m, 5H), 5.97 (d, *J* = 8.4 Hz, 1H), 2.91– 2.78 (m, 1H), 2.08 (s, 3H), 1.76 (quintet, *J* = 7.4 Hz, 2H), 0.96 (t, *J* = 7.4 Hz, 3H).

$^{13}C\{^1H\}$ NMR (75.48 MHz, $CDCl_3$, δ): 178.6, 170.1, 138.3, 128.5 (2C), 128.5, 127.1 (2C), 76.7, 53.3, 21.8, 21.1, 11.7.

Minor: ^1H NMR (300.23 MHz, $CDCl_3$, δ): 10.19 (brs, 1H), 7.40 – 7.26 (m, 5H), 5.82 (d, *J* = 10.4 Hz, 1H), 2.91 – 2.78 (m, 1H), 1.98 (s, 3H), 1.55 – 1.38 (m, 1H), 1.33 – 1.18 (m, 1H), 0.86 (t, *J* = 7.4 Hz, 3H).

$^{13}C\{^1H\}$ NMR (75.48 MHz, $CDCl_3$, δ): 179.6, 169.9, 137.9, 128.7, 128.7 (2C), 127.6 (2C), 75.5, 52.9, 22.0, 21.0, 11.3.

2-(Acetoxy(phenyl)methyl)pentanoic acid, 2n



Yield of mixture was 58% (145.1 mg, 0.58 mmol). The diastereomer ratio was 10:6.5. Colorless oil. HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{14}H_{18}O_4Na]^+$ 273.1097; Found 273.1107.

Anal. Calcd for $C_{14}H_{18}O_4$: C, 67.18; H, 7.25. Found: C, 67.47; H, 7.28.

IR (ATR): 3035, 2963, 2934, 2874, 1741, 1707, 1373, 1228, 1022, 870, 700 cm^{-1}.

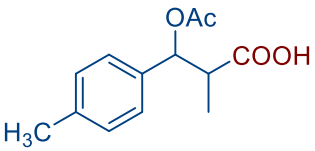
Major: ^1H NMR (300.13 MHz, $CDCl_3$): 10.09 (brs, 1H), 7.37 – 7.26 (m, 5H), 5.95 (d, *J* = 8.3 Hz, 1H), 2.98 – 2.86 (m, 1H), 2.08 (s, 3H), 1.79 – 1.55 (m, 2H), 1.52 – 1.29 (m, 2H), 0.91 (t, *J* = 7.3 Hz, 3H).

$^{13}C\{^1H\}$ NMR (75.48 MHz, $CDCl_3$): δ 177.5, 170.0, 138.4, 128.5 (2C), 128.5, 127.2 (2C), 75.7, 51.6, 30.7, 21.2, 20.6, 14.0.

Minor: ^1H NMR (300.13 MHz, $CDCl_3$): 10.09 (brs, 1H), 7.37 – 7.26 (m, 5H), 5.79 (d, *J* = 10.4 Hz, 1H), 2.98 – 2.86 (m, 1H), 1.98 (s, 3H), 1.52 – 1.03 (m, 4H), 0.79 (t, *J* = 7.2 Hz, 3H).

$^{13}C\{^1H\}$ NMR (75.48 MHz, $CDCl_3$): δ 178.7, 169.7, 138.0, 128.8, 128.8 (2C), 127.7 (2C), 76.9, 51.2, 30.8, 21.1, 20.3, 13.9.

3-Acetoxy-2-methyl-3-(p-tolyl)propanoic acid, 2o



Yield of mixture was 56% (132.2 mg, 0.56 mmol). The diastereomer ratio was 10:1.5. Colorless oil. HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_{13}H_{16}O_4Na]^+$ 259.0941; Found 259.0941.

Anal. Calcd for $C_{13}H_{16}O_4$: C, 66.09; H, 6.83. Found: C, 66.09; H, 6.80.

IR (ATR): 2979, 2960, 2924, 1742, 1702, 1222, 1201, 1018, 813 cm^{-1}.

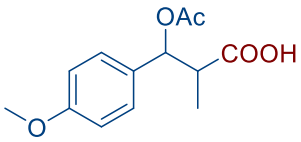
Major: ^1H NMR (300.13 MHz, $CDCl_3$): 7.25 – 7.08 (m, 4H), 6.05 (d, *J* = 6.6 Hz, 1H), 2.99 – 2.87 (m, 1H), 2.33 (s, 3H), 2.08 (s, 3H), 1.23 (d, *J* = 7.1 Hz, 3H).

$^{13}C\{^1H\}$ NMR (75.48 MHz, $CDCl_3$): 179.3, 170.1, 138.1, 135.4, 129.2 (2C), 126.6 (2C), 75.5, 45.6, 21.3, 21.2, 12.4.

Minor: ^1H NMR (300.13 MHz, $CDCl_3$): 7.25 – 7.08 (m, 4H), 5.78 (d, *J* = 10.1 Hz, 1H), 3.03 – 2.91 (m, 1H), 2.33 (s, 3H), 1.99 (s, 3H), 0.99 (d, J = 7.2 Hz, 3H).

$^{13}C\{^1H\}$ NMR (75.48 MHz, $CDCl_3$): 180.4, 169.9, 138.6, 134.6, 129.4 (2C), 127.5 (2C), 77.1, 45.2, 21.4, 21.2, 14.1.

3-Acetoxy-3-(4-methoxyphenyl)-2-methylpropanoic acid, 2p



Yield of mixture was 35% (88.4 mg, 0.35 mmol). The diastereomer ratio was 10:3.9. Brownish oil. HRMS (ESI/TOF) m/z: $[M + NH_4]^+$ Calcd for $[C_{13}H_{16}O_5NH_4]^+$ 270.1336; Found 270.1337.

Anal. Calcd for $C_{13}H_{16}O_5$: C, 61.90; H, 6.39. Found: C, 61.82; H, 6.29.

IR (ATR): 2985, 2940, 2915, 2839, 1736, 1707, 1515, 1227, 1175, 1022, 825 cm^{-1}.

Major: ^1H NMR (300.13 MHz, $CDCl_3$): 10.02 (brs, 1H), 7.33 – 7.19 (m, 2H), 6.92 – 6.81 (m, 2H), 6.00 (d, *J* = 7.1 Hz, 1H), 3.78 (s, 3H), 2.97 (quintet, *J* = 7.1 Hz, 1H), 2.07 (s, 3H), 1.25 (d, *J* = 7.1 Hz, 3H).

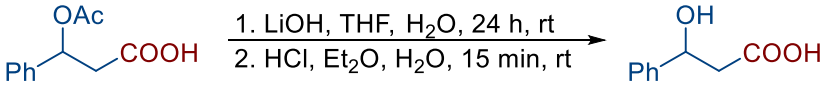
$^{13}C\{^1H\}$ NMR (75.48 MHz, $CDCl_3$): 179.2, 170.1, 159.5, 130.5, 128.2 (2C), 113.9 (2C), 75.5, 55.3, 45.7, 21.1, 12.9.

Minor: ^1H NMR (300.13 MHz, $CDCl_3$): 10.02 (brs, 1H), 7.33 – 7.19 (m, 2H), 6.92 – 6.81 (m, 2H), 5.77 (d, *J* = 10.1 Hz, 1H), 3.79 (s, 3H), 3.04 – 2.89 (m, 1H), 1.98 (s, 3H), 0.99 (d, *J* = 7.2 Hz, 3H).

$^{13}C\{^1H\}$ NMR (75.48 MHz, $CDCl_3$): 180.2, 169.9, 159.8, 129.7, 128.9 (2C), 114.1 (2C), 76.9, 55.4, 45.3, 21.1, 14.1.

Experimental Procedures for Scheme 4.

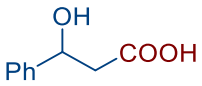
Synthesis of 3-Hydroxy -3-phenylpropanoic acid, 3a[24]



2a **3a**

LiOH (1.15 g, 5.55 mmol) in H_2O (15 mL) was added to a solution of acid **2a** (300.0 mg, 1.44 mmol) in THF (180 mL) at 20-25 °C. The mixture was stirred at room temperature 24 h. Et_2O (200 mL) and dilute aqueous HCl (1M, 200 mL) were added and the mixture was stirred for 15 min before separating the layers. The aqueous layer was extracted with Et_2O (3 x 70 mL). The combined organic layer was washed with water (2 x 70 mL), dried over Na_2SO_4, filtered and concentrated under reduced pressure using rotary evaporator (15-20 mmHg), (bath temperature, ca. 25–30 °C) to give the target product **3a**.

3-Hydroxy-3-phenylpropanoic acid 3a[25]



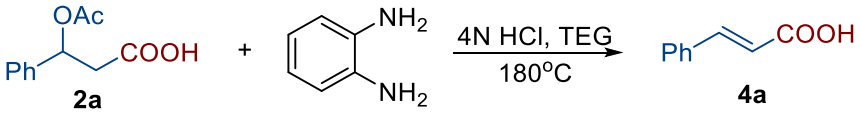
Yield 89% (212.4 mg, 1.28 mmol). Brownish oil.

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 7.44 – 7.27 (m, 5H), 6.66 – 5.45 (brs, 1H), 5.16 (dd, *J* = 8.9, 4.0 Hz, 1H), 2.85 (dd, *J* = 16.6, 8.9 Hz, 1H), 2.76 (dd, *J* = 16.6, 4.0 Hz, 1H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): δ 177.2, 142.2, 128.8 (2C), 128.2, 125.8 (2C), 70.4, 43.2.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_9H_{10}O_3Na]^+$ 189.0522; Found 189.0535.

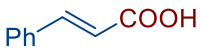
Synthesis 3-phenylacrylic acid, 4a



2a **4a**

A solution *o*-phenylenediamine (108.0 mg, 1.0 mmol) in 4N solution HCl (8 mL) was added to a solution of acid **2a** (250.0 mg, 1.2 mmol) in triethylene glycol (3 mL). The resulting mixture was refluxed at 180°C for 6 h. Later the reaction mixture was cooled at 20 °C and adjusted to pH ~ 6 with aq. NaOH (5N). Extraction with CH_2Cl_2 (1:1, 5x5 mL) afforded the organic phase, which was washed with 10 mL brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 25–30 °C). The crude mixture was purified by column chromatography on silica gel (PE:EA = 2:1) to give the target product.

3-Phenylacrylic acid, 4a[26]



Yield 91% (161.4 mg, 1.09 mmol). Brownish powder.

^{1}H NMR (300.13 MHz, $CDCl_3$, δ): 11.22 (brs, 1H), 7.81 (d, *J* = 16.0 Hz, 1H), 7.57 (m, 2H), 7.49 – 7.35 (m, 3H), 6.47 (d, *J* = 16.0 Hz, 1H).

^{13}C{^{1}H} NMR (75.48 MHz, $CDCl_3$, δ): δ 172.3, 147.2, 134.2, 130.9, 129.1 (2C), 128.5 (2C), 117.4.

HRMS (ESI/TOF) m/z: $[M + Na]^+$ Calcd for $[C_9H_8O_2Na]^+$ 171.0417; Found 171.0425.

Experimental Procedures for Scheme 5b.

A 5 mL electrolysis cell IKA ElectraSyn 2.0 containing zinc cathode and glassy carbon anode (30 mm × 7 mm × 0.1 mm) was charged with a solution of enol acetate **1a** (1.0 mmol, 162 mg), *n*Bu_4NClO_4 (0.5 mmol, 171 mg), TEOA (1.0 mmol, 149 mg) and phenanthrene (0.1 mmol, 17.8 mg) in DMF (3 mL). The resulting mixture was stirred and degassed by bubbling argon through the solvent for ca. 5 minutes. Than mixture electrolyzed at a constant current of 50 mA (*j* = 24 mA/cm^2) with stirring at 20-25 °C for 32, 64, 129 or 194 min. Then, aq. HCl (5M, 4 mL) was added to the crude reaction mixture. Extraction with PE/EtOAc (1:1, 5×5 mL) afforded the organic phase, which was washed with 10mL brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure using a rotary evaporator (15-20 mmHg), (bath temperature, ca. 25–30 °C). The crude reaction mixture was studied by ^{1}H NMR.

CV study.

Cyclic voltammetry (CV) was implemented on an IPC-Pro M computer-assisted potentiostat manufactured by «Econix» (scan rate error 1.0%). The starting potential was set to 0.25 mV, and the initial sweep was carried out in the negative (cathode) region at a rate of 100 mV/s. The experiments were performed in a 10 mL fiveneck glass conic electrochemical cell with a water jacket for thermostating. CV curves were recorded using a three-electrode scheme. In a typical case, 2 mL of a solution was utilized. The working electrode was a glassy-carbon disk electrode (d = 3 mm, surface area ~0.07 cm^2). A platinum wire served as an auxiliary electrode. An $Ag/AgNO_3$ electrode was used as the reference electrode and was linked to the solution by a porous glass diaphragm. The solutions were kept under thermally controlled conditions at 25±0.5 °C and deaerated by bubbling argon. Electrochemical experiments were performed under an argon atmosphere. The working electrode was polished with figure-eight motions on a synthetic chamois leather pad using a Cr_2O_3-based polishing paste (~5 μm particle size) down to the mirror-like surface, and rinsed with acetonitrile. Polishing was carried before each recording of CV curve. In the case of testing CV with carbon dioxide, CO_2 was used instead of argon.

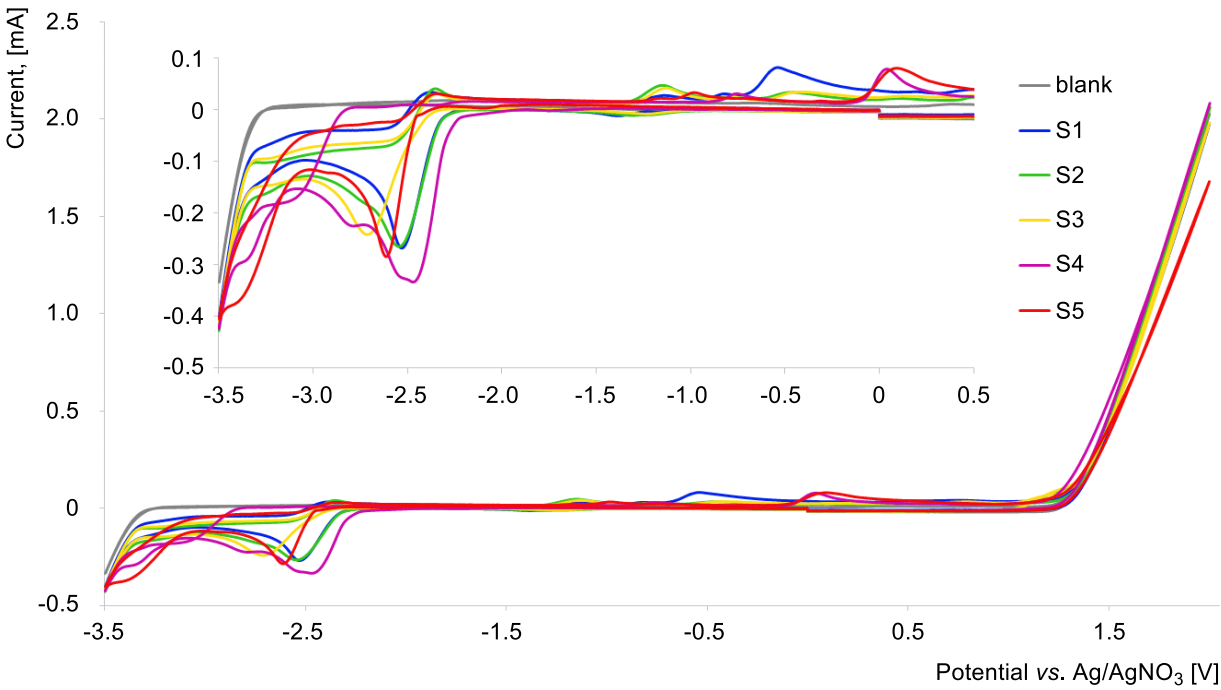


Figure S1. CV curves on a working glassy-carbon electrode (d = 3 mm) under a scan rate of 0.1 V/s for (grey) blank, (blue) acetophenone **S1** (0.02 M), TMS-enol ether **S2** (0.02 M), TBS-enol ether **S3** (0.02 M), vinyl tosylate **S4** (0.02 M), enol acetate **S5** (0.02 M) in 0.1 M $n$$Bu_4NClO_4$ solution in DMF.

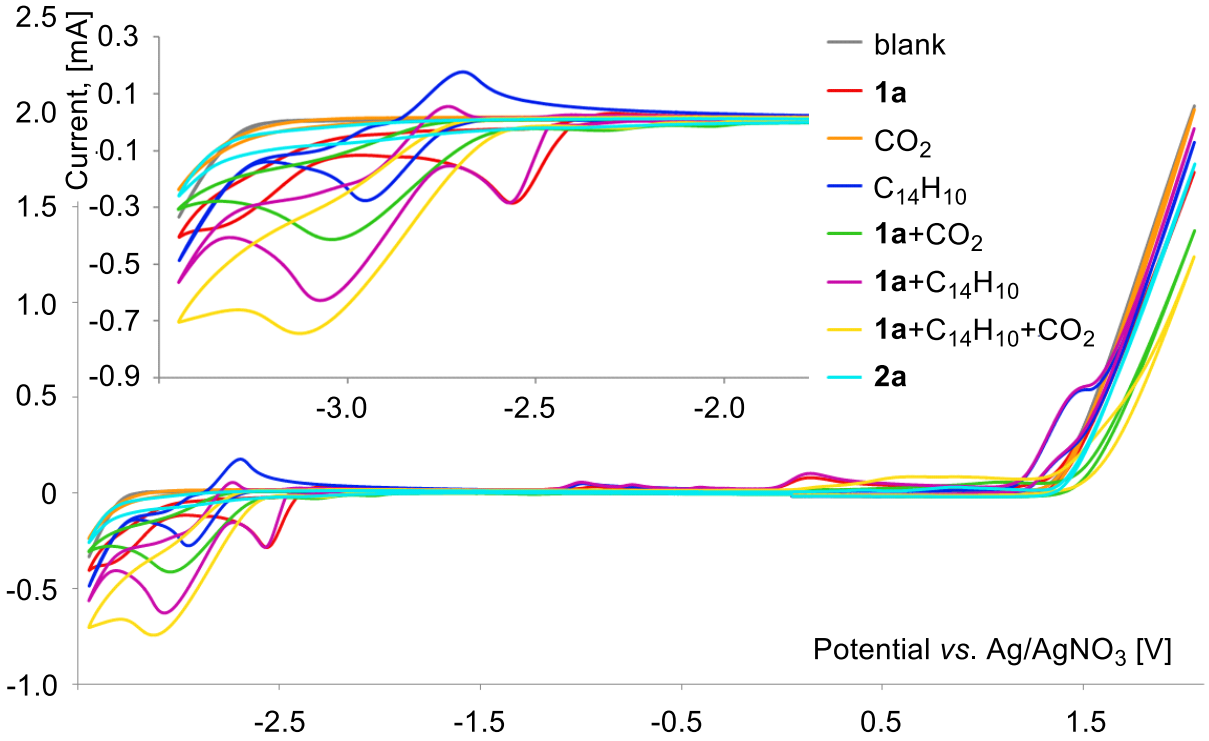


Figure S2. CV curves on a working glassy-carbon electrode (d = 3 mm) under a scan rate of 0.1 V/s for (grey) blank, (red) enol acetate **1a** (0.02 M), (orange) CO_2, (blue) Phenanthrene (0.02 M), (green) enol acetate **1a** (0.02 M) with CO_2, (purple) enol acetate **1a** (0.02 M) with phenanthrene (0.02 M), (yellow) enol acetate **1a** (0.02 M) with CO_2 and phenanthrene (0.02 M), (turquoise) 3-acetoxy-3-phenyl-propanoic acid **2a** (0.02 M) in 0.1 M *n*Bu_4NClO_4 solution in DMF.

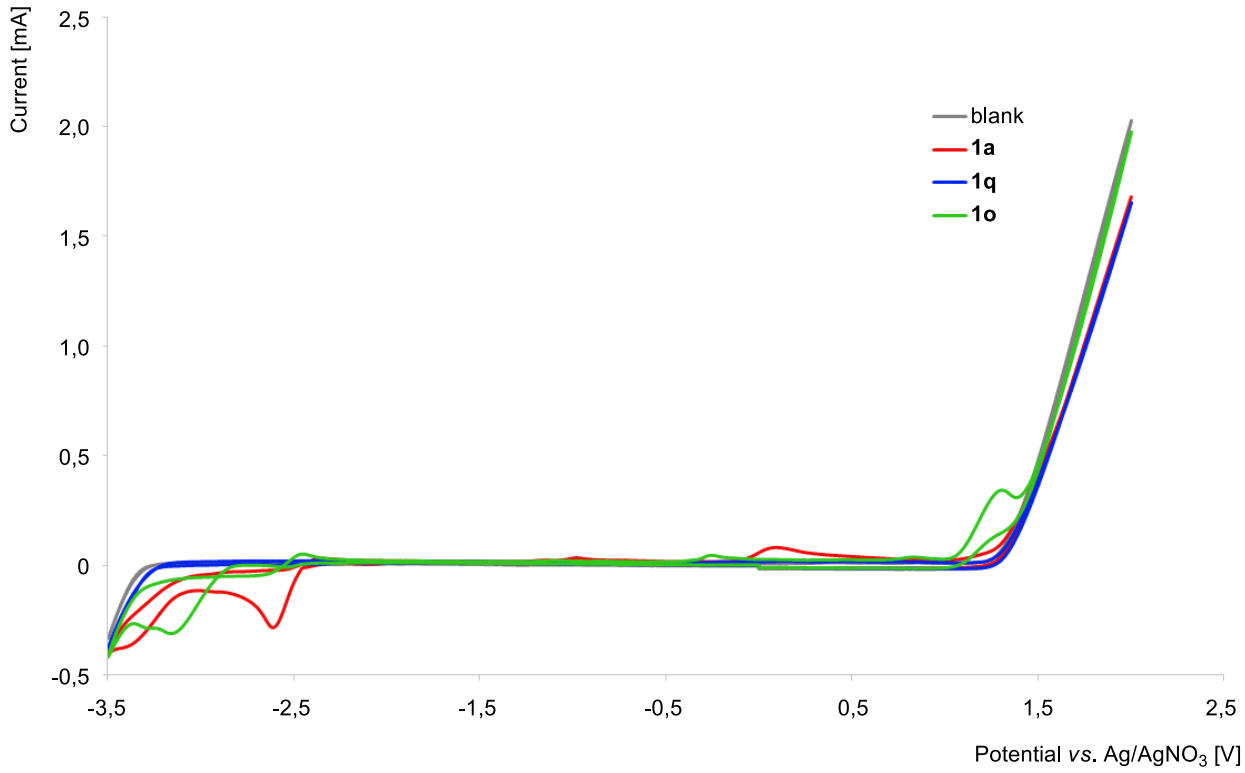


Figure S3. CV curves on a working glassy-carbon electrode (d = 3 mm) under a scan rate of 0.1 V/s for (grey) blank, (red) enol acetate **1a** (0.02 M), (blue) enol acetate **1q** (0.02 M), (green) enol acetate **1o** (0.02 M) in 0.1 M nBu$_4$NClO$_4$ solution in DMF.

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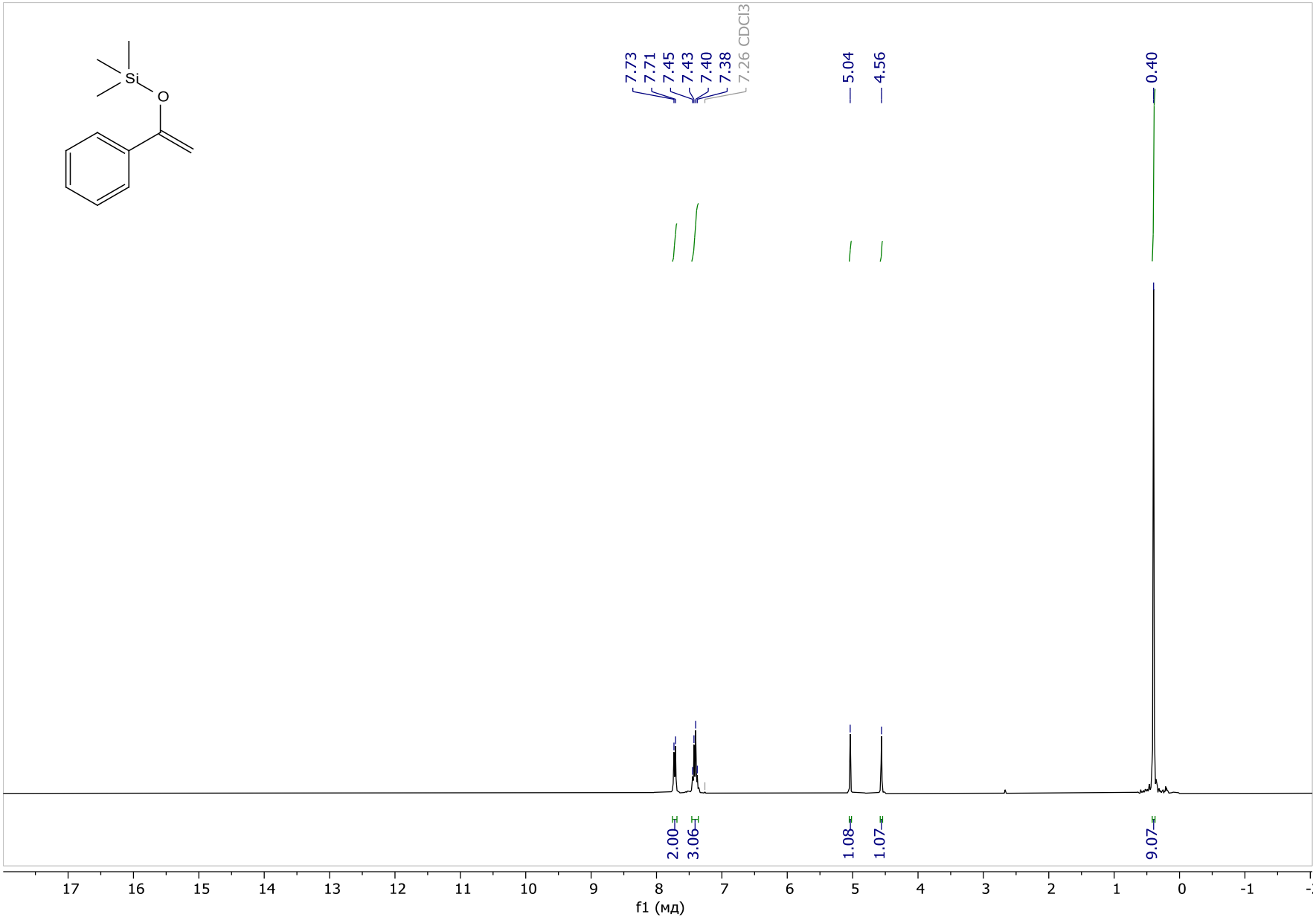
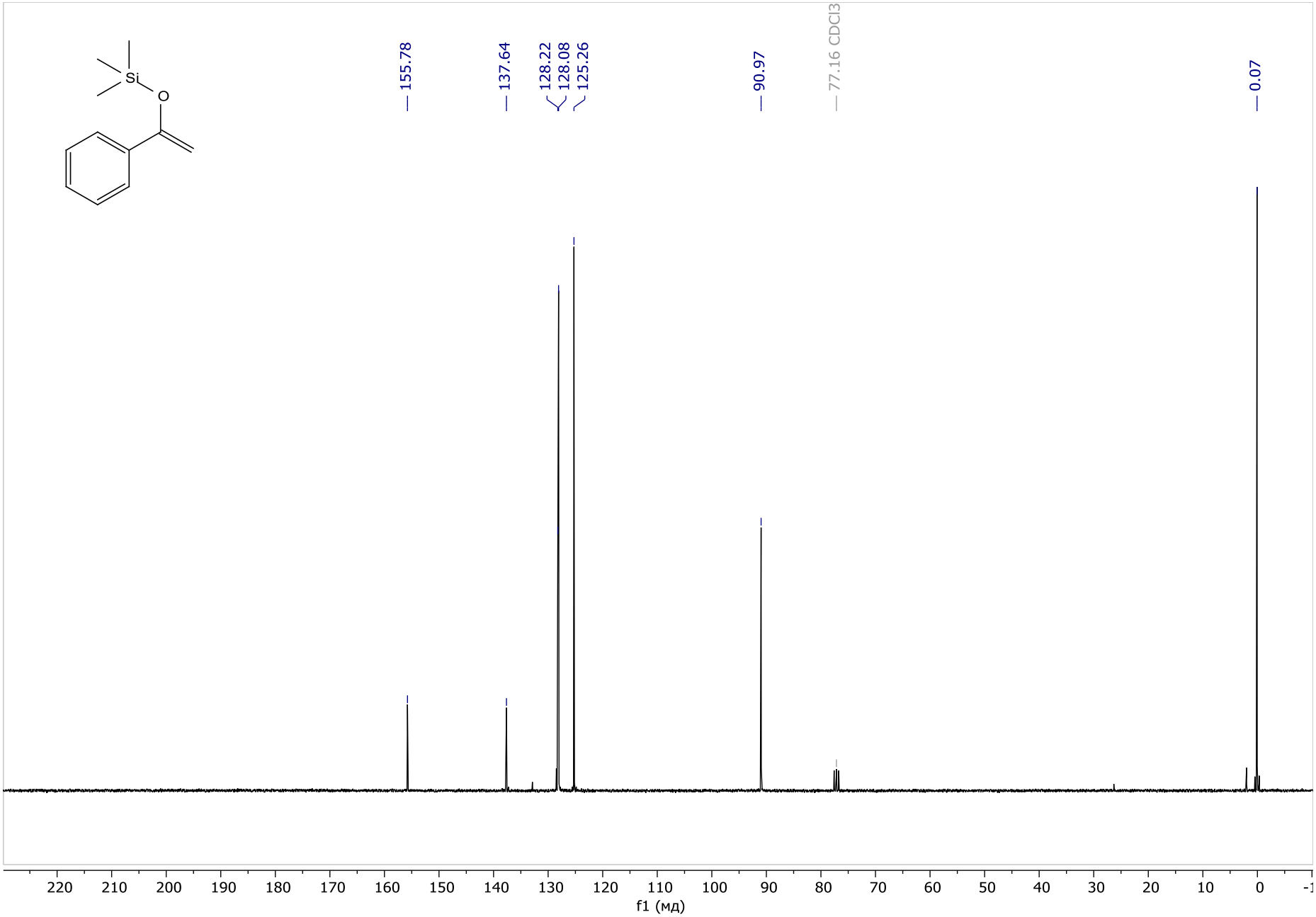
NMR spectra of the starting enol derivatives.

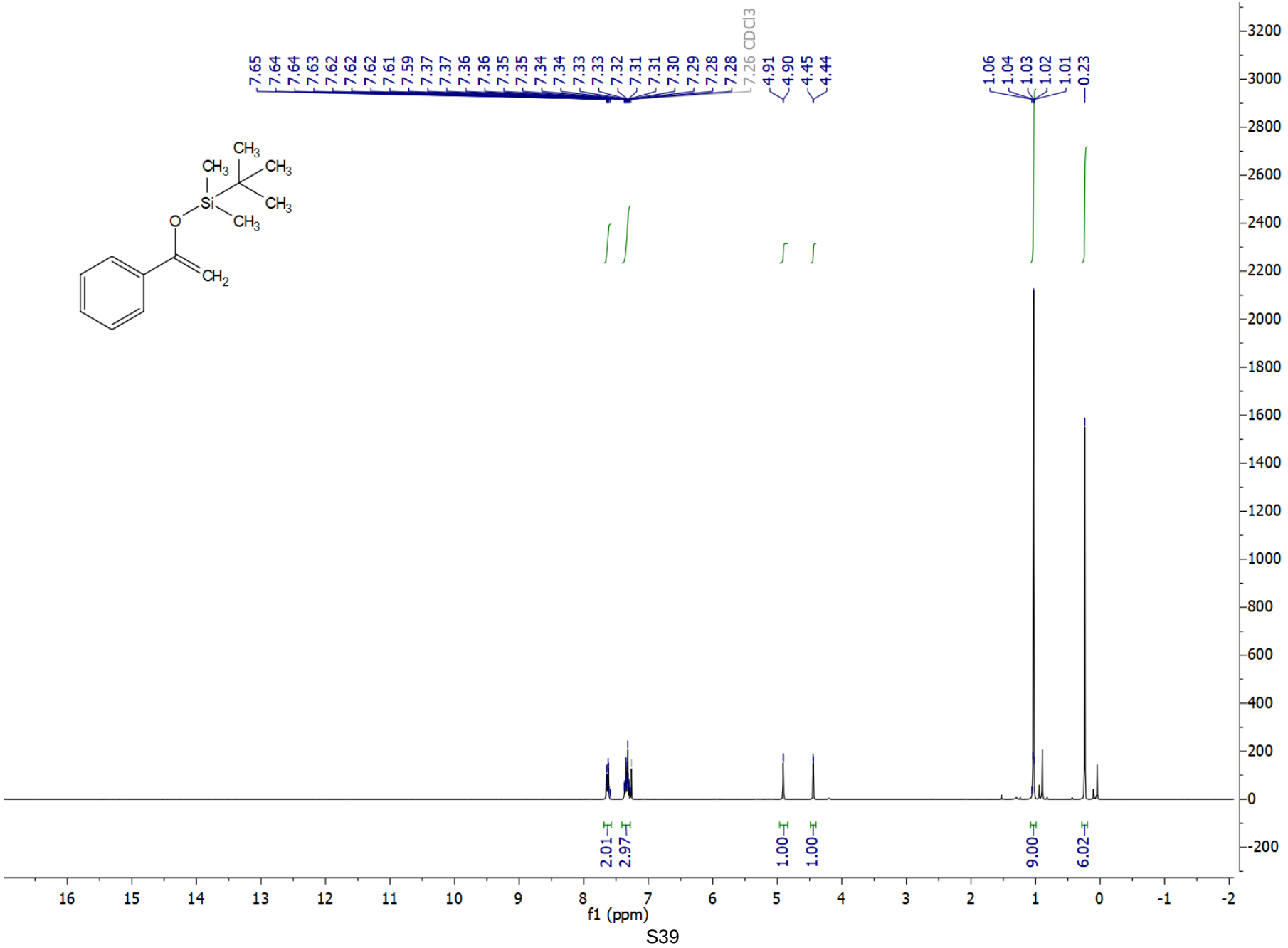
^{1}H NMR (300.13 MHz, $CDCl_3$), **Trimethyl((1-phenylvinyl)oxy)silane, S2**



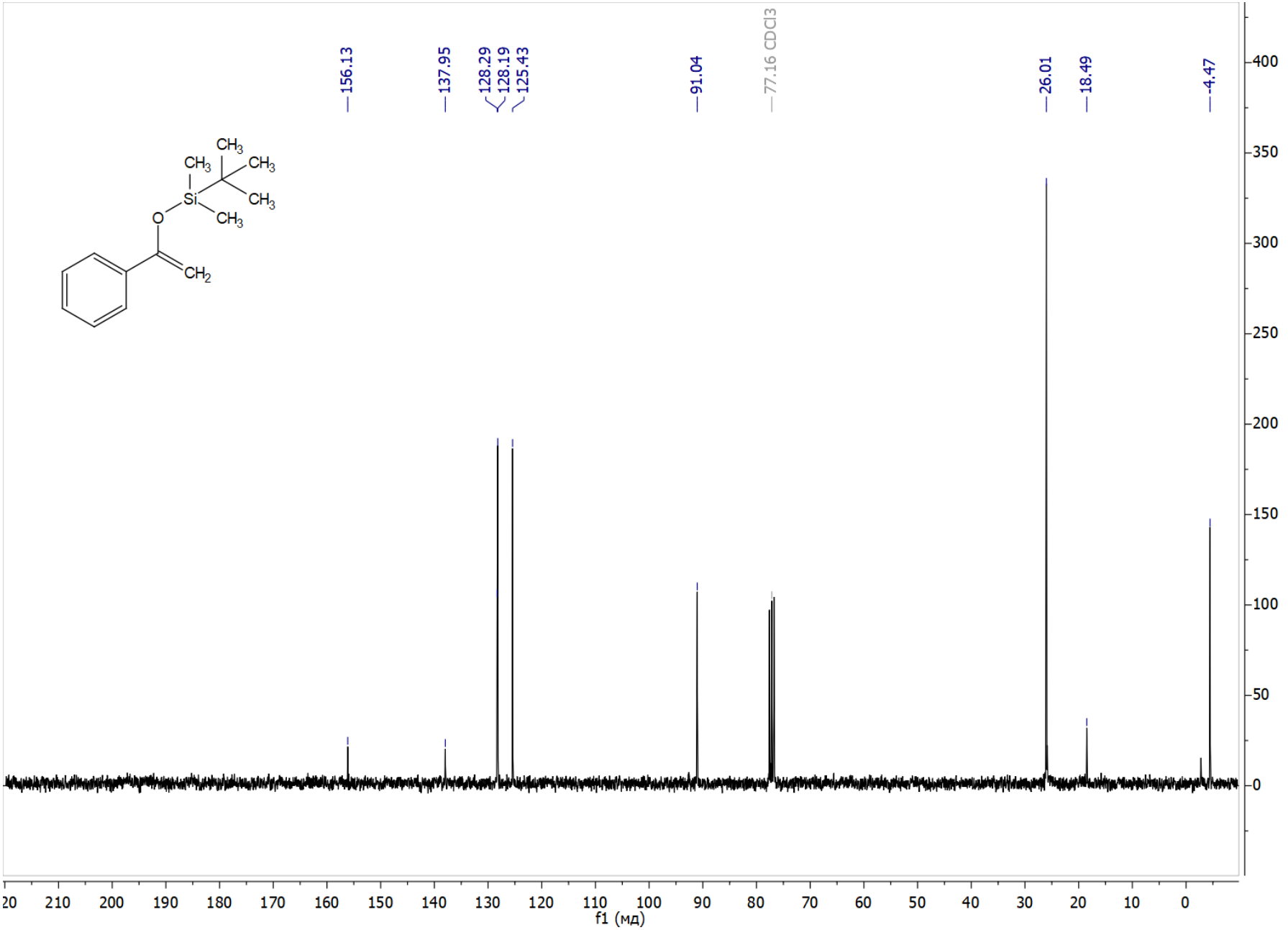
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **Trimethyl((1-phenylvinyl)oxy)silane, S2**



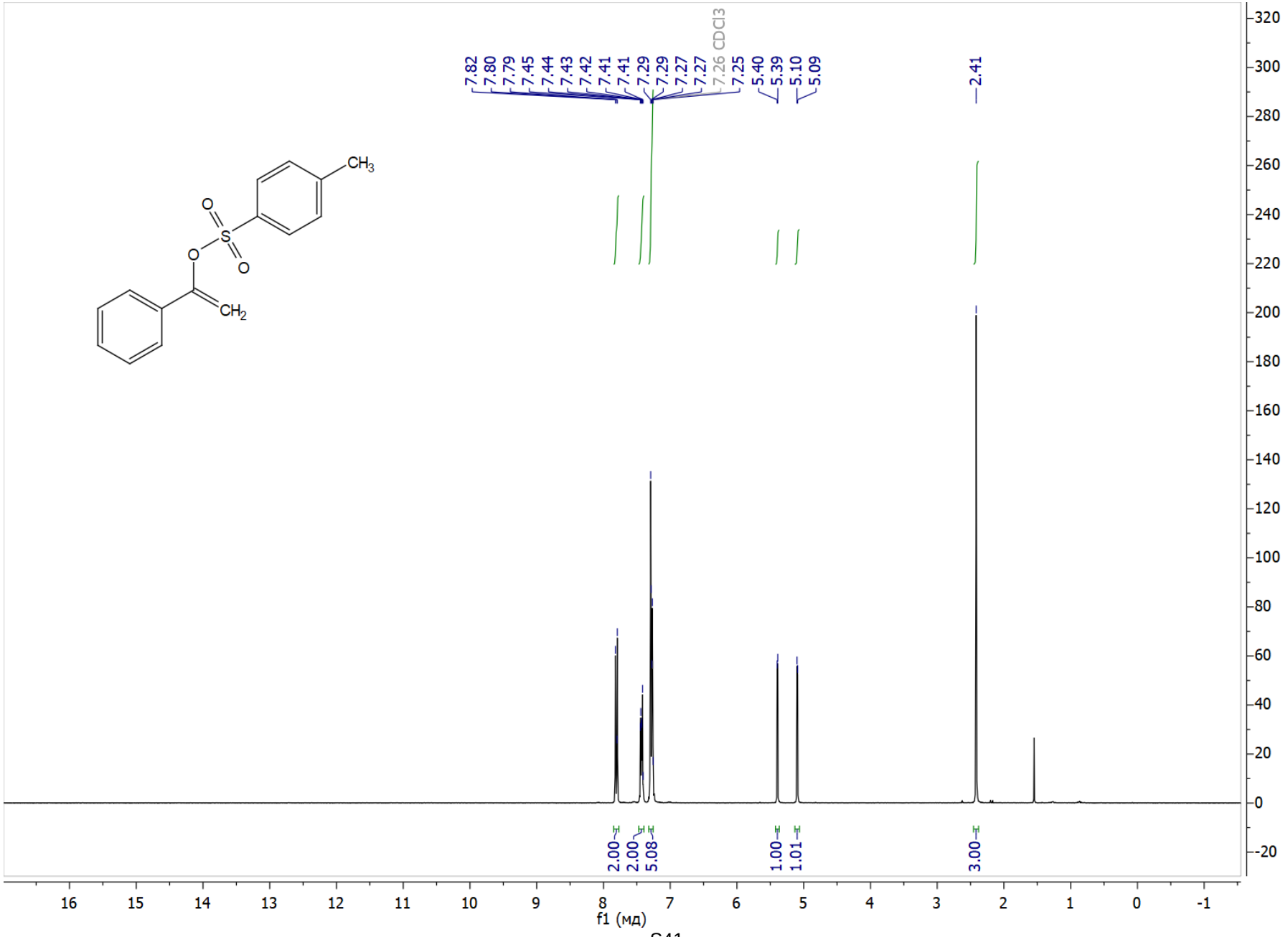
^{1}H NMR (300.13 MHz, $CDCl_3$), ***Tert*-butyldimethyl((1-phenylvinyl)oxy)silane, S3**



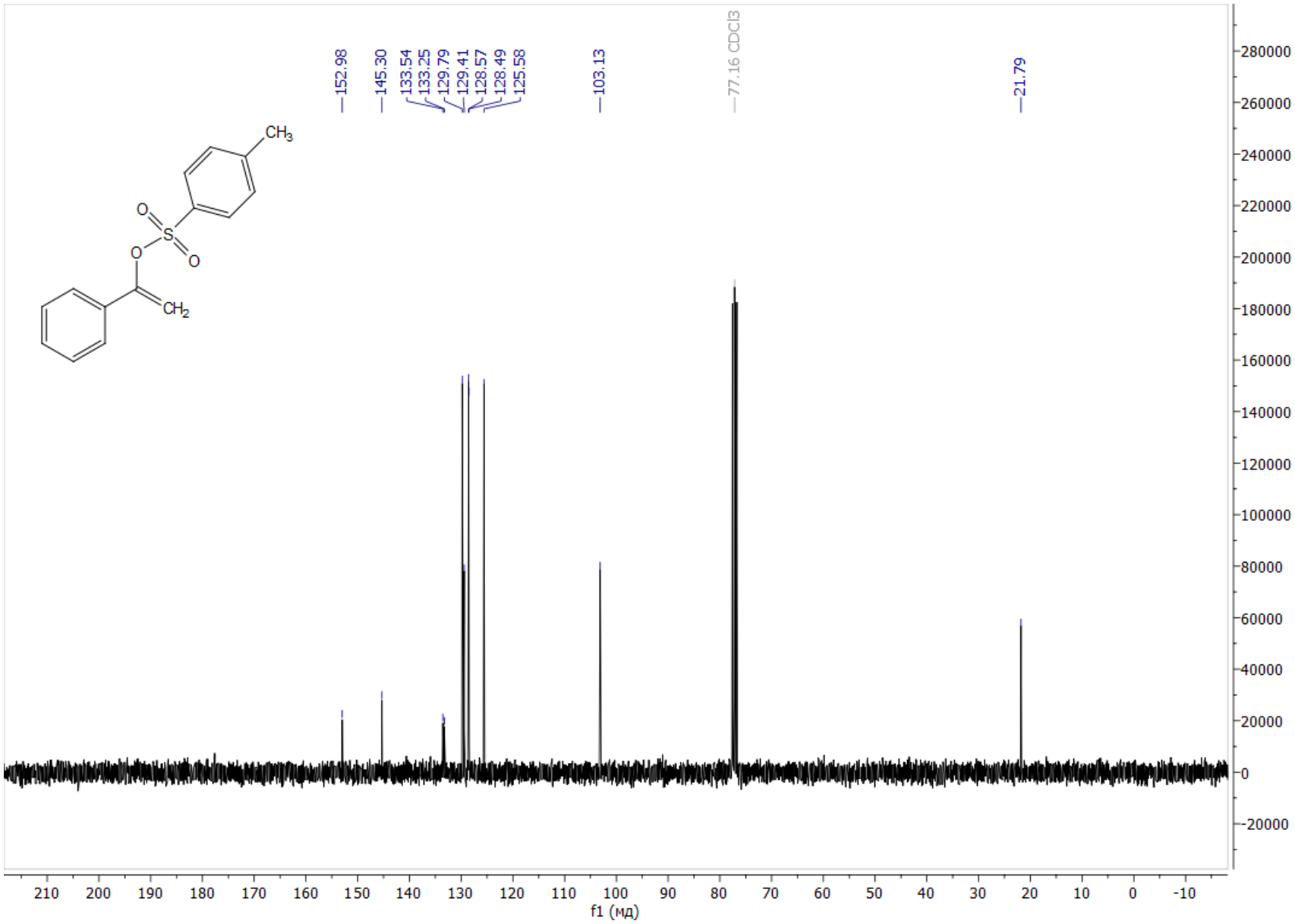
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), ***Tert*-butyldimethyl((1-phenylvinyl)oxy)silane, S3**



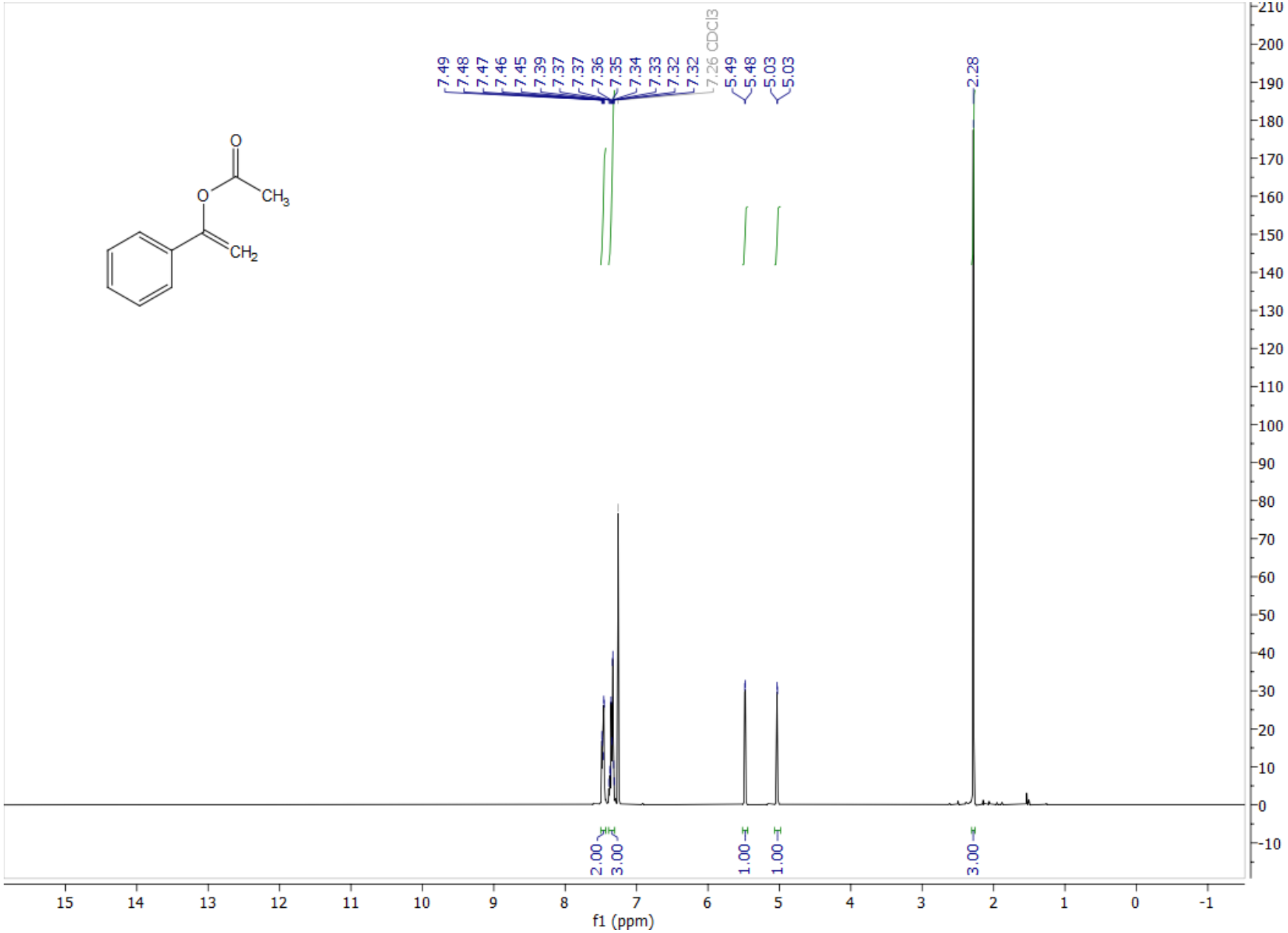
^{1}H NMR (300.13 MHz, $CDCl_3$), **1-Phenylvinyl 4-methylbenzenesulfonate, S4**



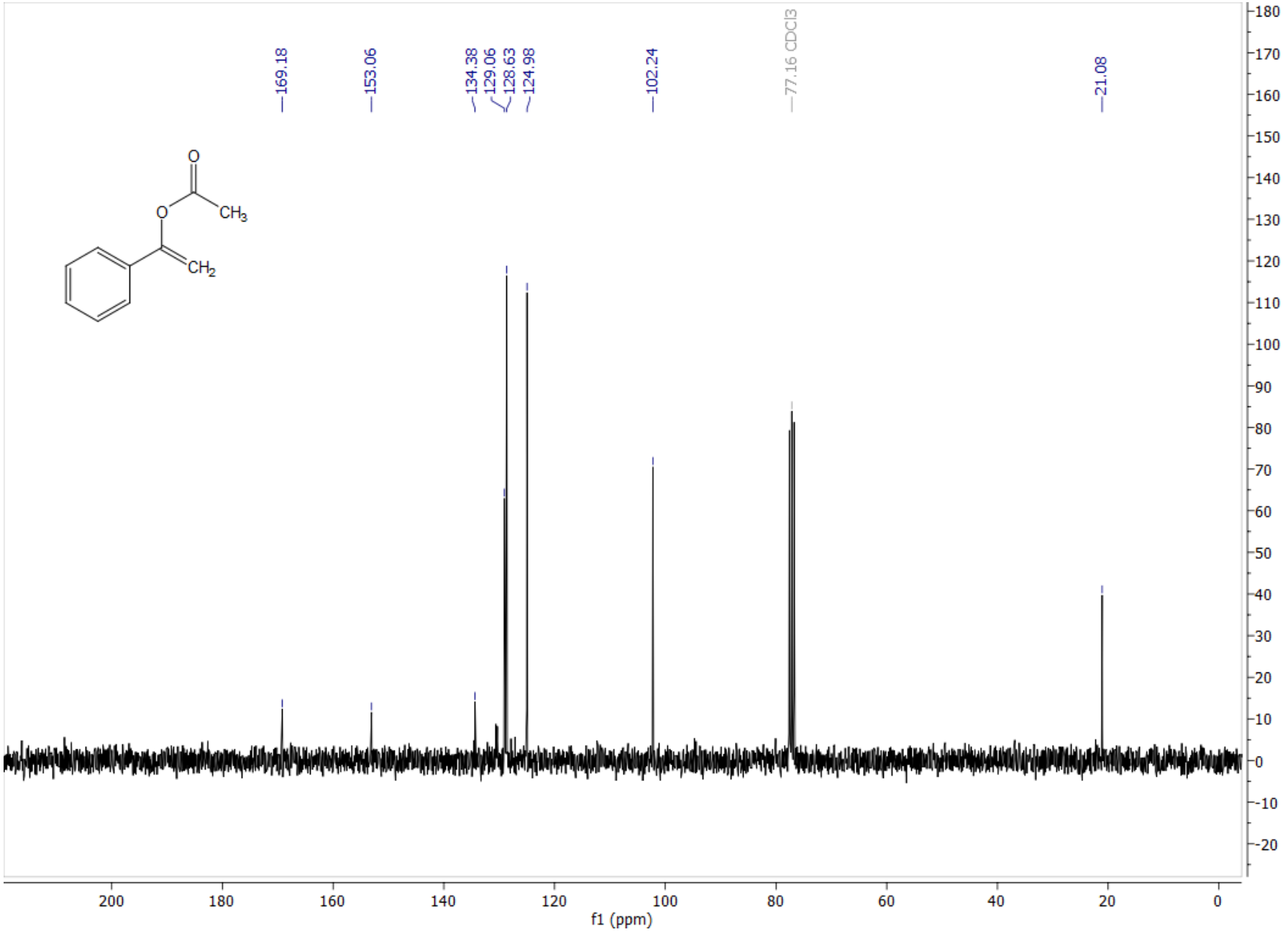
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **1-Phenylvinyl 4-methylbenzenesulfonate, S4**



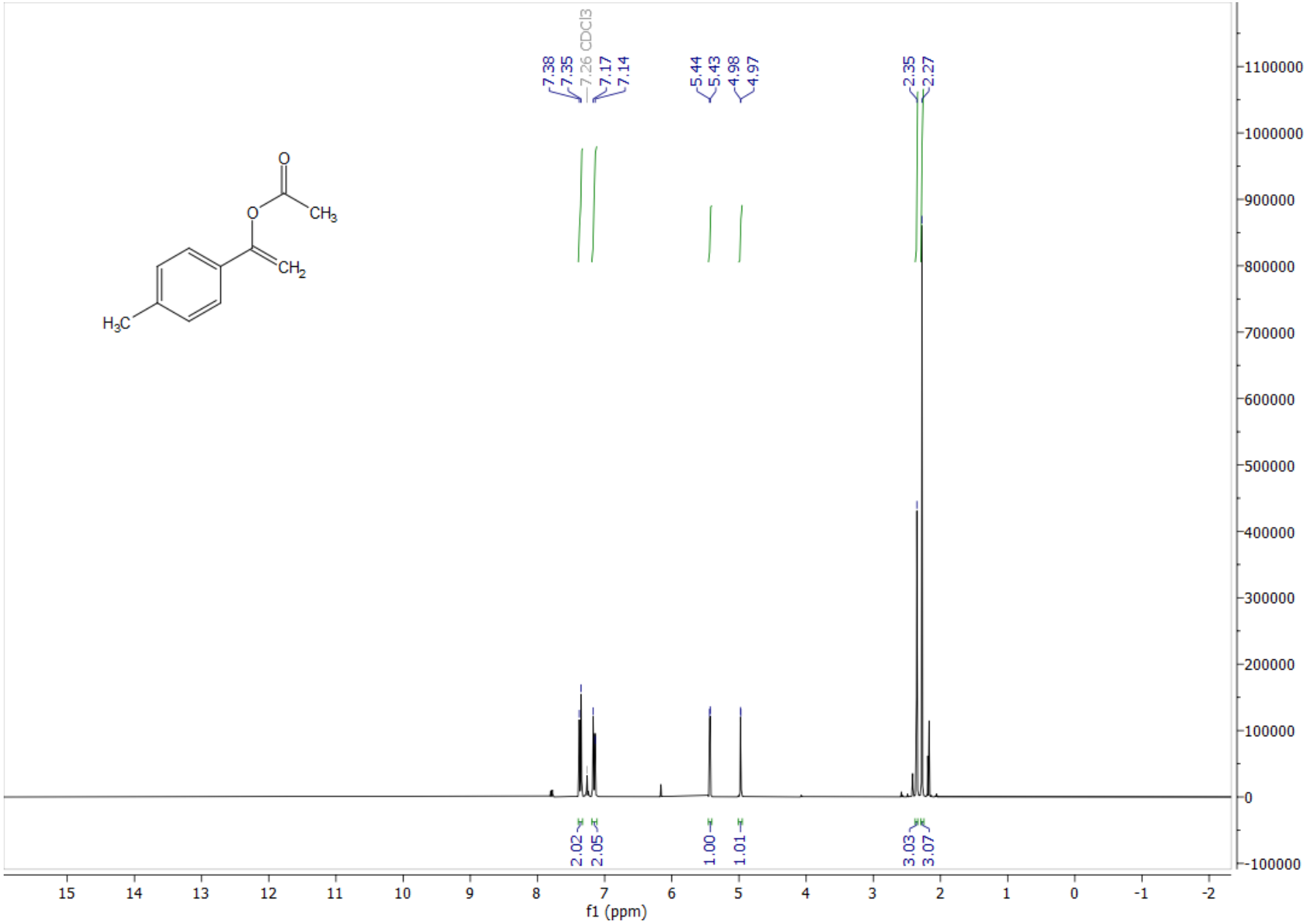
^{1}H NMR (300.13 MHz, CDCl3),**1-Phenylvinyl acetate, 1a**



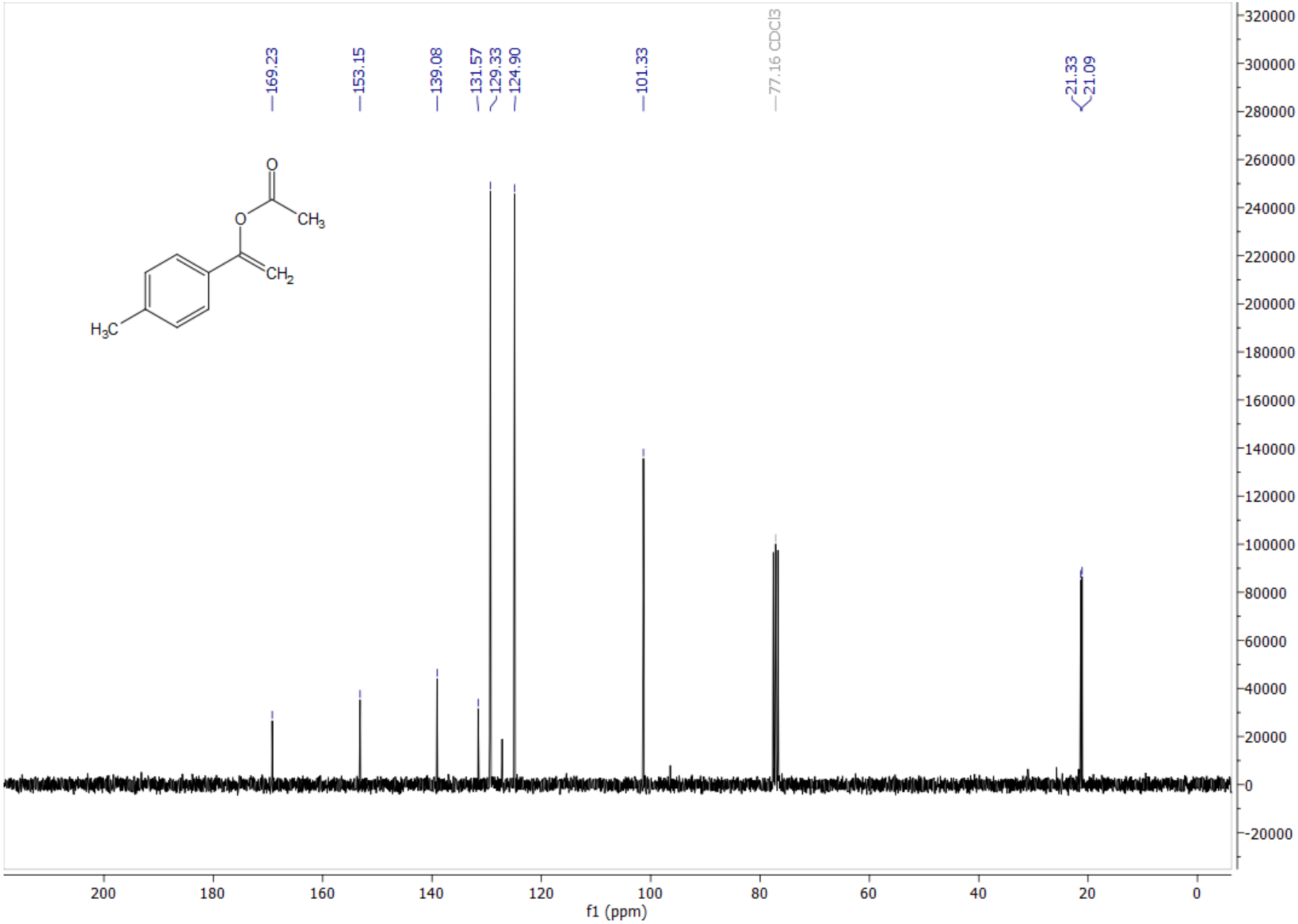
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **1-Phenylvinyl acetate, 1a**



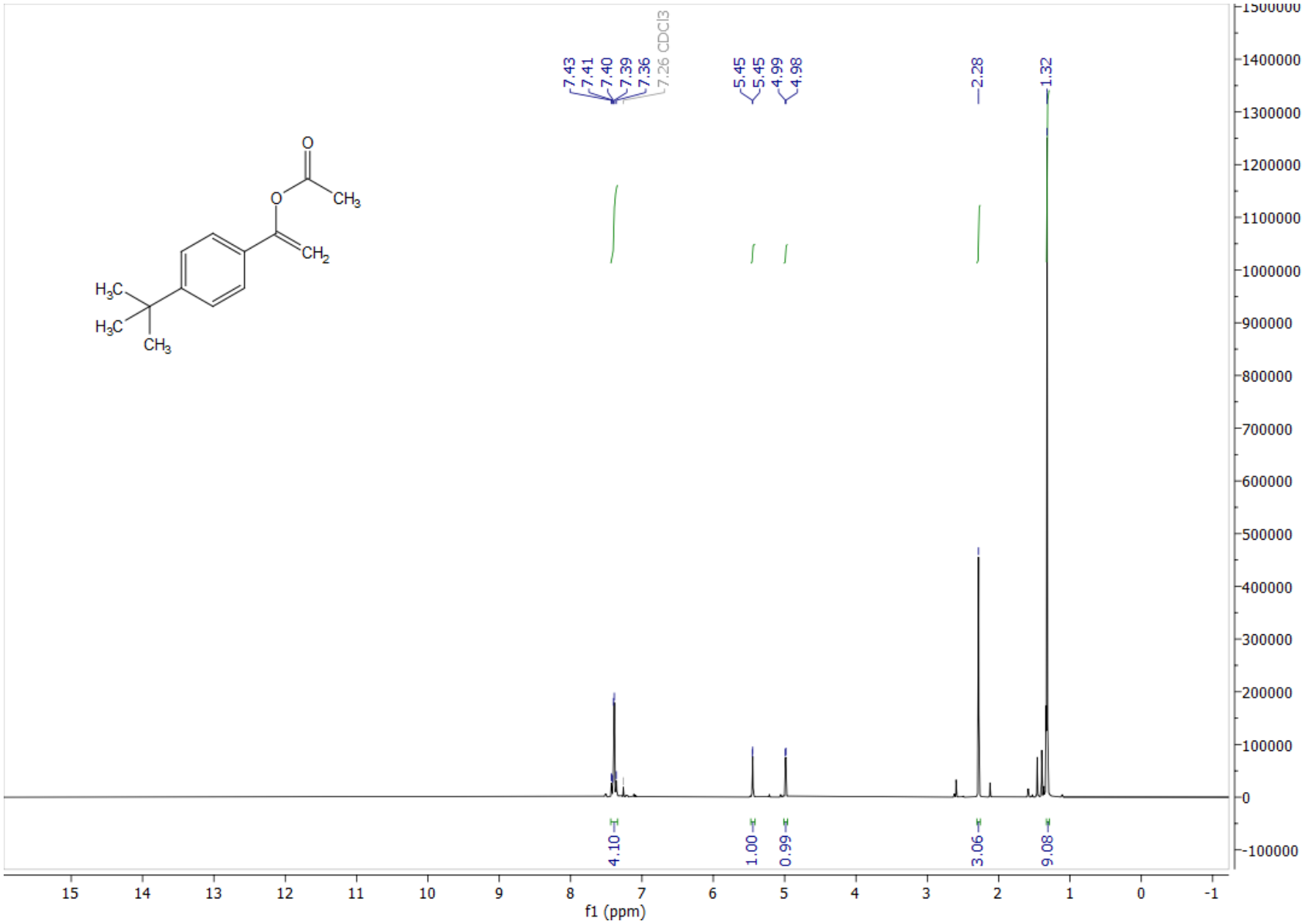
^{1}H NMR (300.13 MHz, CDCl3)**, 1-(*p*-Tolyl)vinyl acetate, 1b**



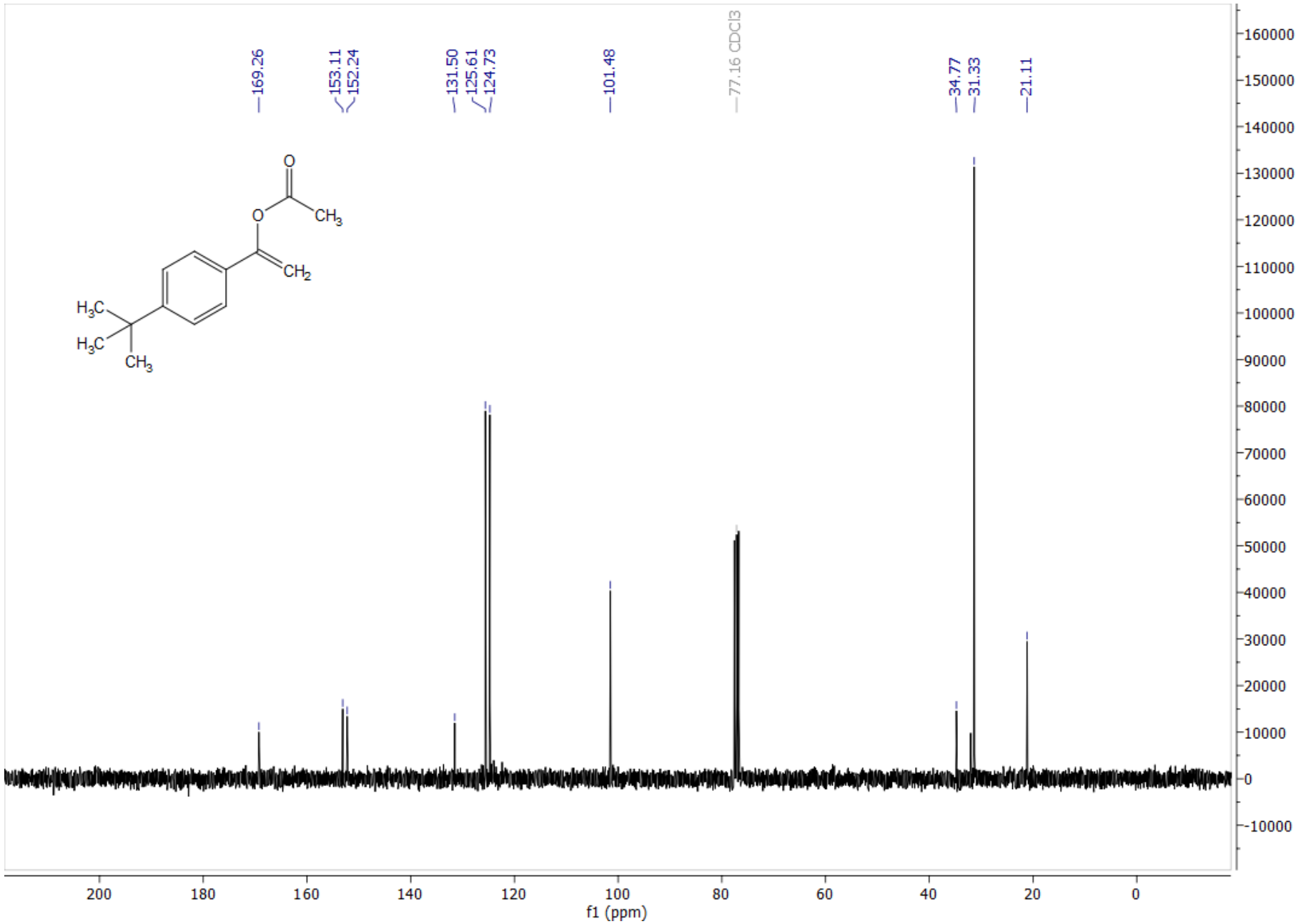
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$),**1-(*p*-Tolyl)vinyl acetate, 1b**



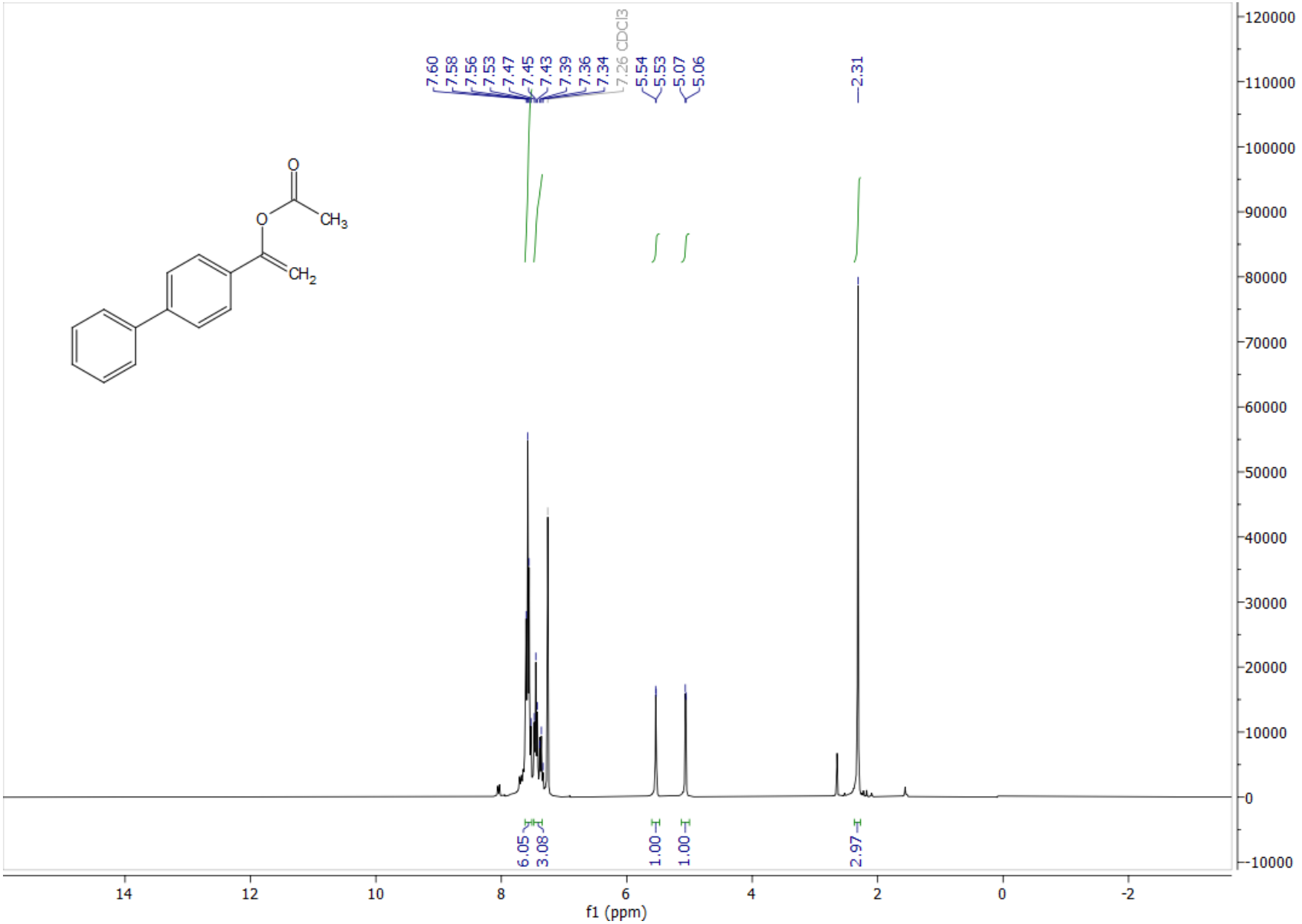
^{1}H NMR (300.13 MHz, $CDCl_3$), **1-(4-(*Tert*-butyl)phenyl)vinyl acetate, 1c**



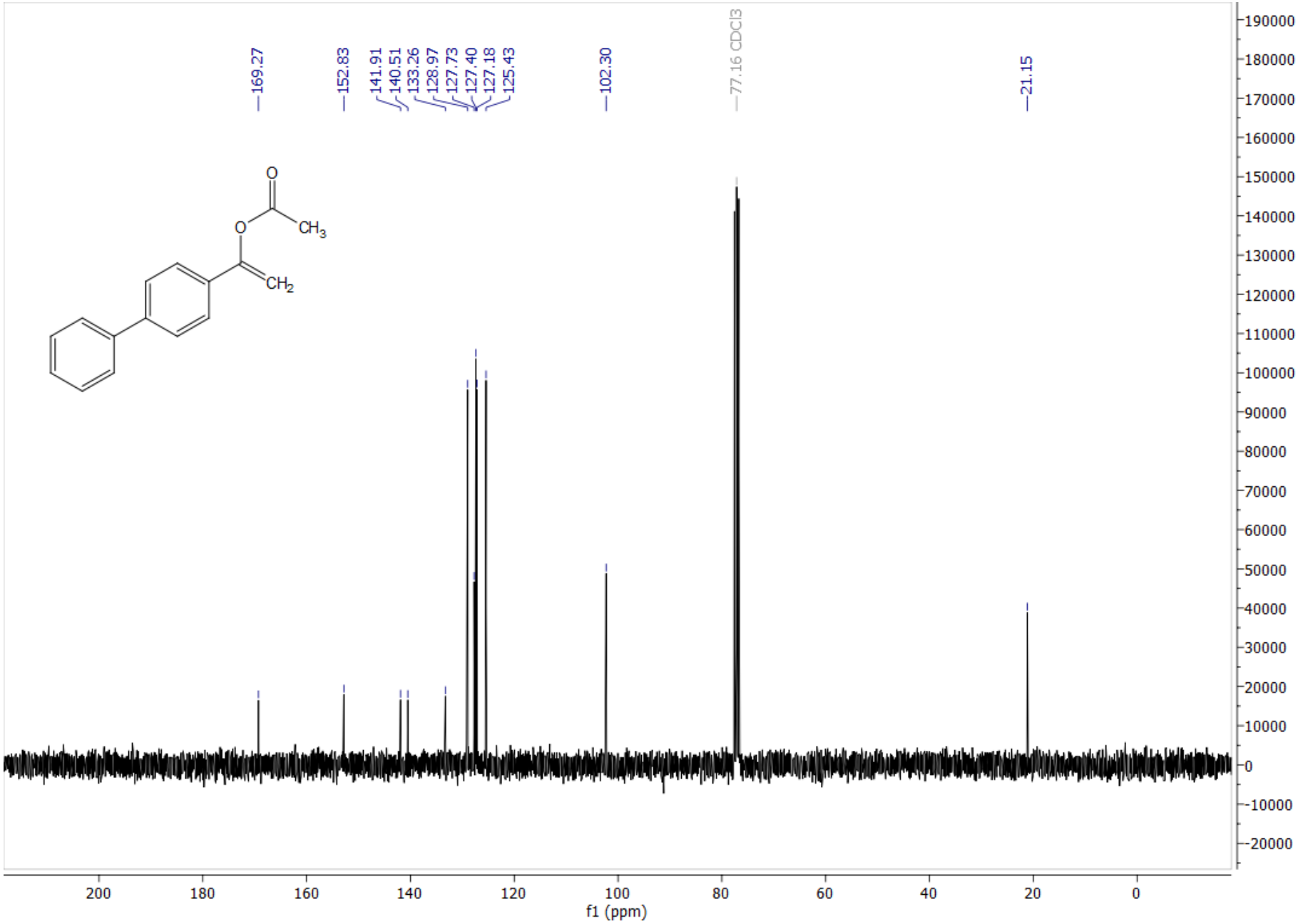
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **1-(4-(*Tert*-butyl)phenyl)vinyl acetate, 1c**



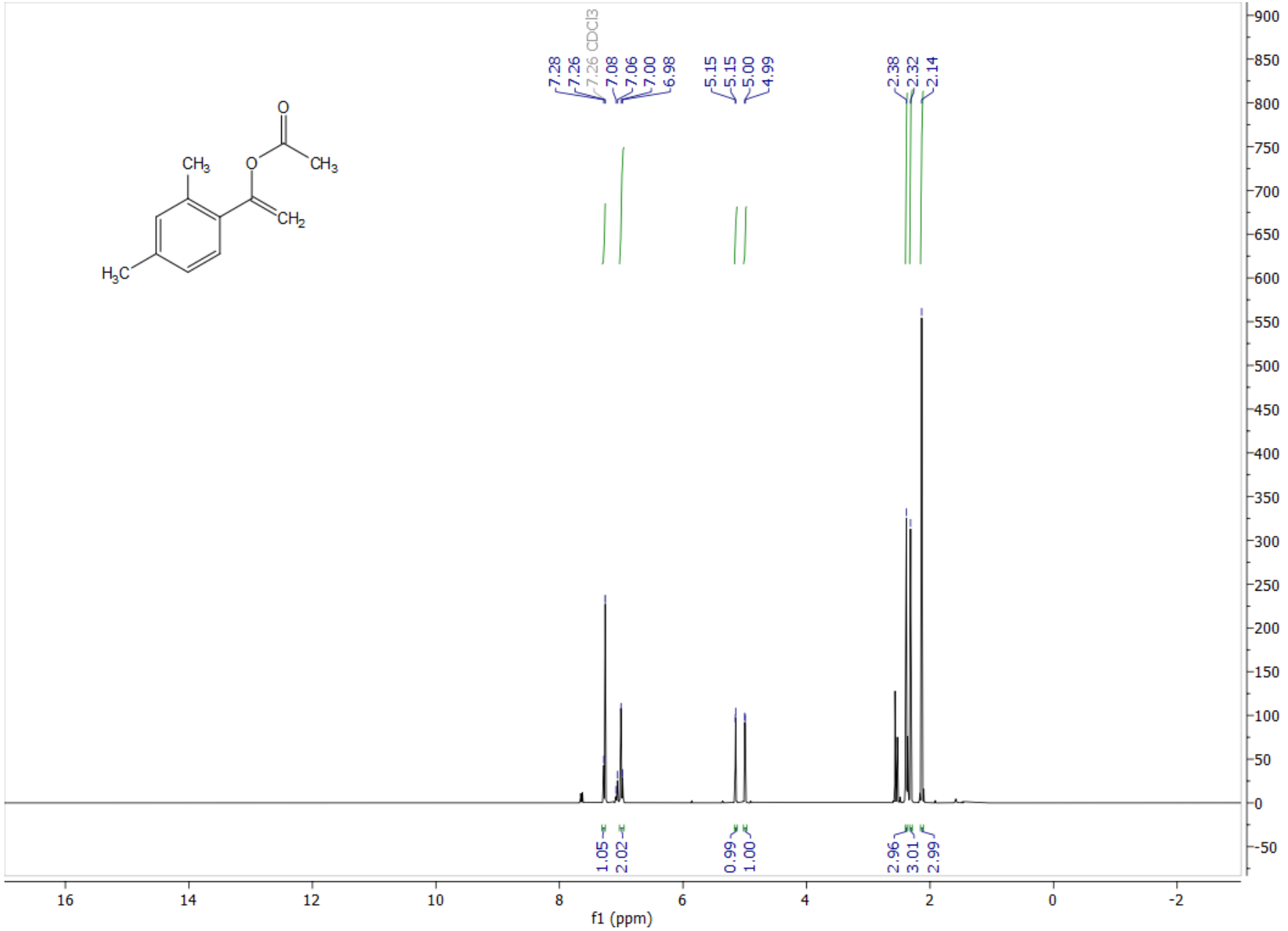
^{1}H NMR (300.13 MHz, $CDCl_3$), **1-([1,1'-Biphenyl]-4-yl)vinyl acetate, 1d**



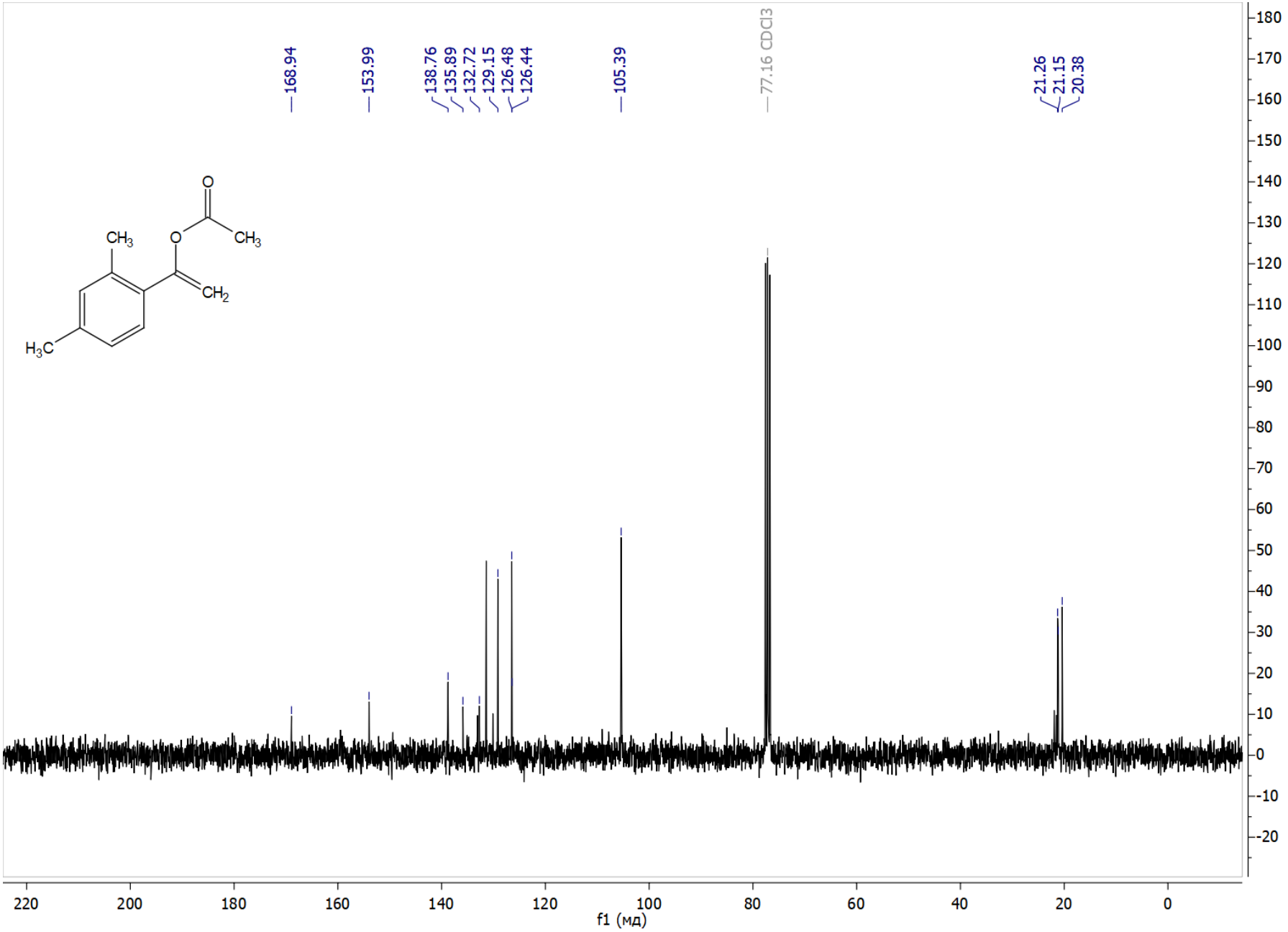
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **1-([1,1'-Biphenyl]-4-yl)vinyl acetate, 1d**



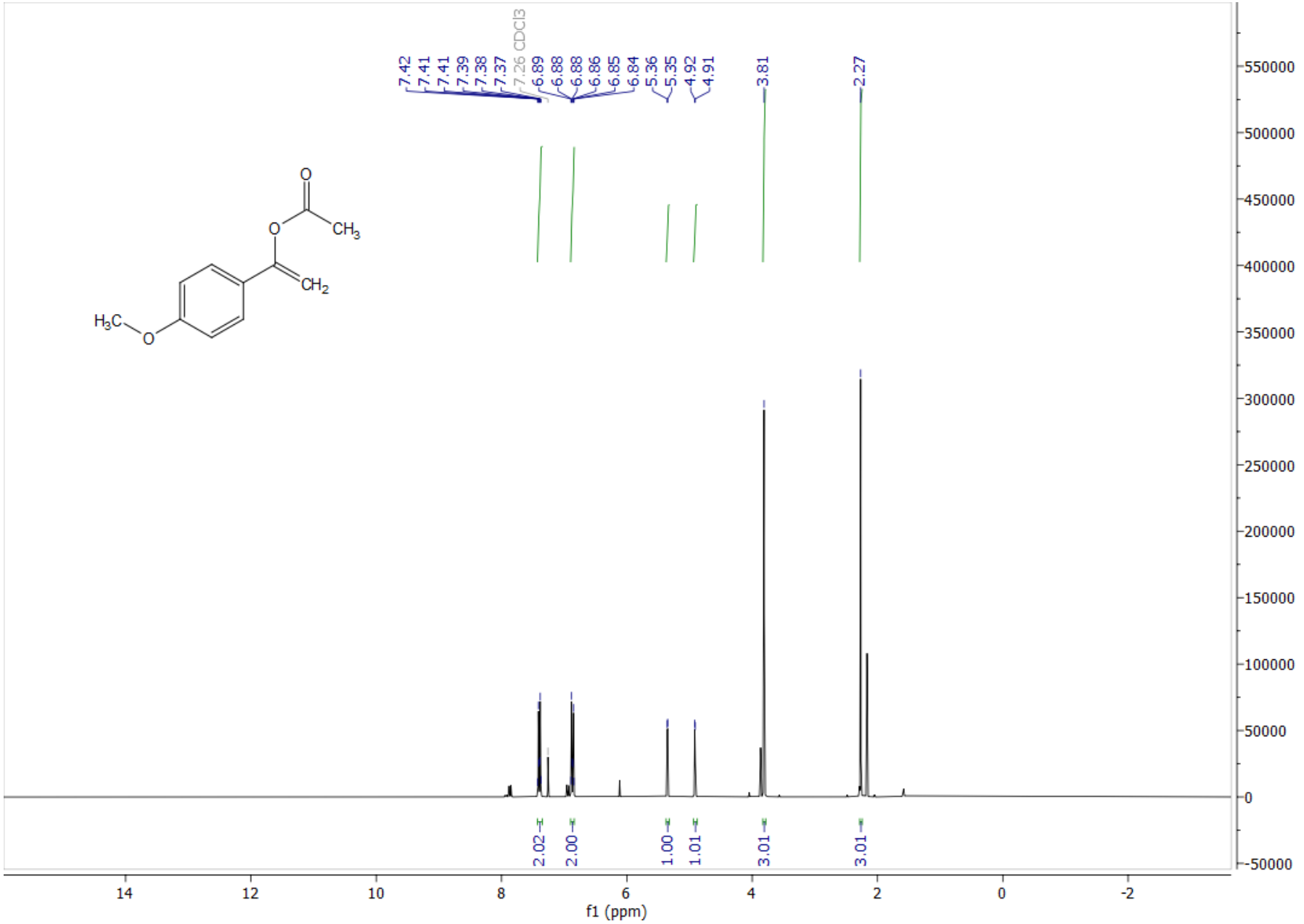
^{1}H NMR (300.13 MHz, $CDCl_3$), **1-(2,4-Dimethylphenyl)vinyl acetate1 1e**



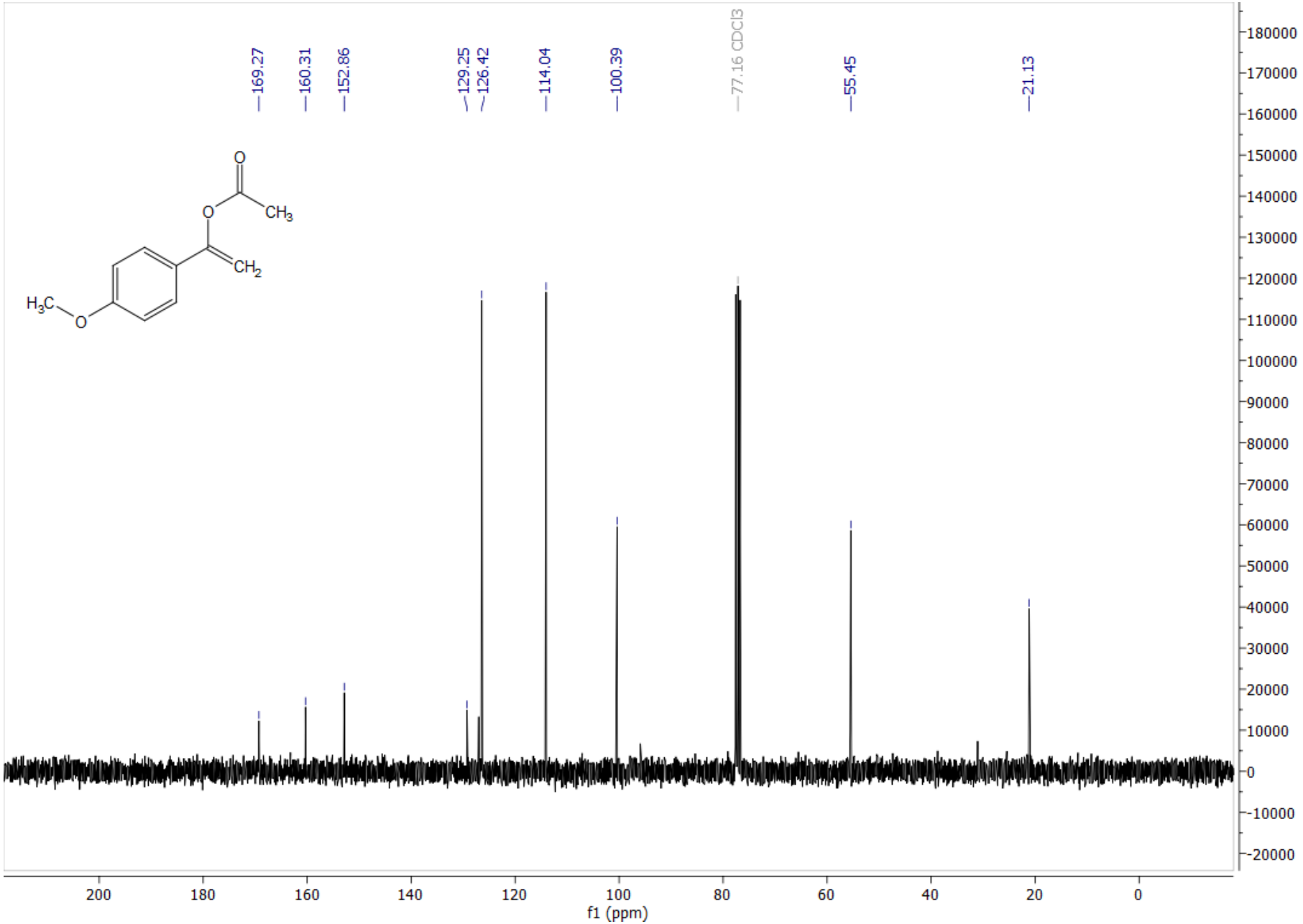
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **1-(2,4-Dimethylphenyl)vinyl acetate, 1e**



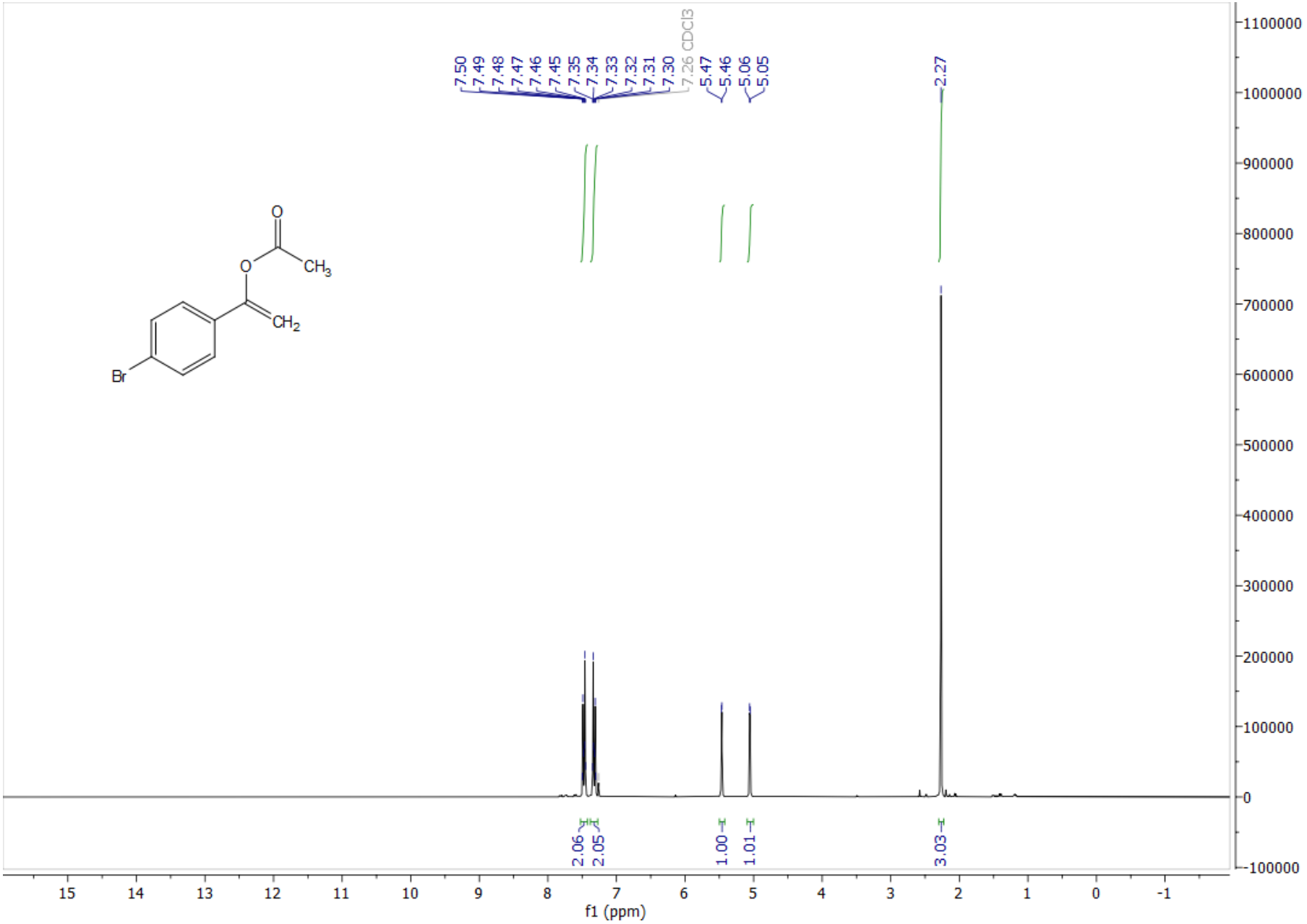
^{1}H NMR (300.13 MHz, $CDCl_3$), **1-(4-Methoxyphenyl)vinyl acetate, 1f**



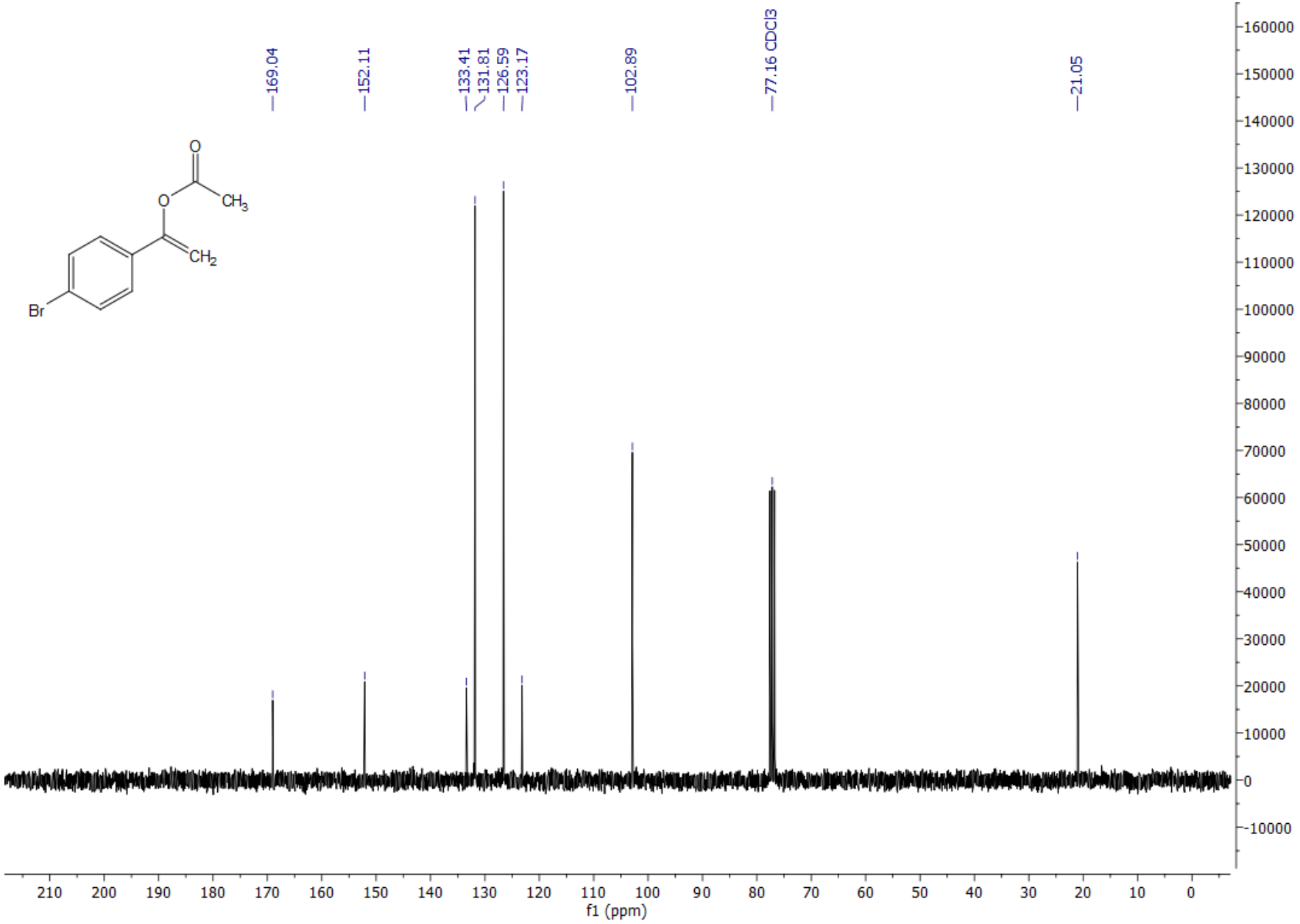
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **1-(4-Methoxyphenyl)vinyl acetate, 1f**



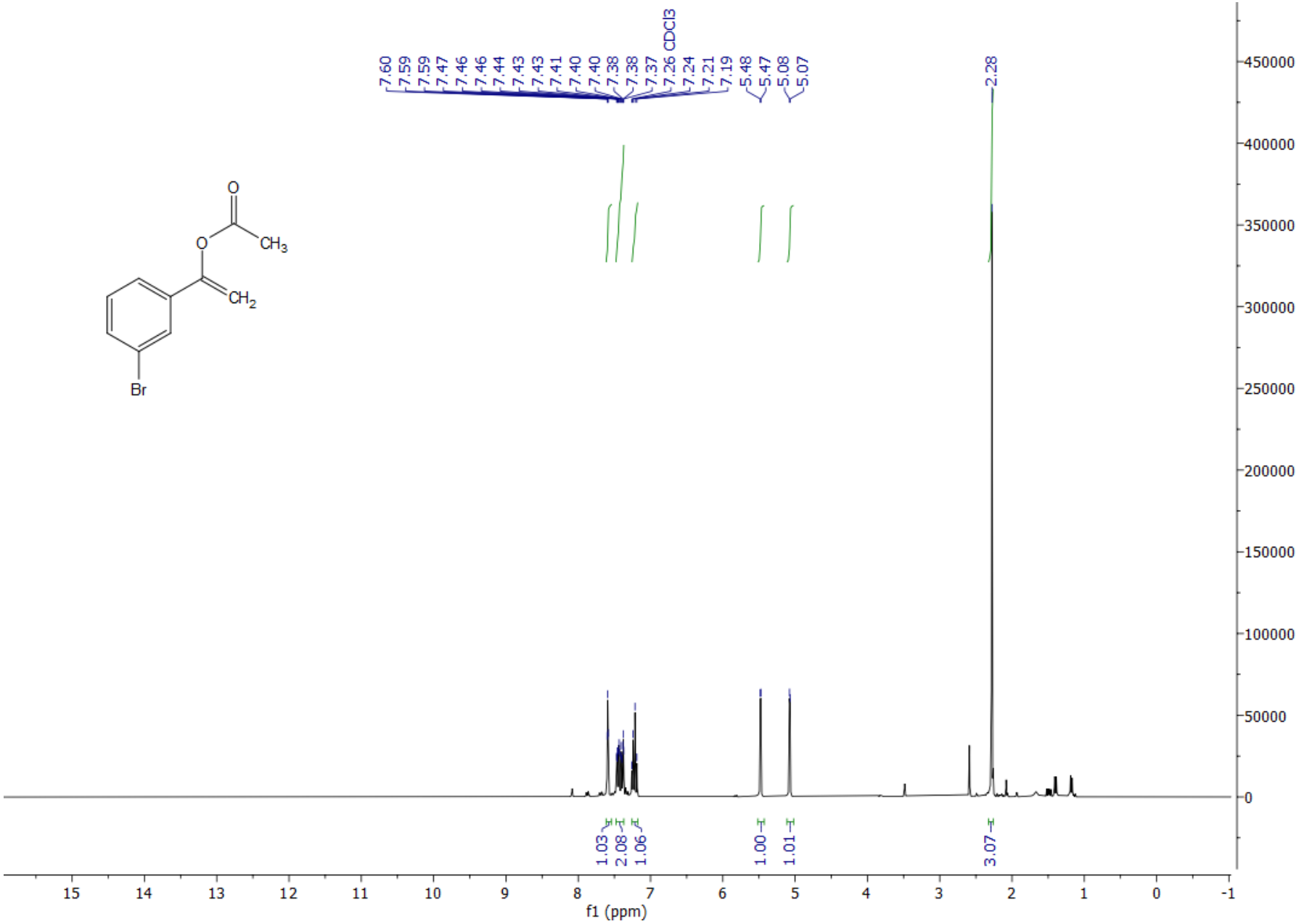
^{1}H NMR (300.13 MHz, $CDCl_3$), **1-(4-Bromophenyl)vinyl acetate, 1r**



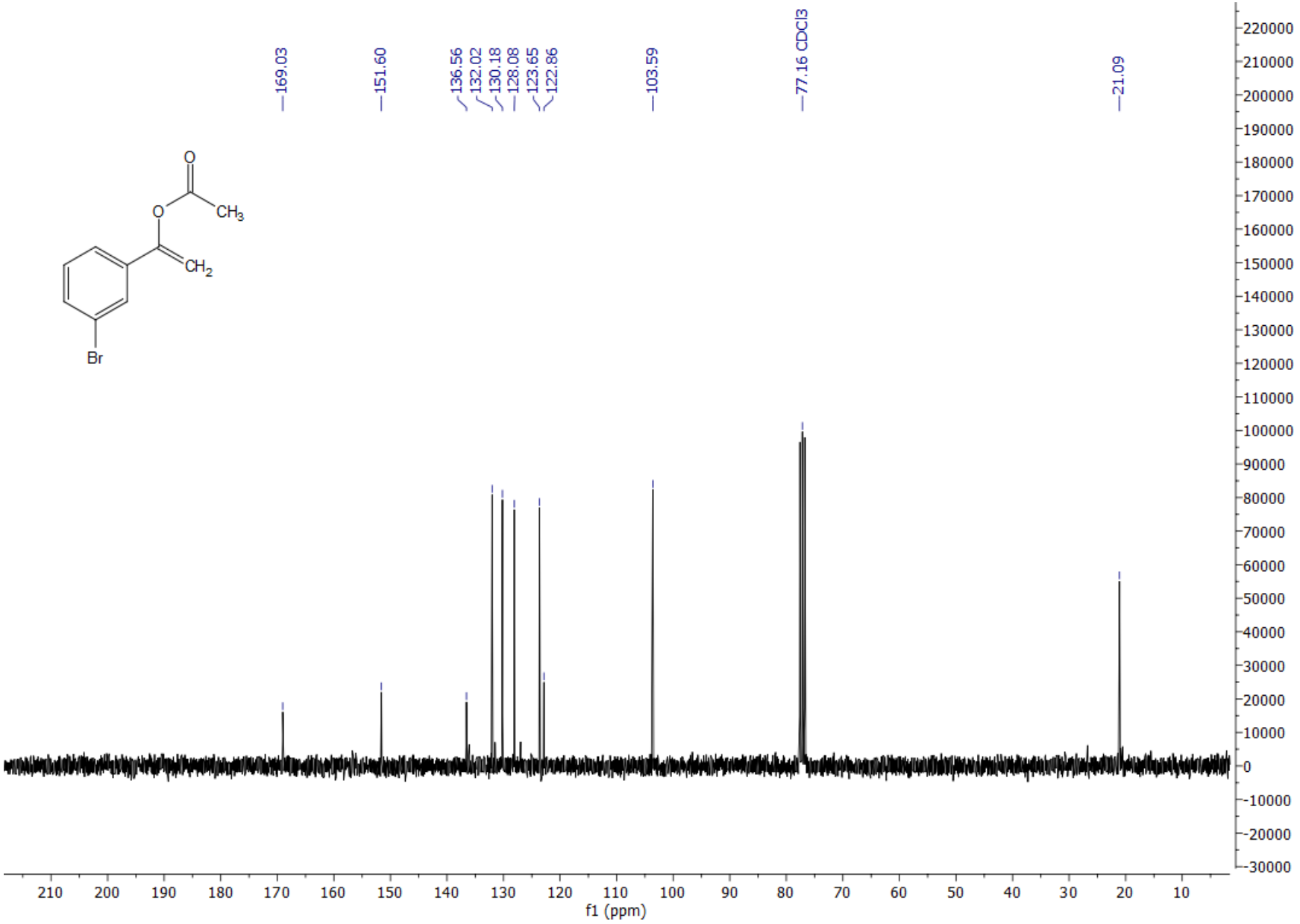
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **1-(4-Bromophenyl)vinyl acetate, 1r**



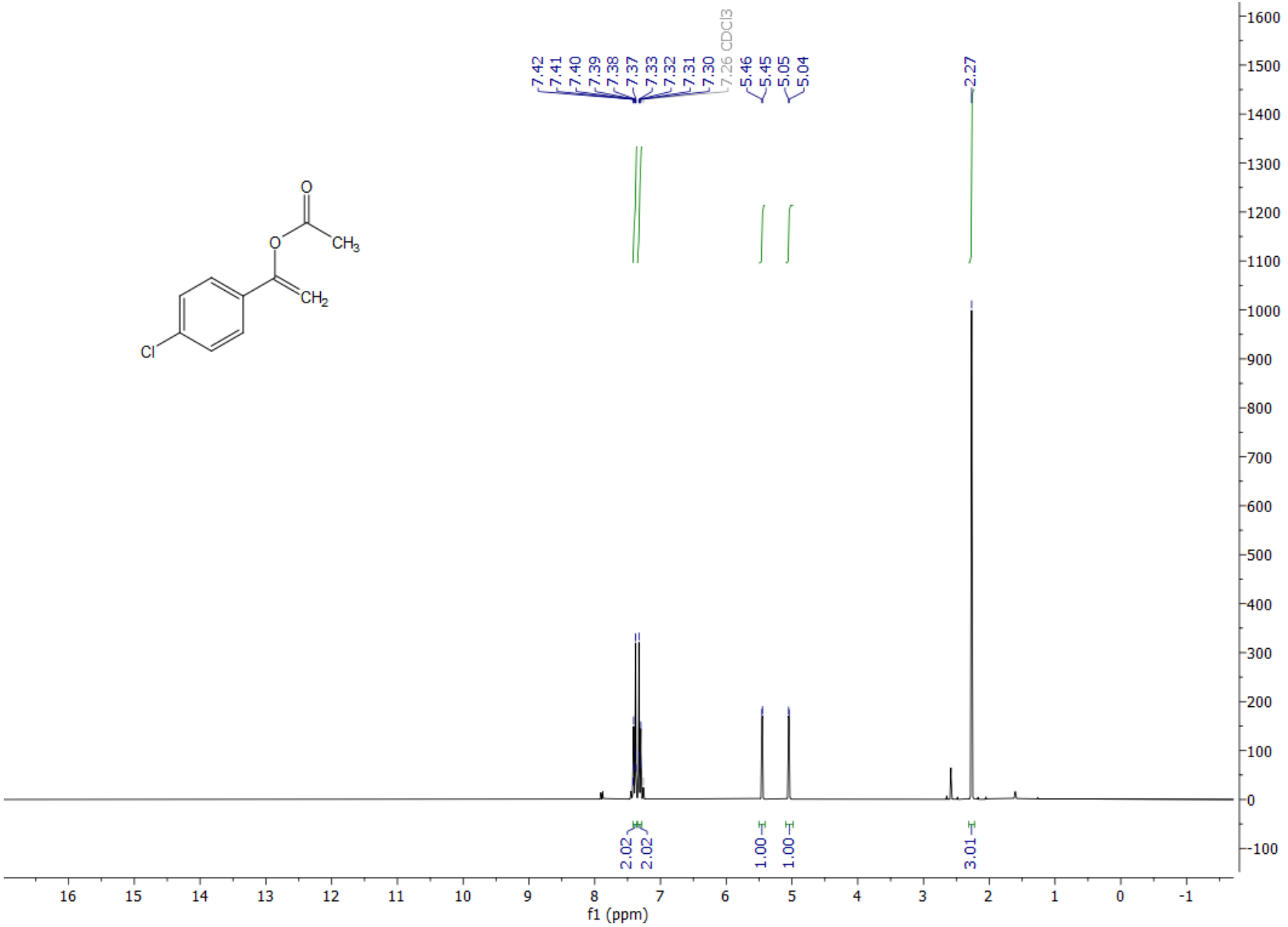
^{1}H NMR (300.13 MHz, $CDCl_3$), **1-(3-Bromophenyl)vinyl acetate, 1s**



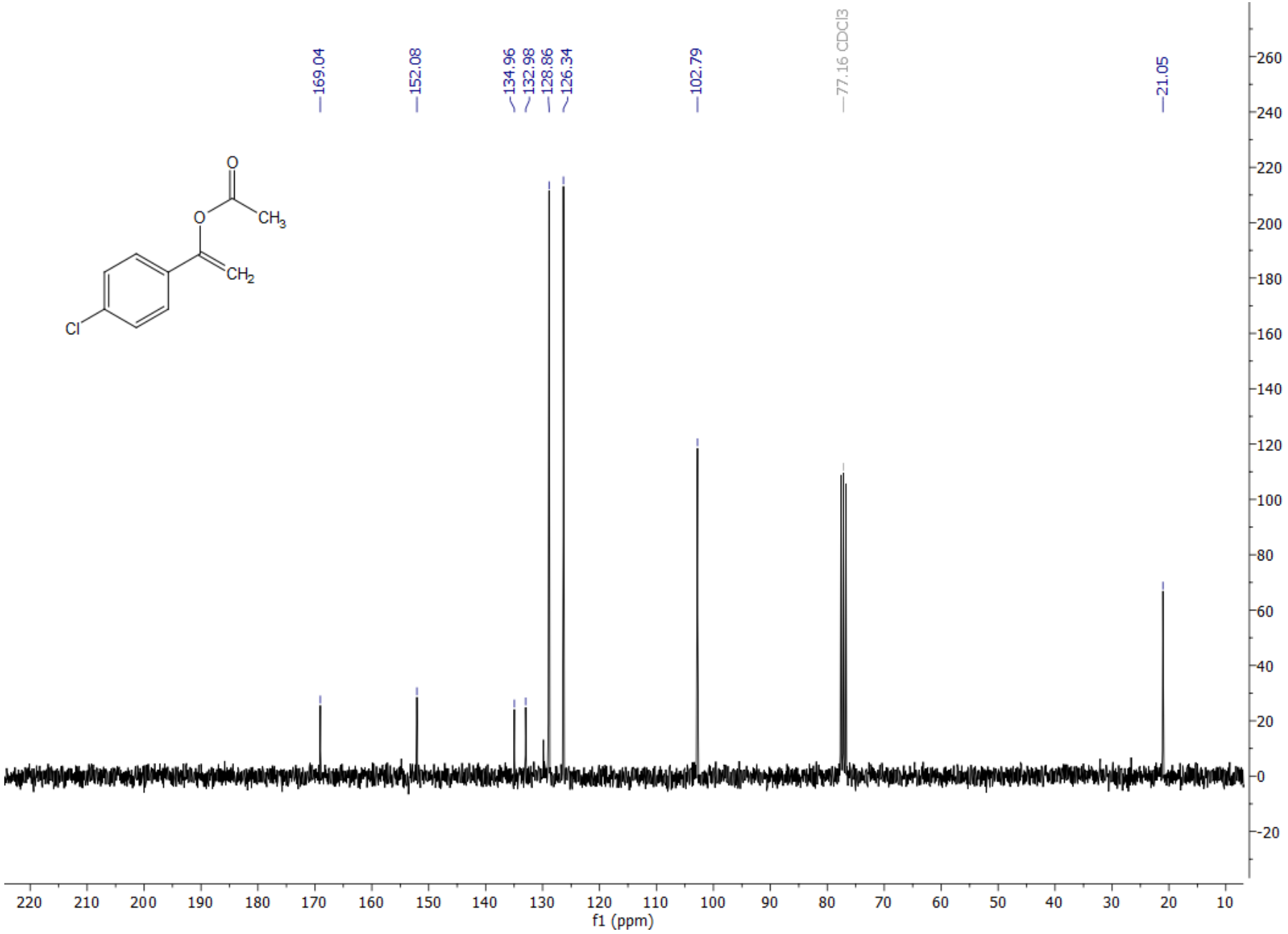
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **1-(3-Bromophenyl)vinyl acetate, 1s**



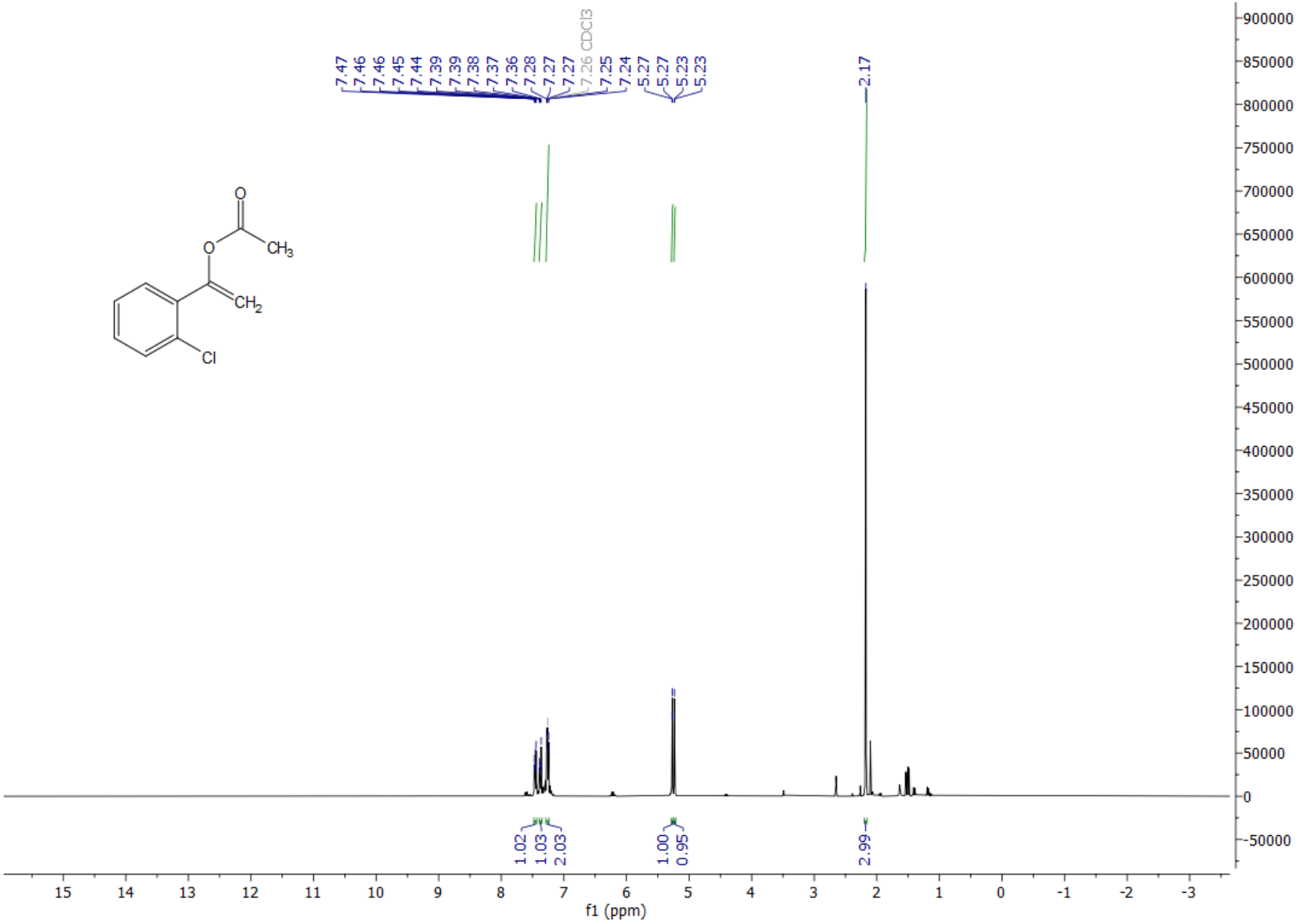
^{1}H NMR (300.13 MHz, $CDCl_3$), **1-(4-Chlorophenyl)vinyl acetate, 1u**



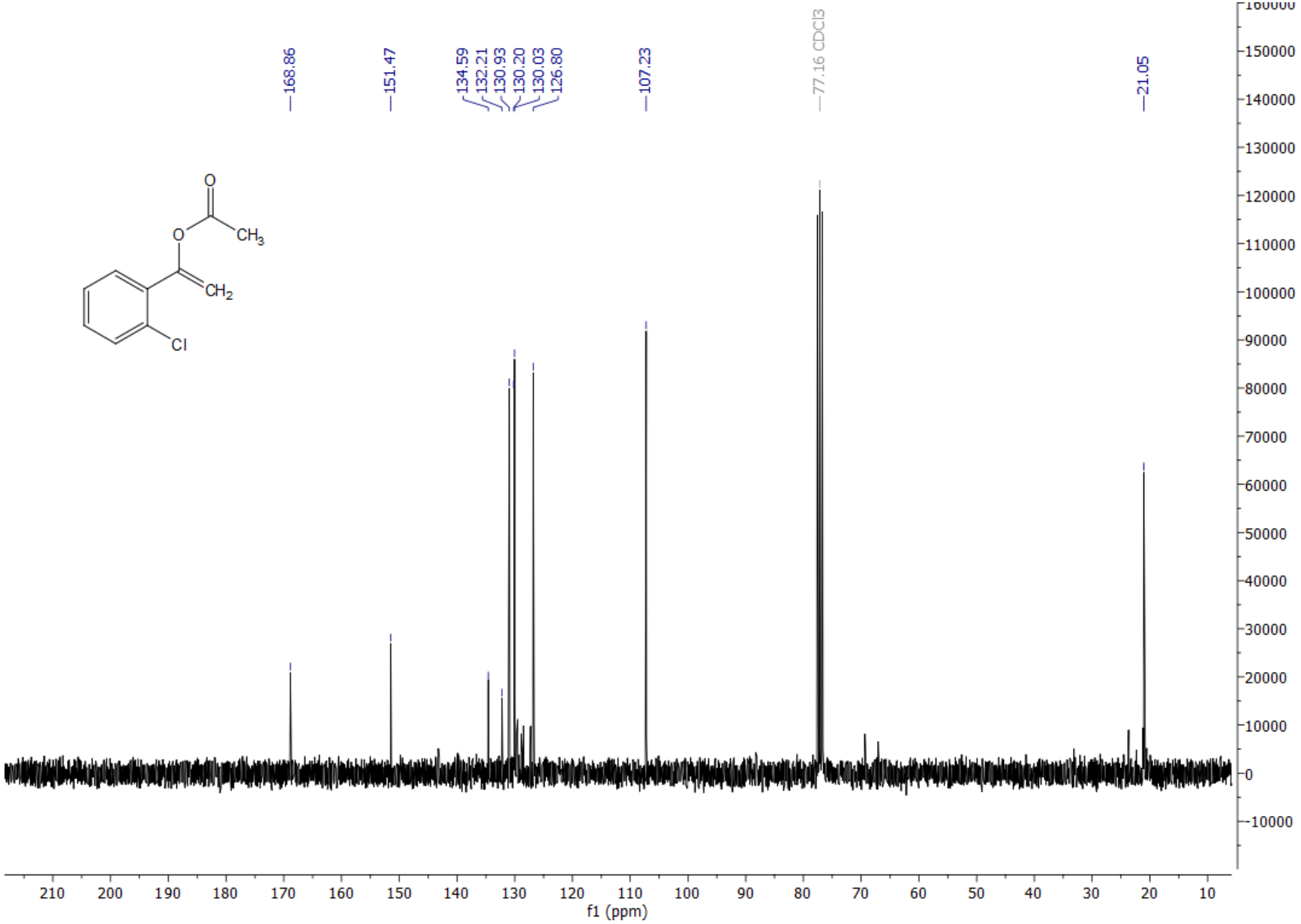
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **1-(4-Chlorophenyl)vinyl acetate, 1u**



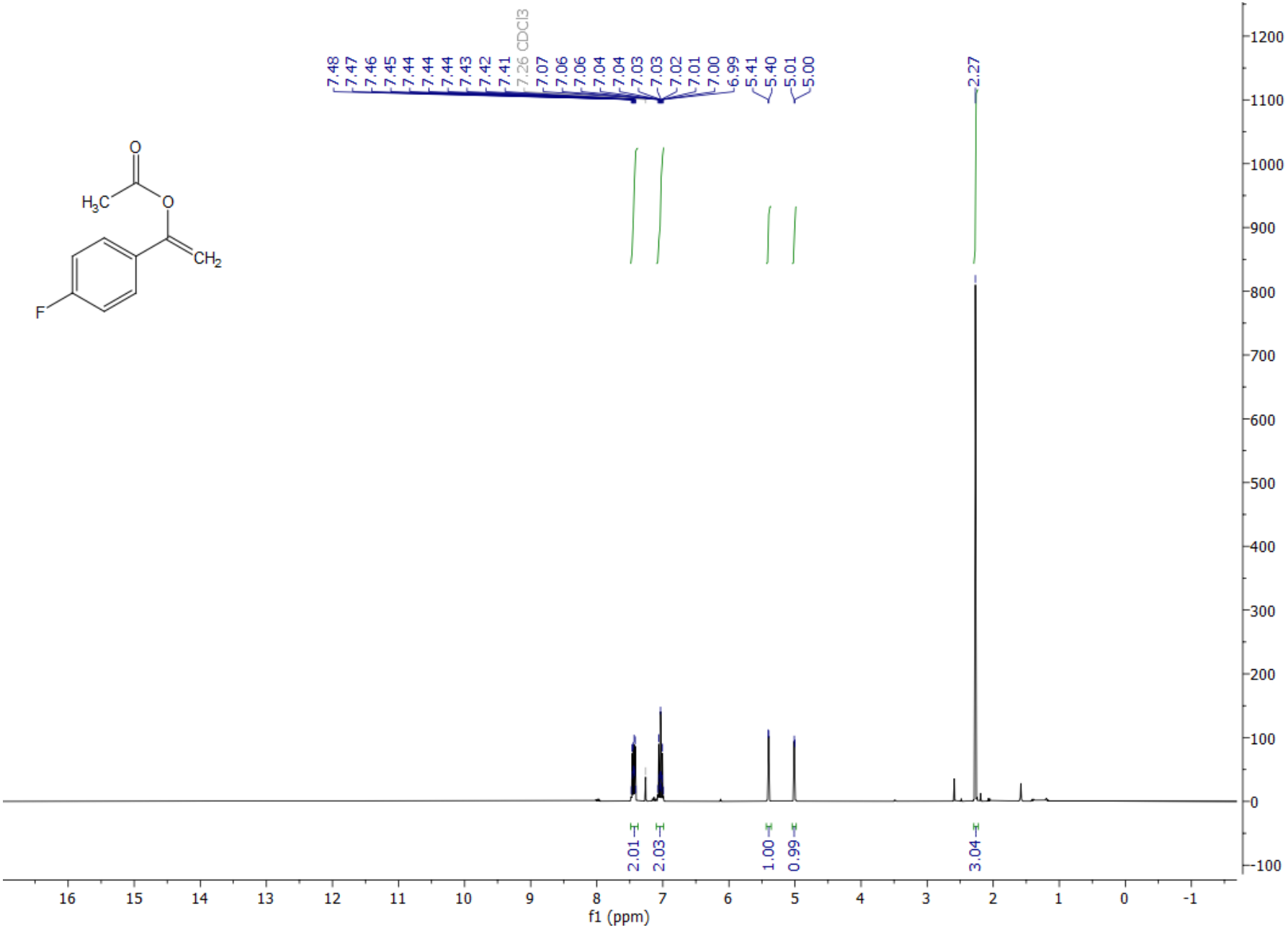
^{1}H NMR (300.13 MHz, $CDCl_3$), **1-(2-Chlorophenyl)vinyl acetate, 1v**



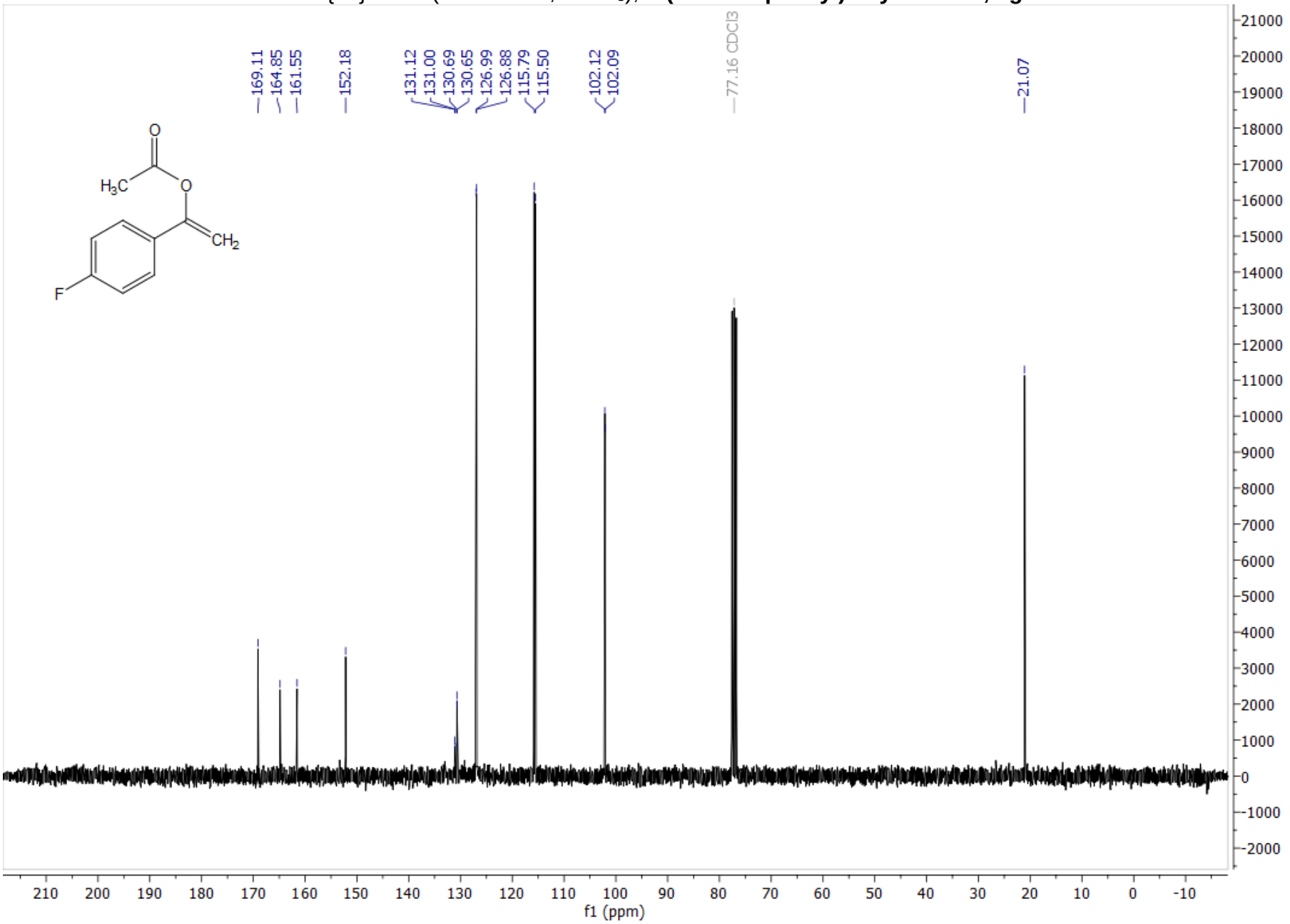
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$),**1-(2-Chlorophenyl)vinyl acetate, 1v**



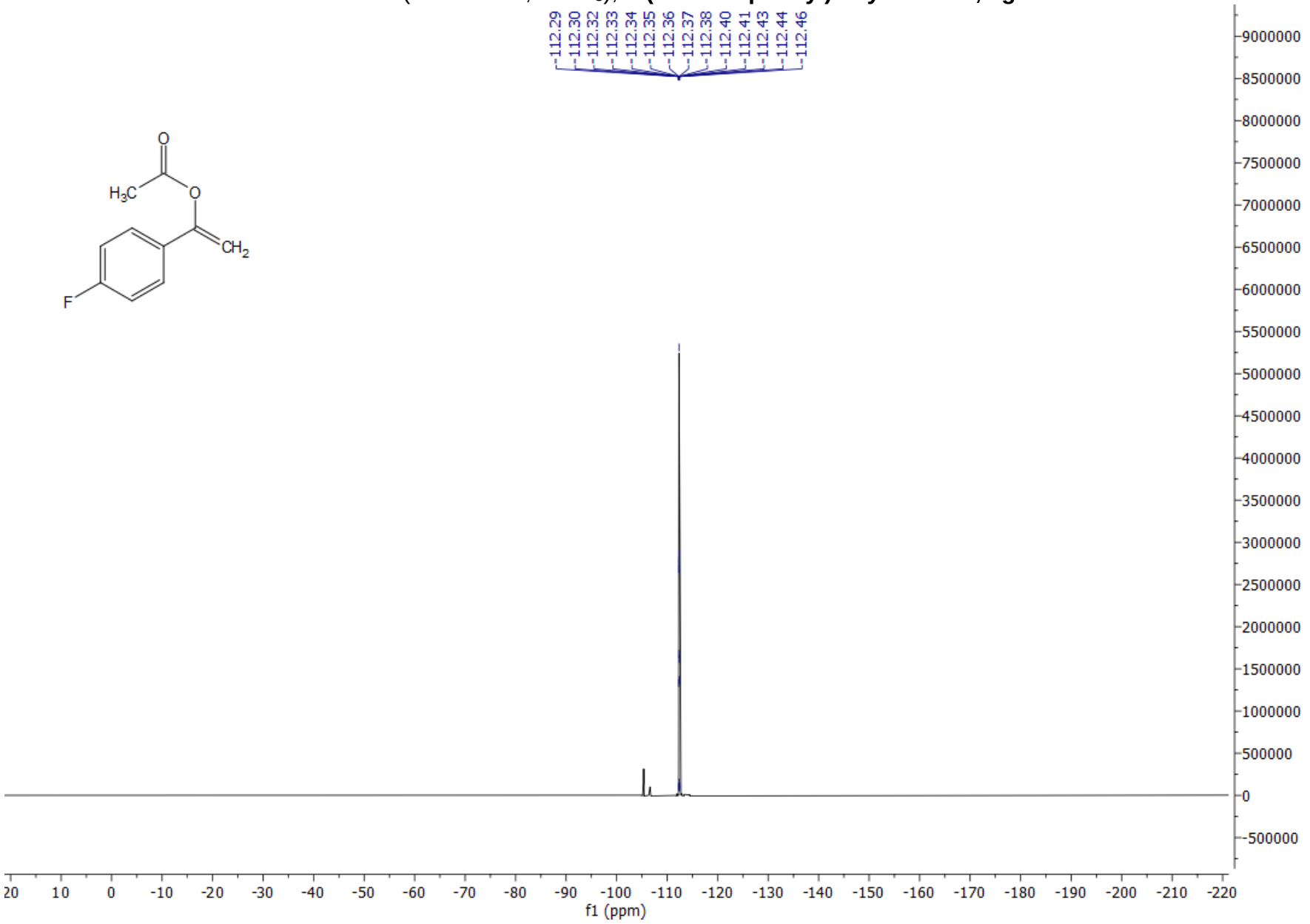
^{1}H NMR (300.13 MHz, $CDCl_3$), **1-(4-Fluorophenyl)vinyl acetate,1g**



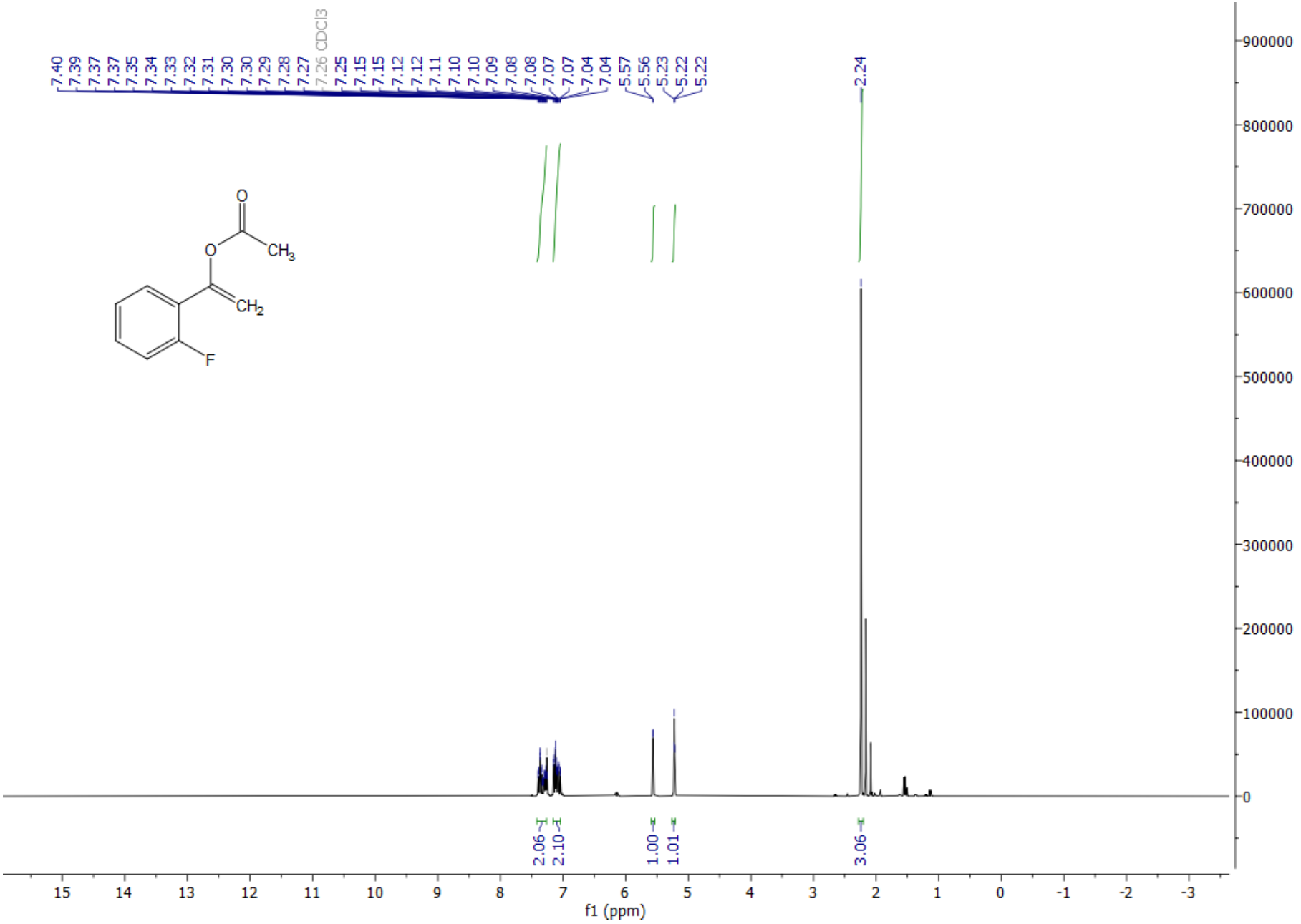
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **1-(4-Fluorophenyl)vinyl acetate, 1g**



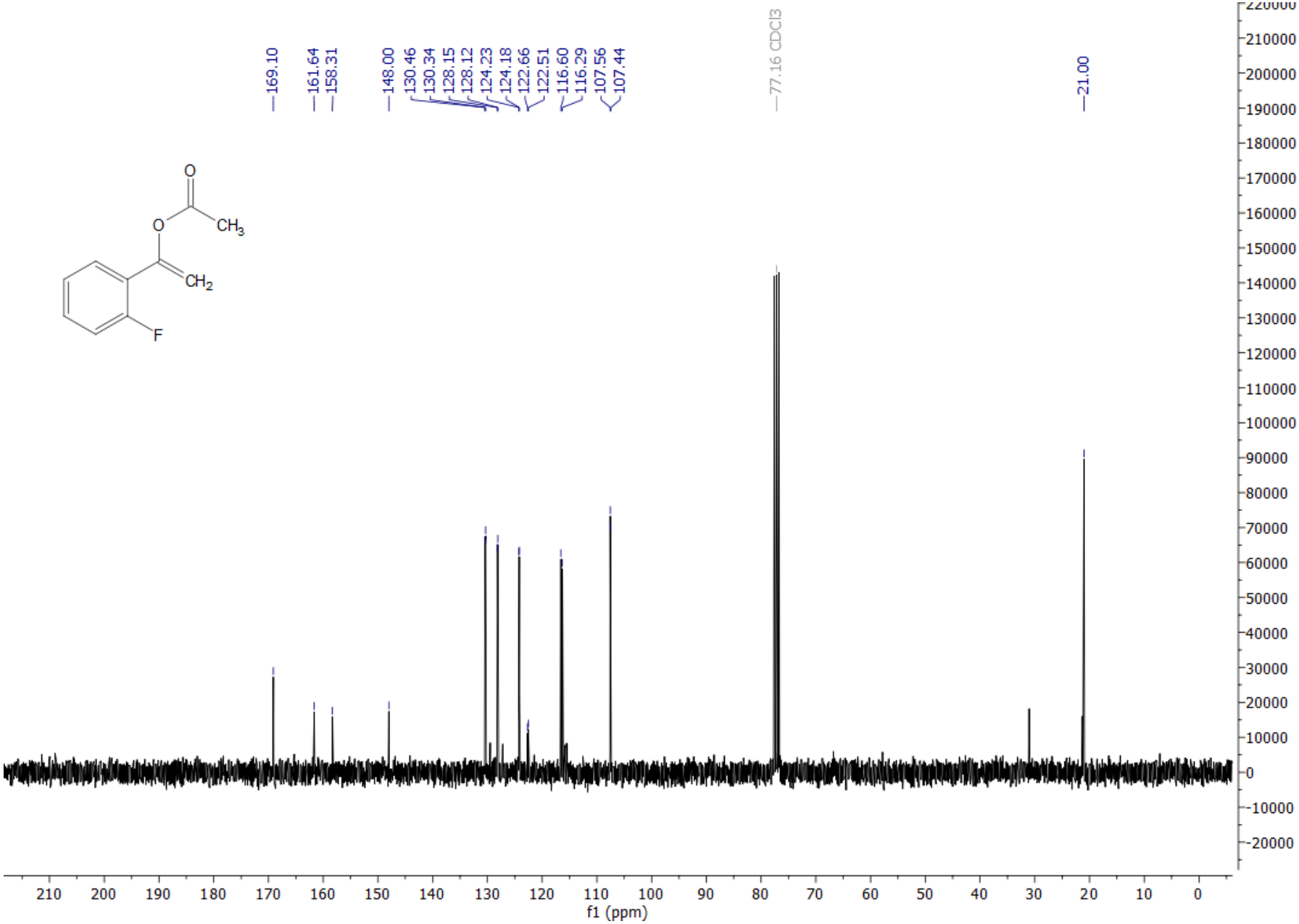
^{19}F NMR (282.5 MHz, $CDCl_3$), **1-(4-Fluorophenyl)vinyl acetate, 1g**



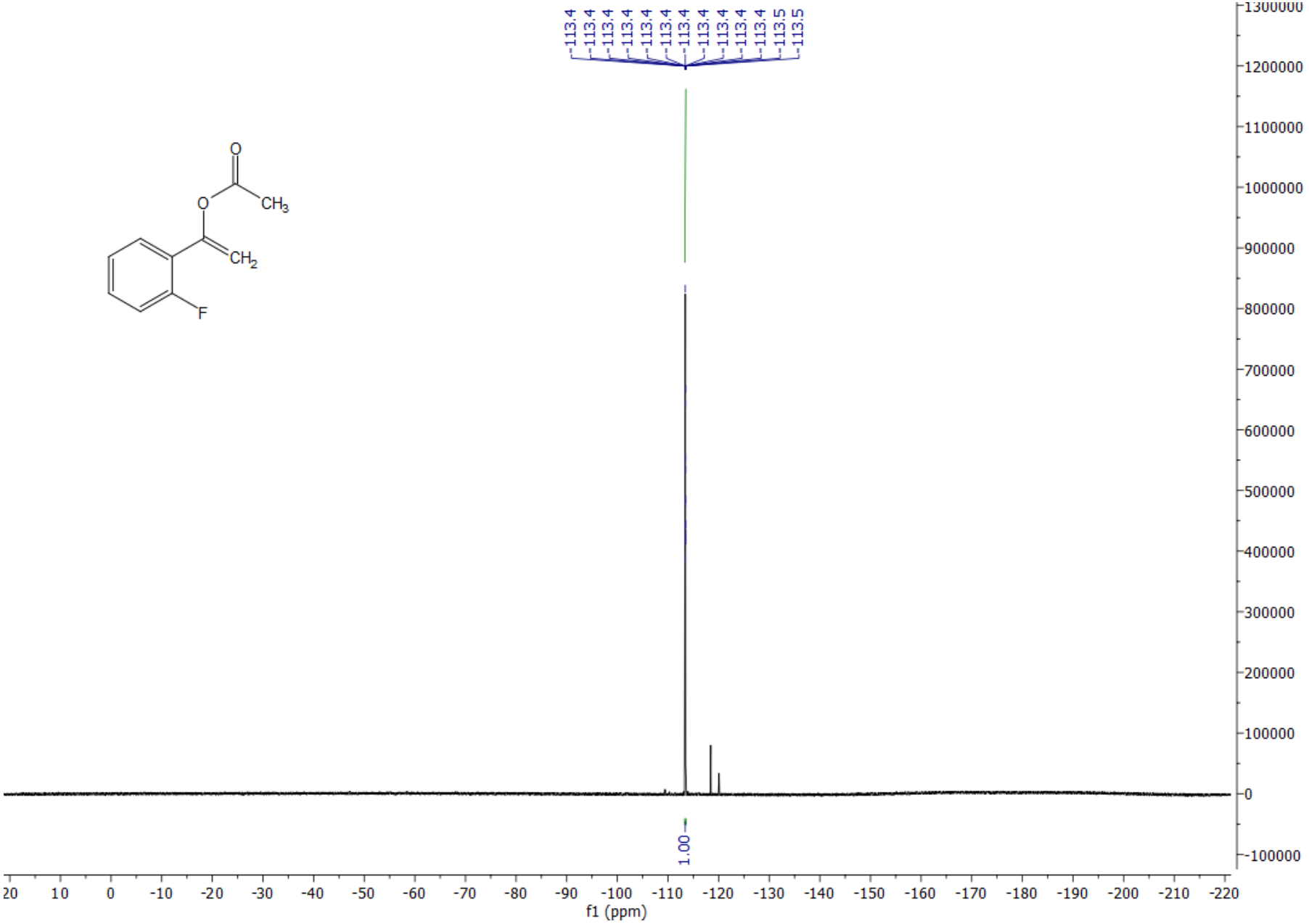
^{1}H NMR (300.13 MHz, $CDCl_3$), **1-(2-Fluorophenyl)vinyl acetate, 1h**



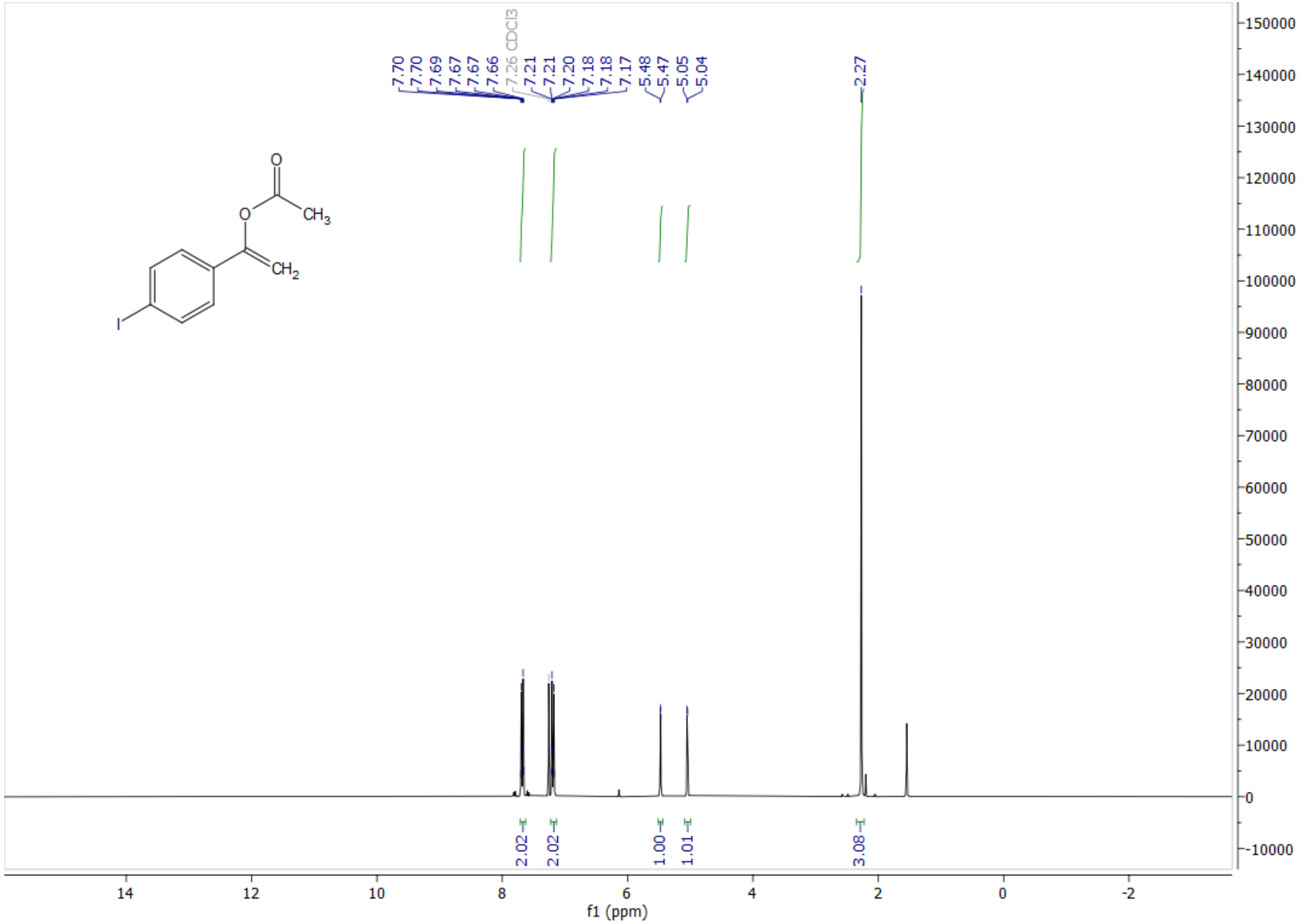
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **1-(2-Fluorophenyl)vinyl acetate, 1h**



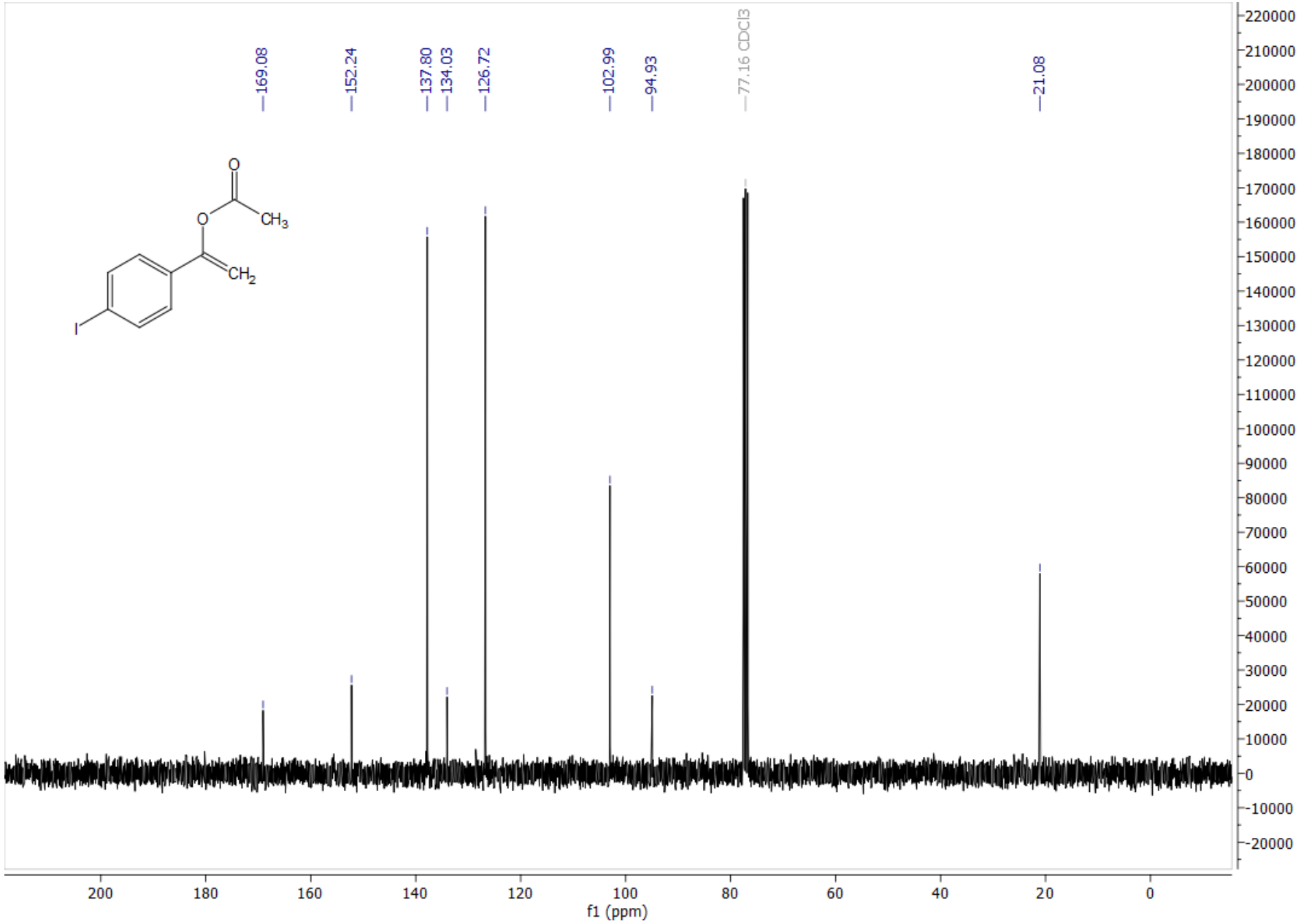
^{19}F NMR (282,5 MHz, $CDCl_3$), **1-(2-Fluorophenyl)vinyl acetate, 1h**



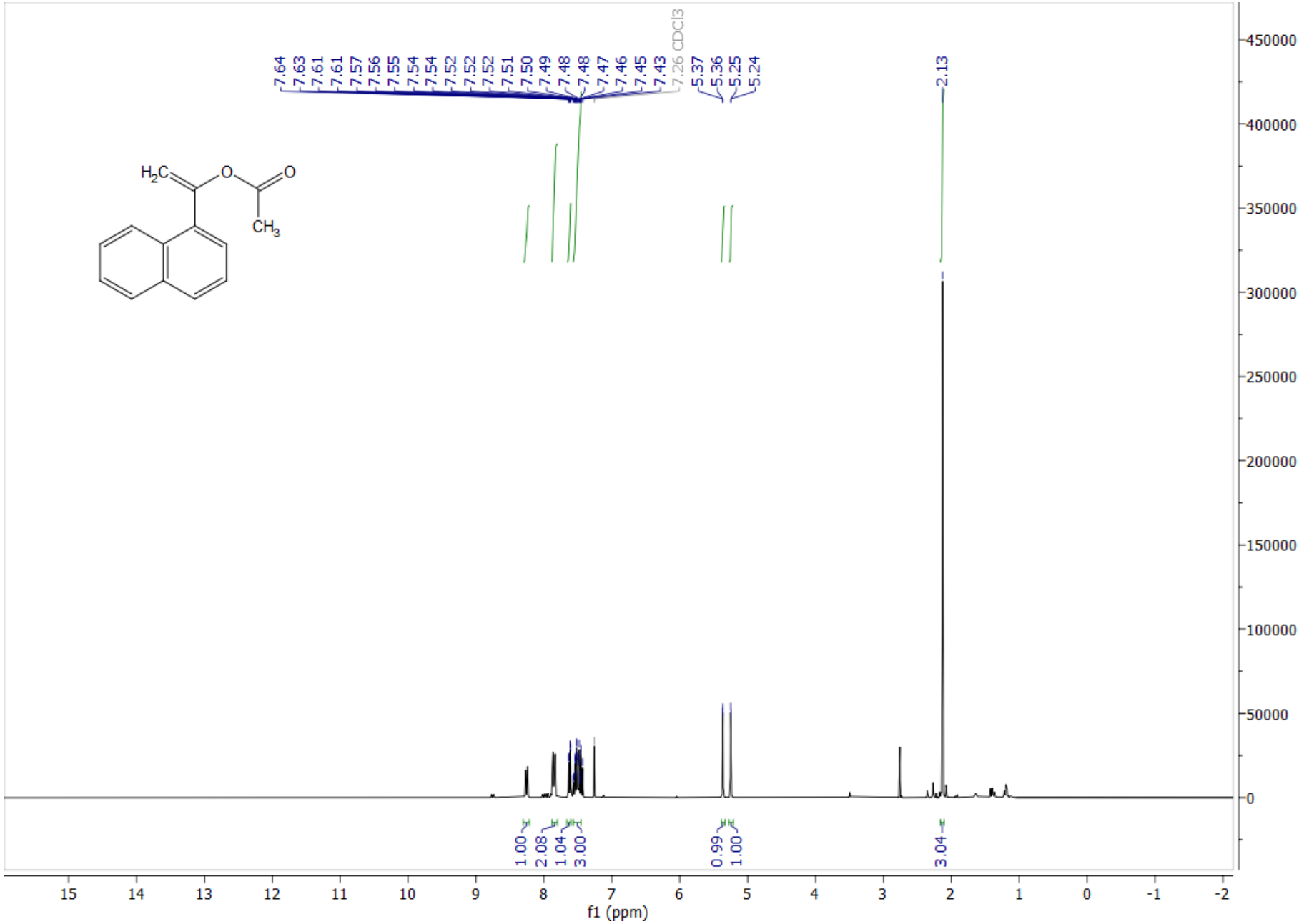
^{1}H NMR (300.13 MHz, $CDCl_3$), **1-(4-Iodophenyl)vinyl acetate, 1t**



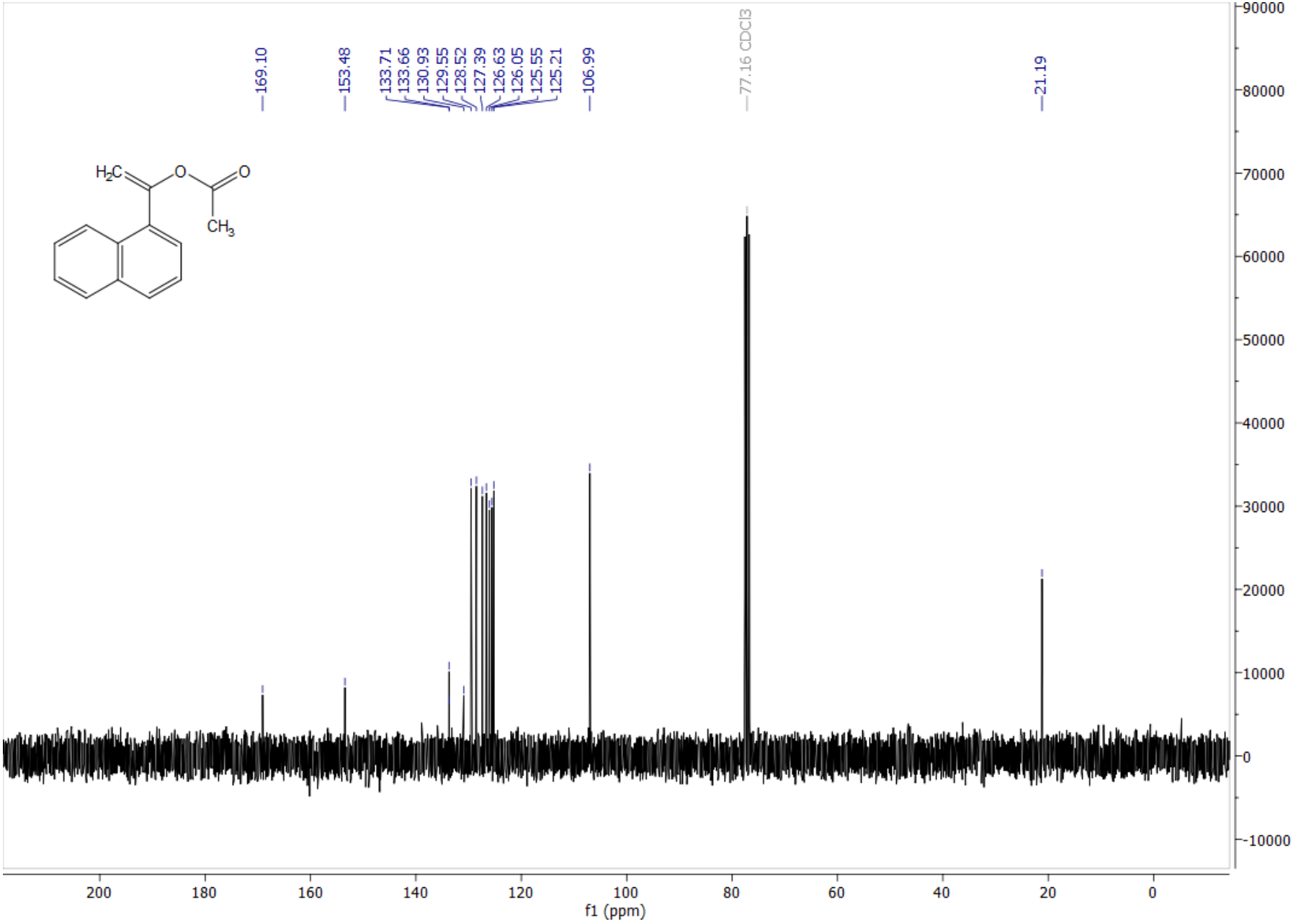
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **1-(4-Iodophenyl)vinyl acetate, 1t**



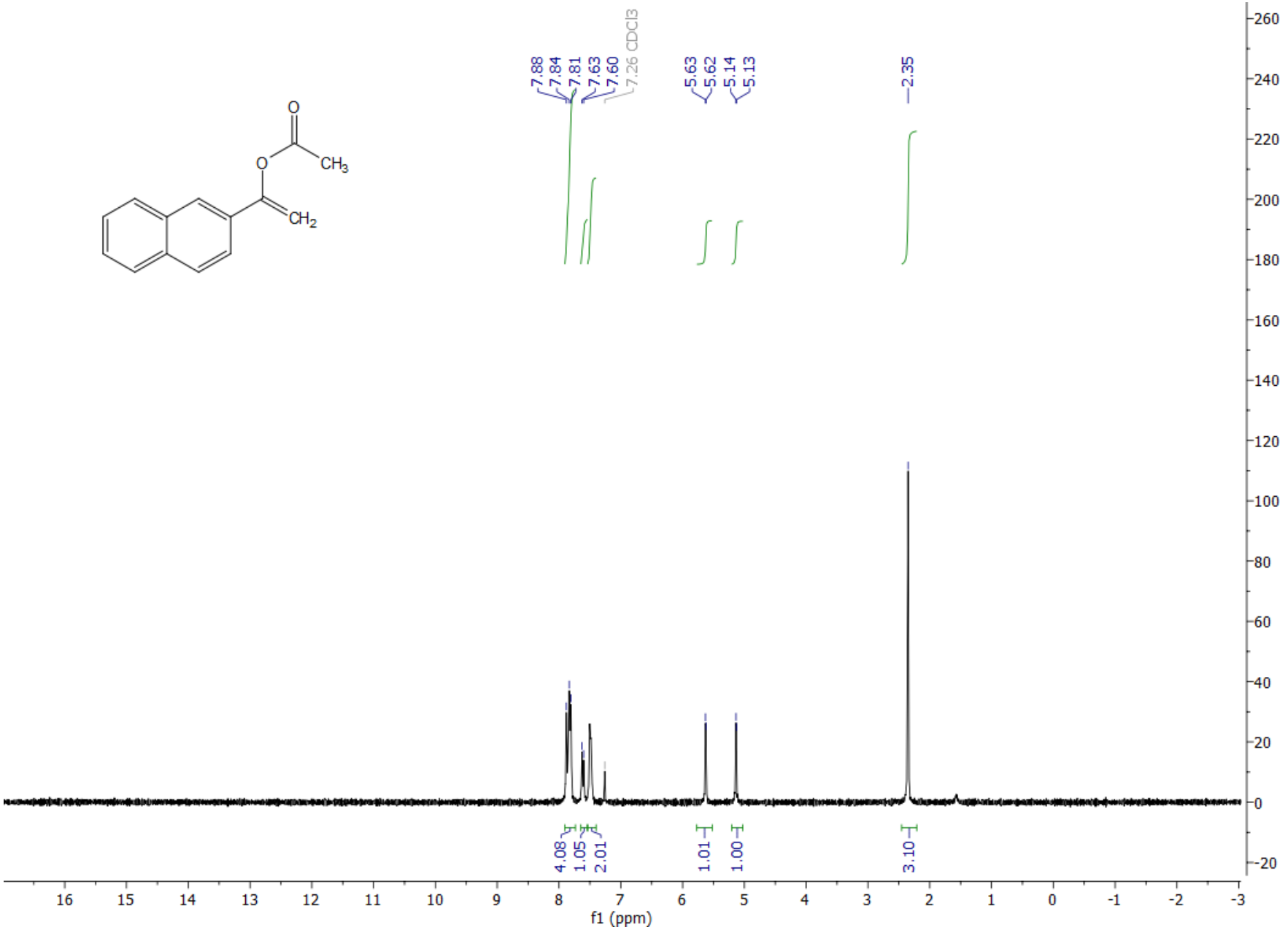
^{1}H NMR (300.13 MHz, $CDCl_3$), **1-(Naphthalen-1-yl)vinyl acetate, 1i**



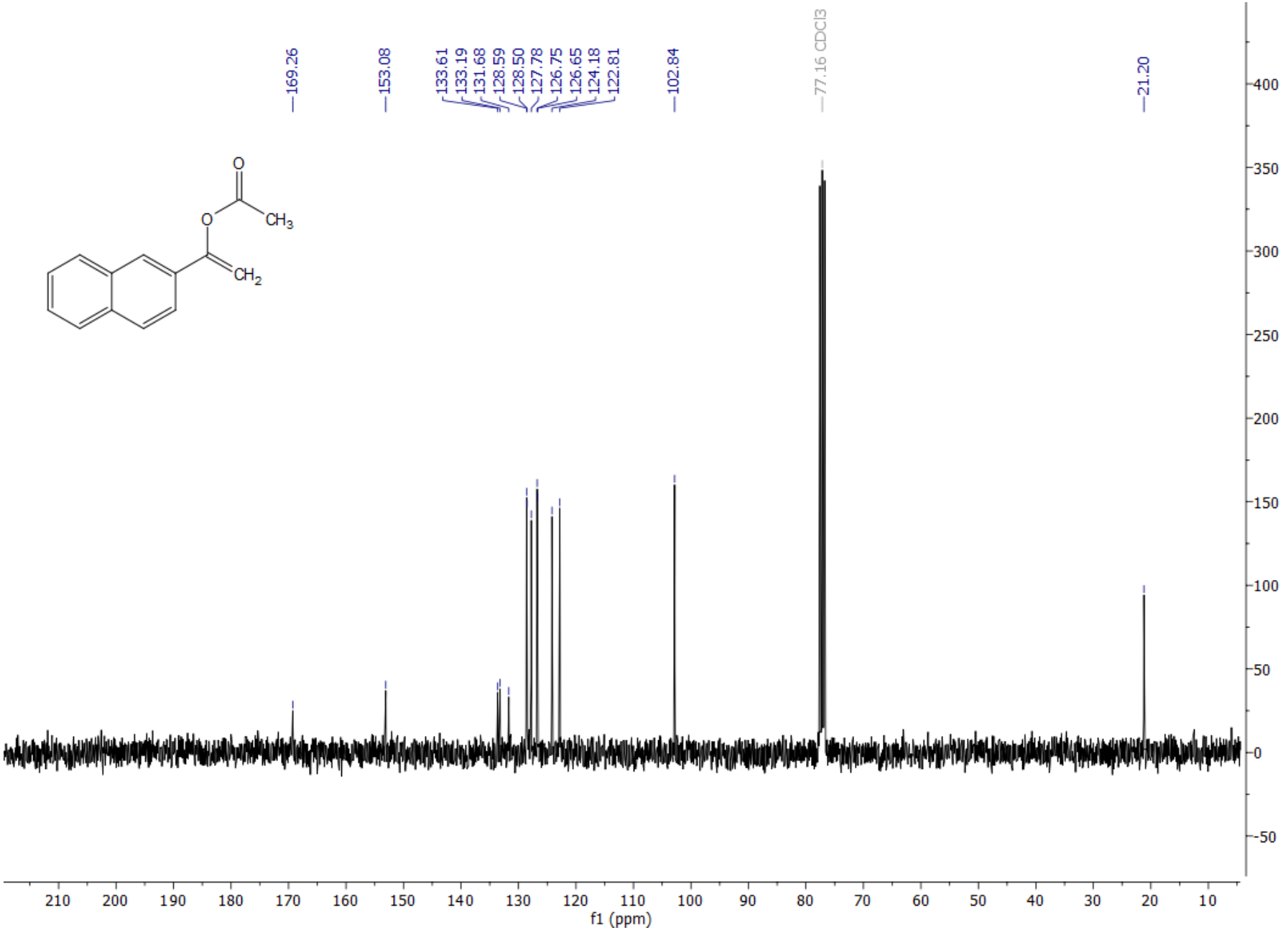
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **1-(Naphthalen-1-yl)vinyl acetate, 1i**



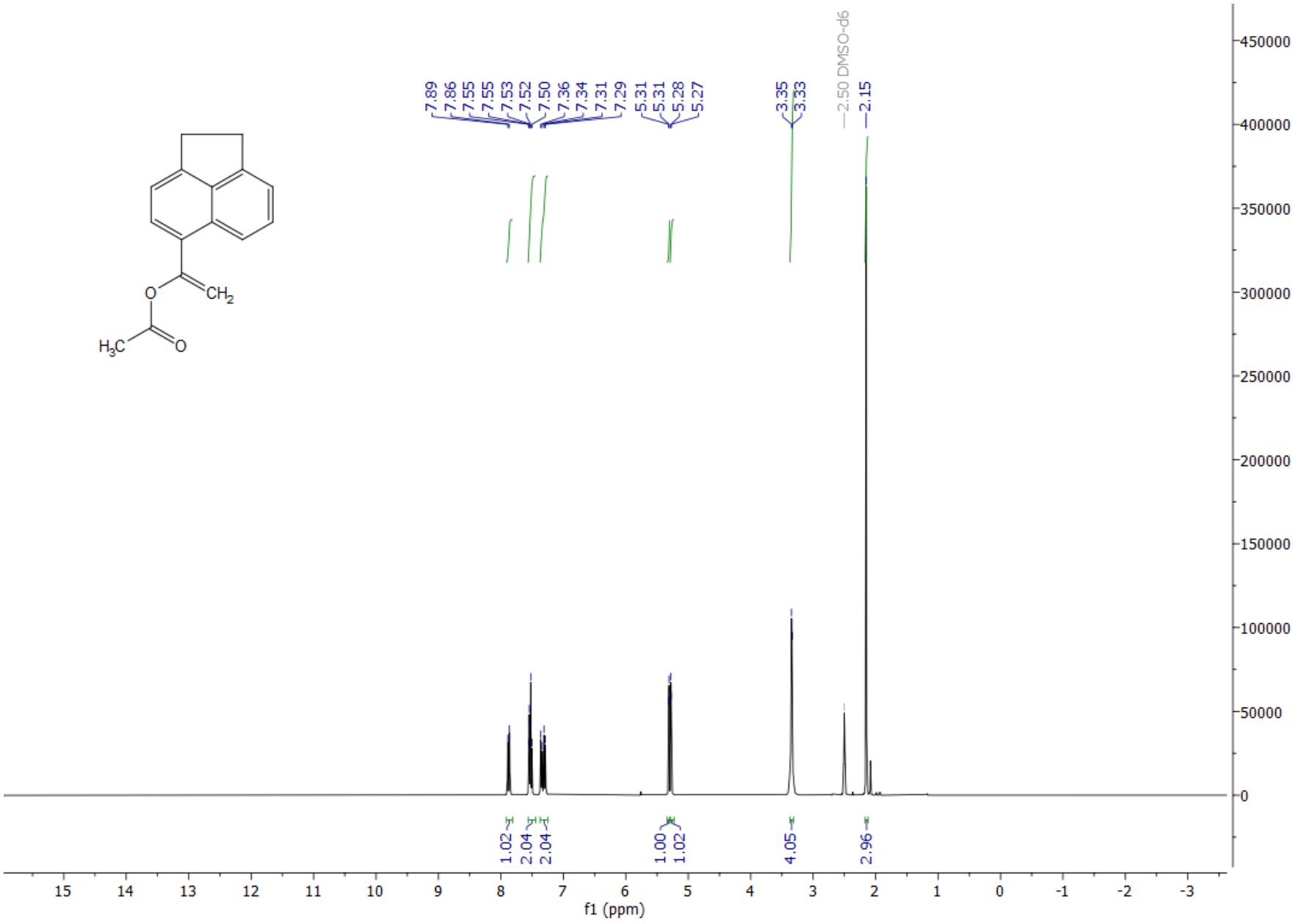
^{1}H NMR (300.13 MHz, $CDCl_3$), **1-(Naphthalen-2-yl)vinyl acetate, 1j**



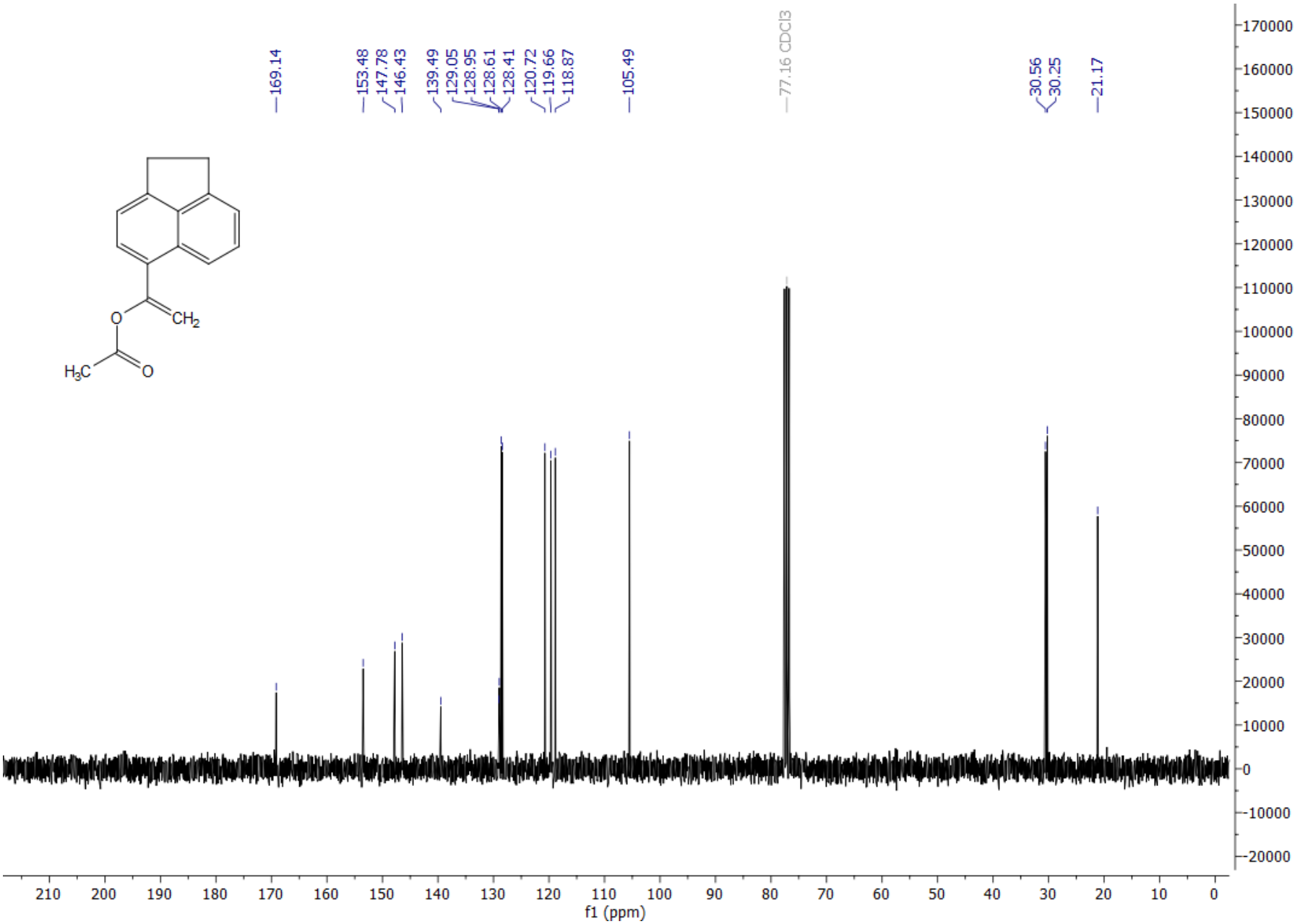
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **1-(Naphthalen-2-yl)vinyl acetate, 1j**



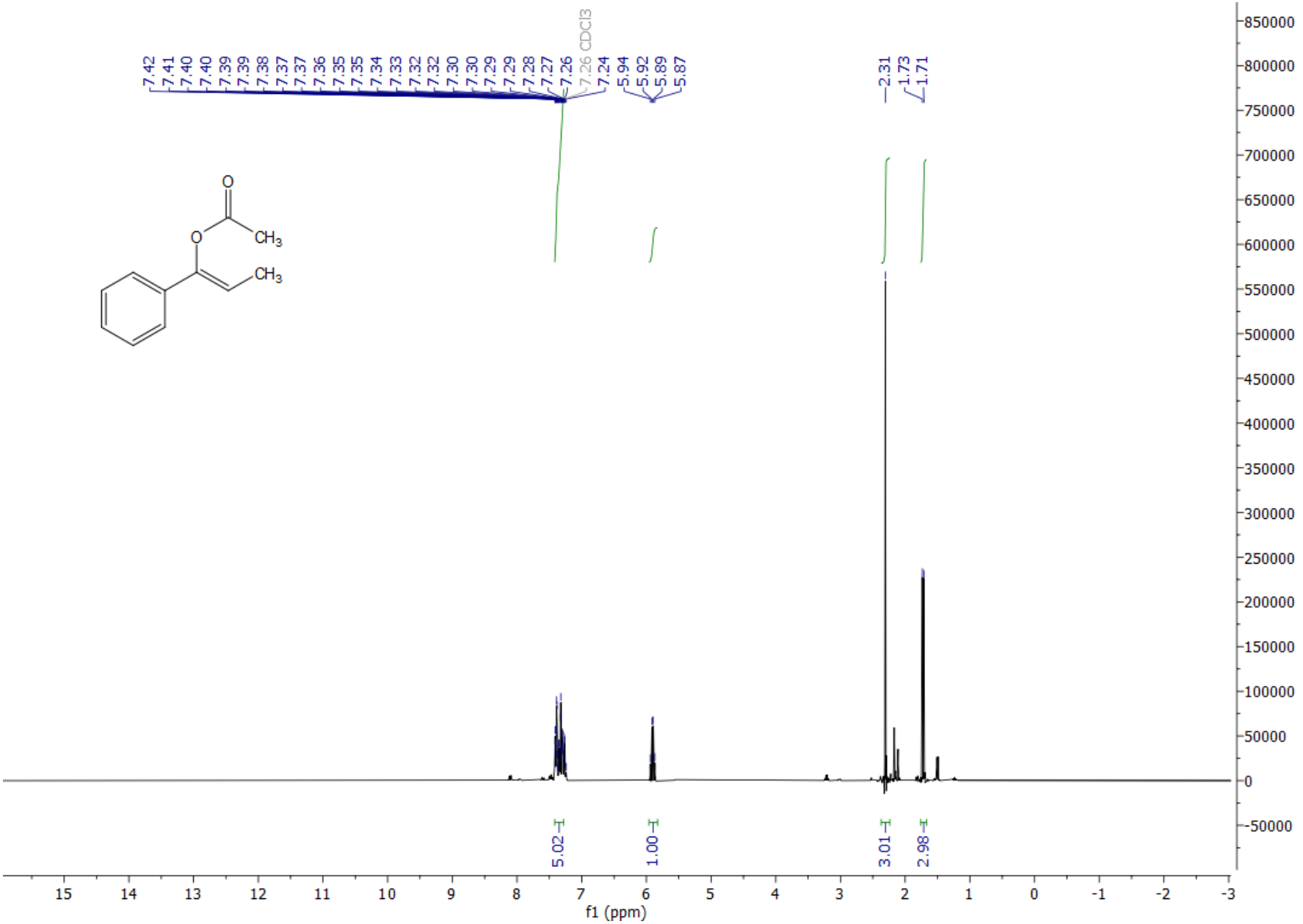
^{1}H NMR (300.13 MHz, DMSO-d6), **1-(1,2-Dihydroacenaphthylen-5-yl)vinyl acetate, 1k**



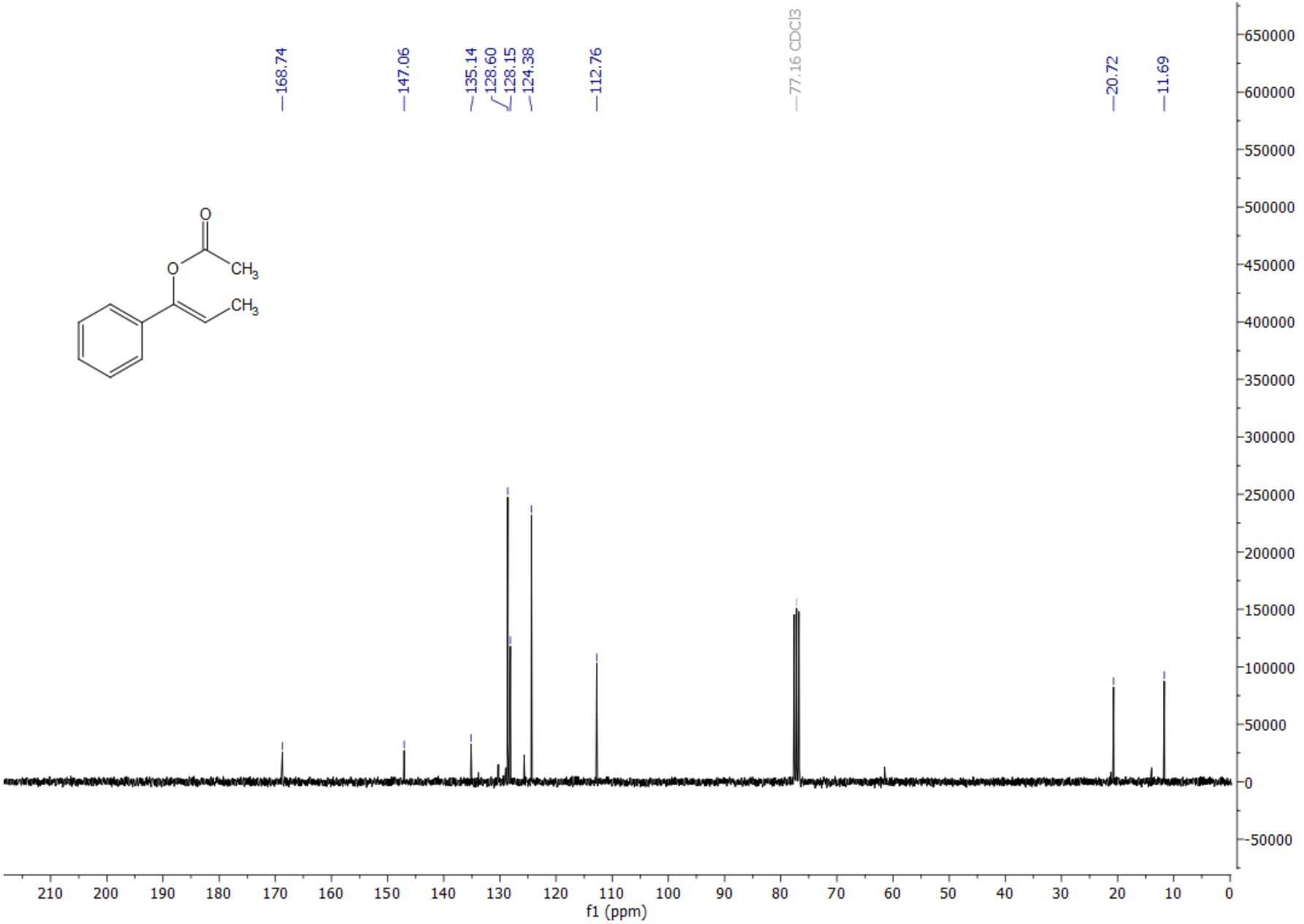
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **1-(1,2-Dihydroacenaphthylen-5-yl)vinyl acetate, 1k**



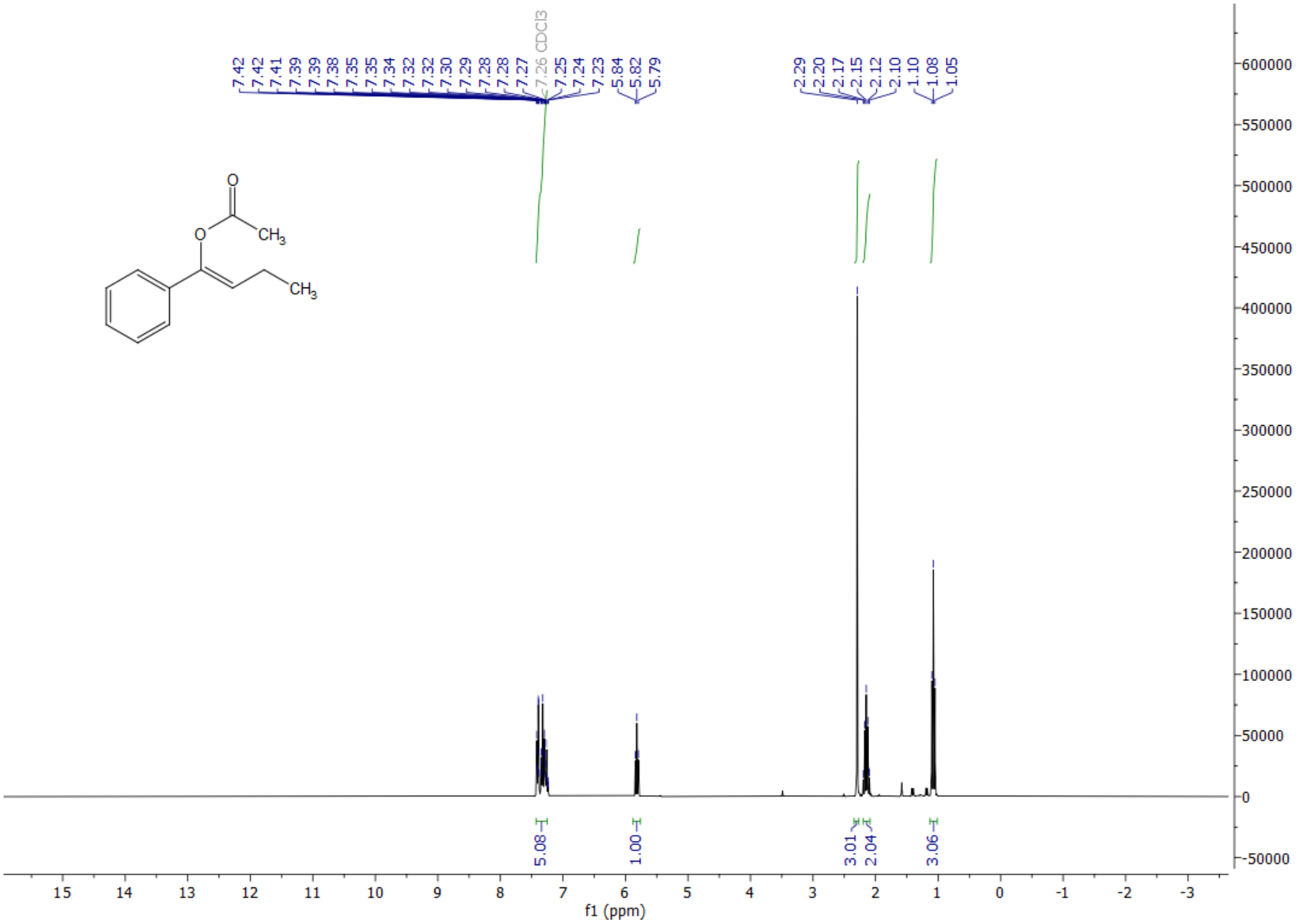
^{1}H NMR (300.13 MHz, $CDCl_3$), **(*Z*)-1-Phenylprop-1-en-1-yl acetate, 1l**



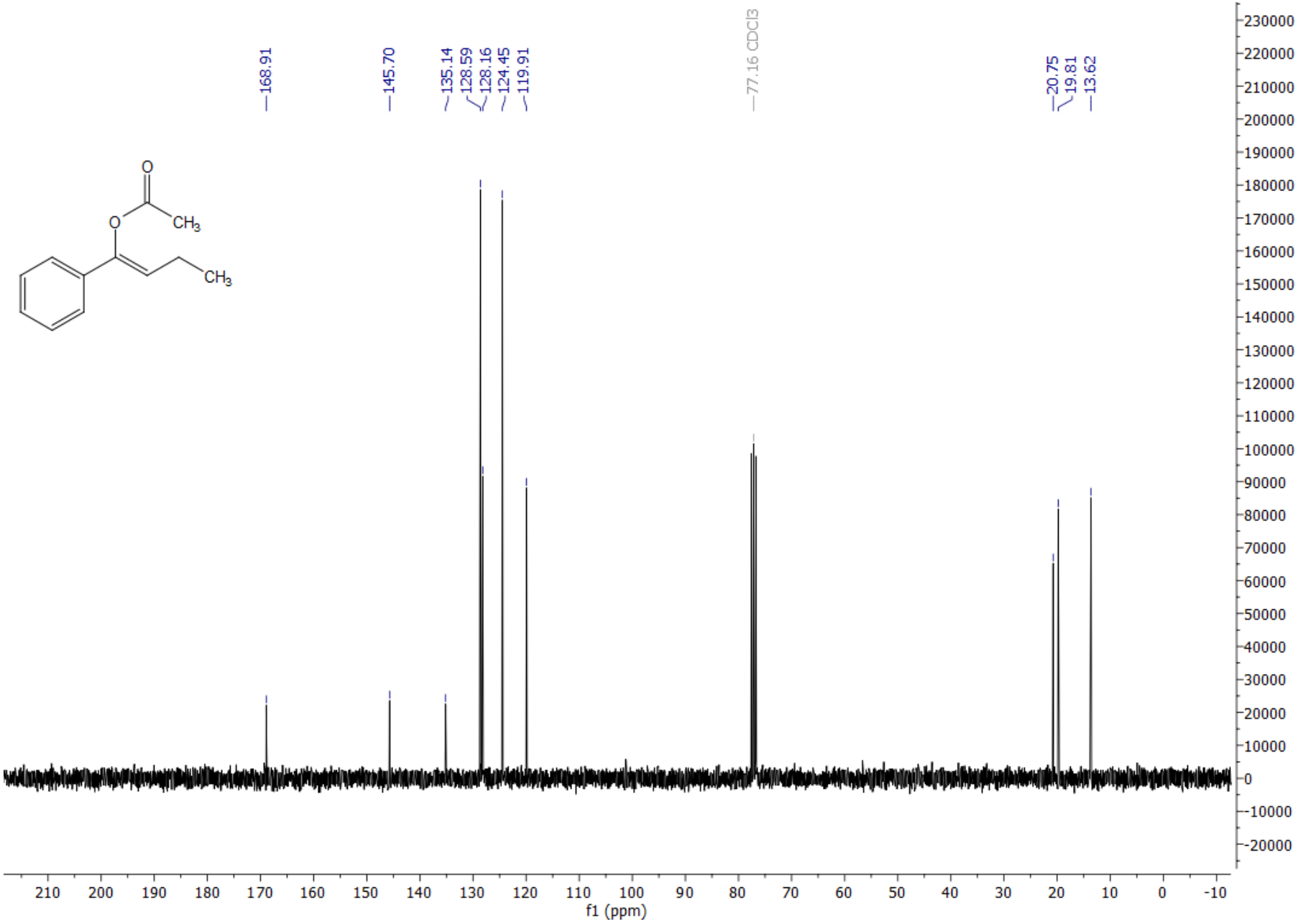
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **(*Z*)-1-Phenylprop-1-en-1-yl acetate, 1l**



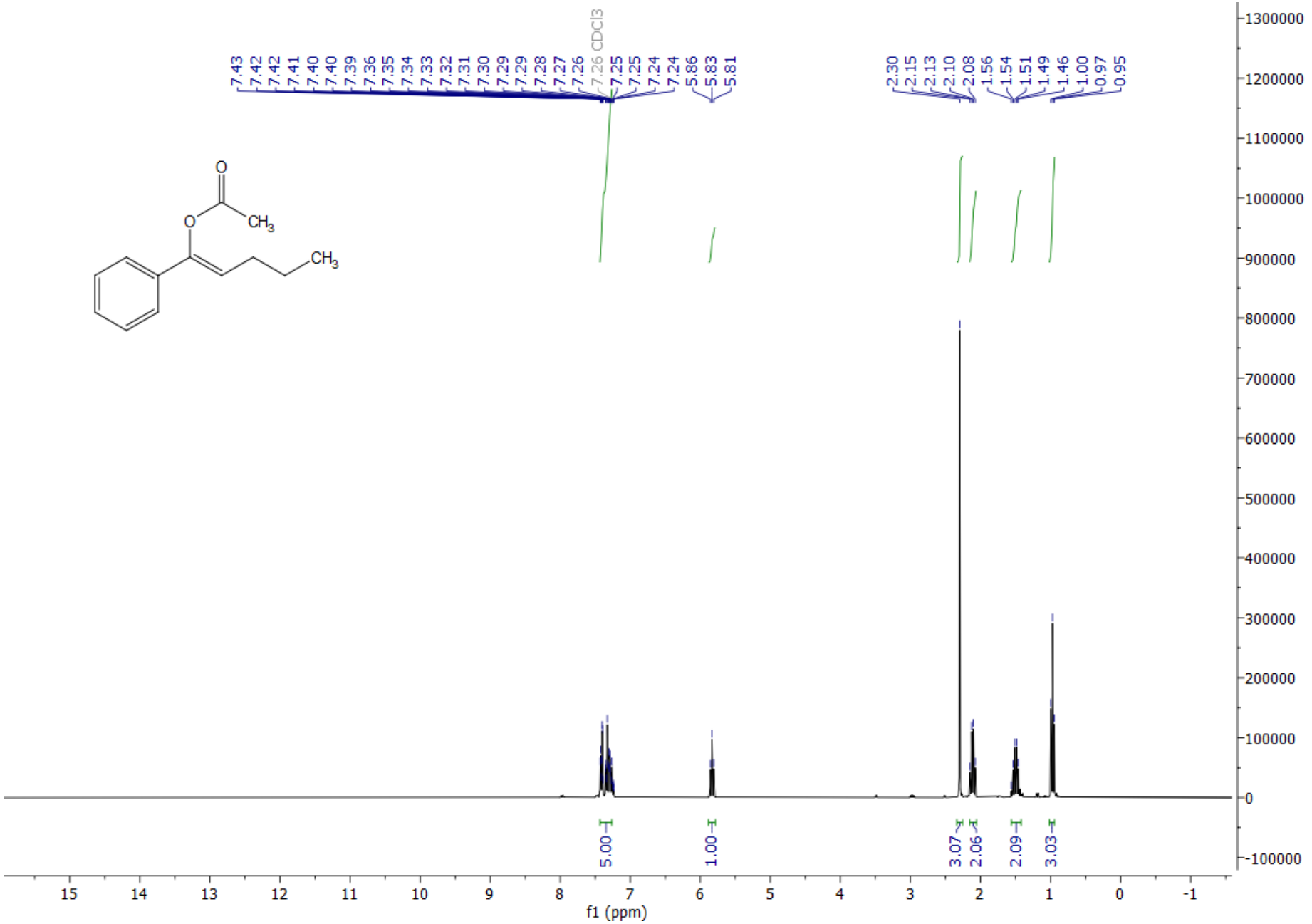
^{1}H NMR (300.13 MHz, $CDCl_3$), **(*Z*)-1-Phenylbut-1-en-1-yl acetate, 1m**



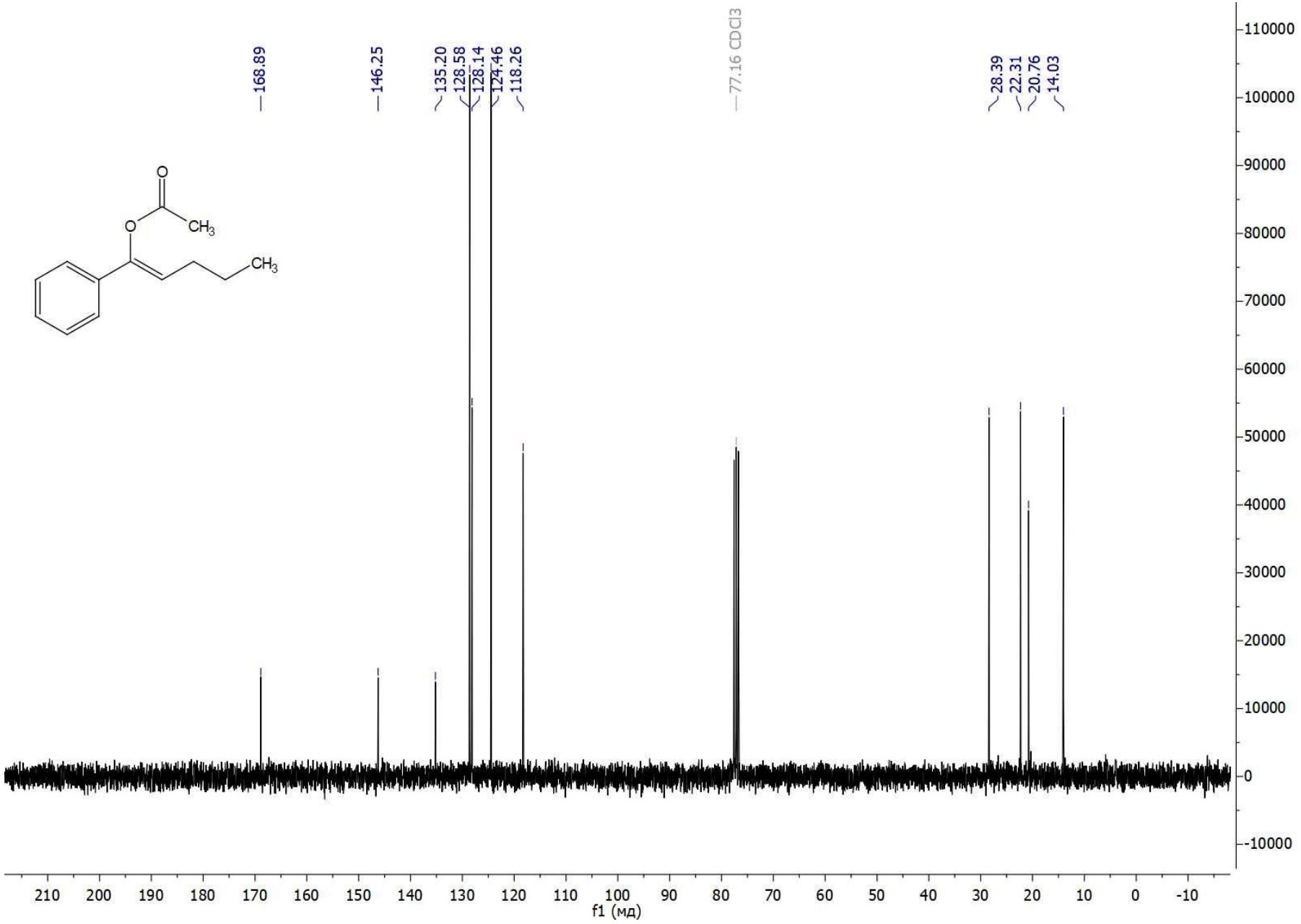
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **(*Z*)-1-Phenylbut-1-en-1-yl acetate, 1m**



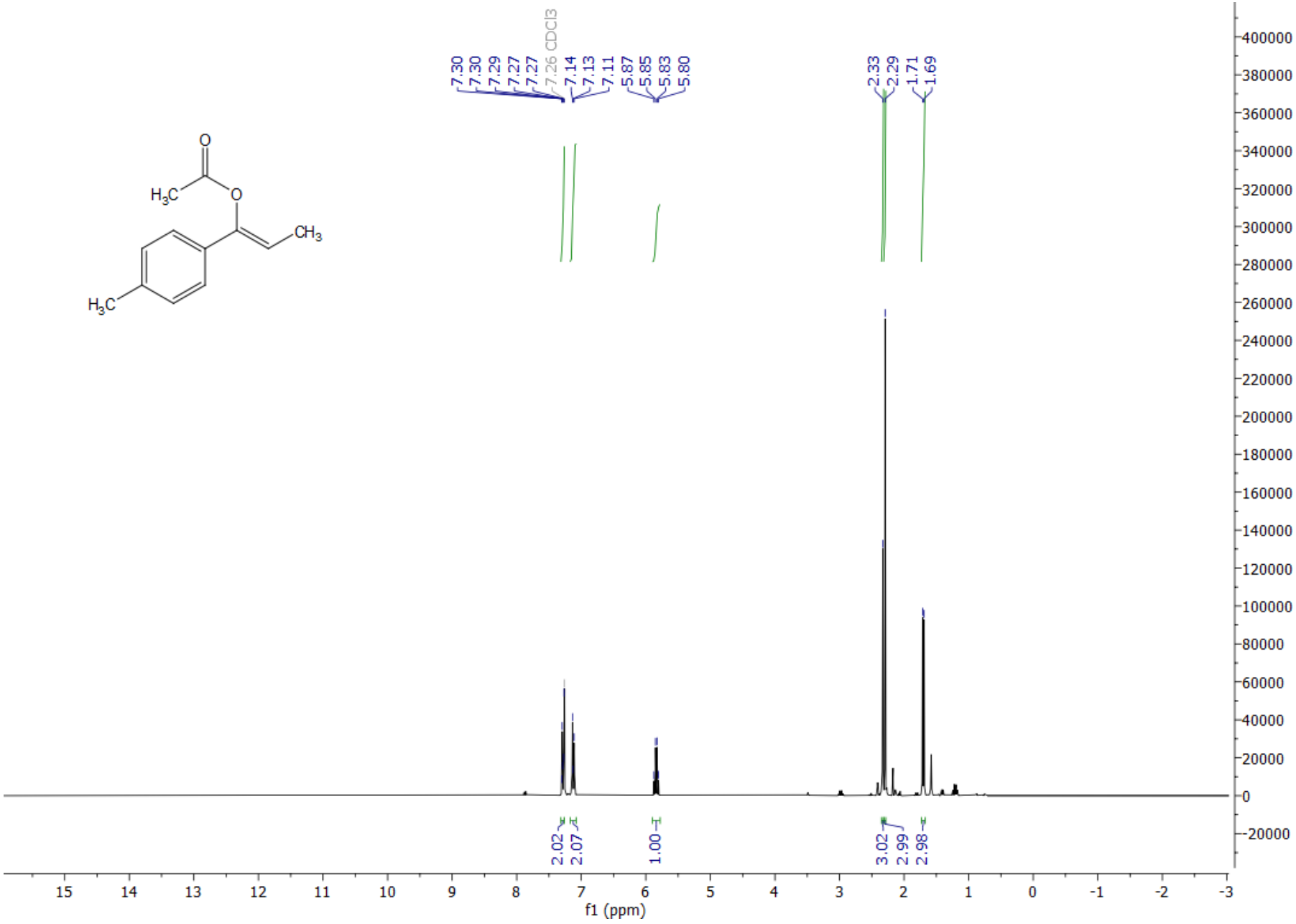
^{1}H NMR (300.13 MHz, $CDCl_3$), **(*Z*)-1-Phenylpent-1-en-1-yl acetate, 1n**



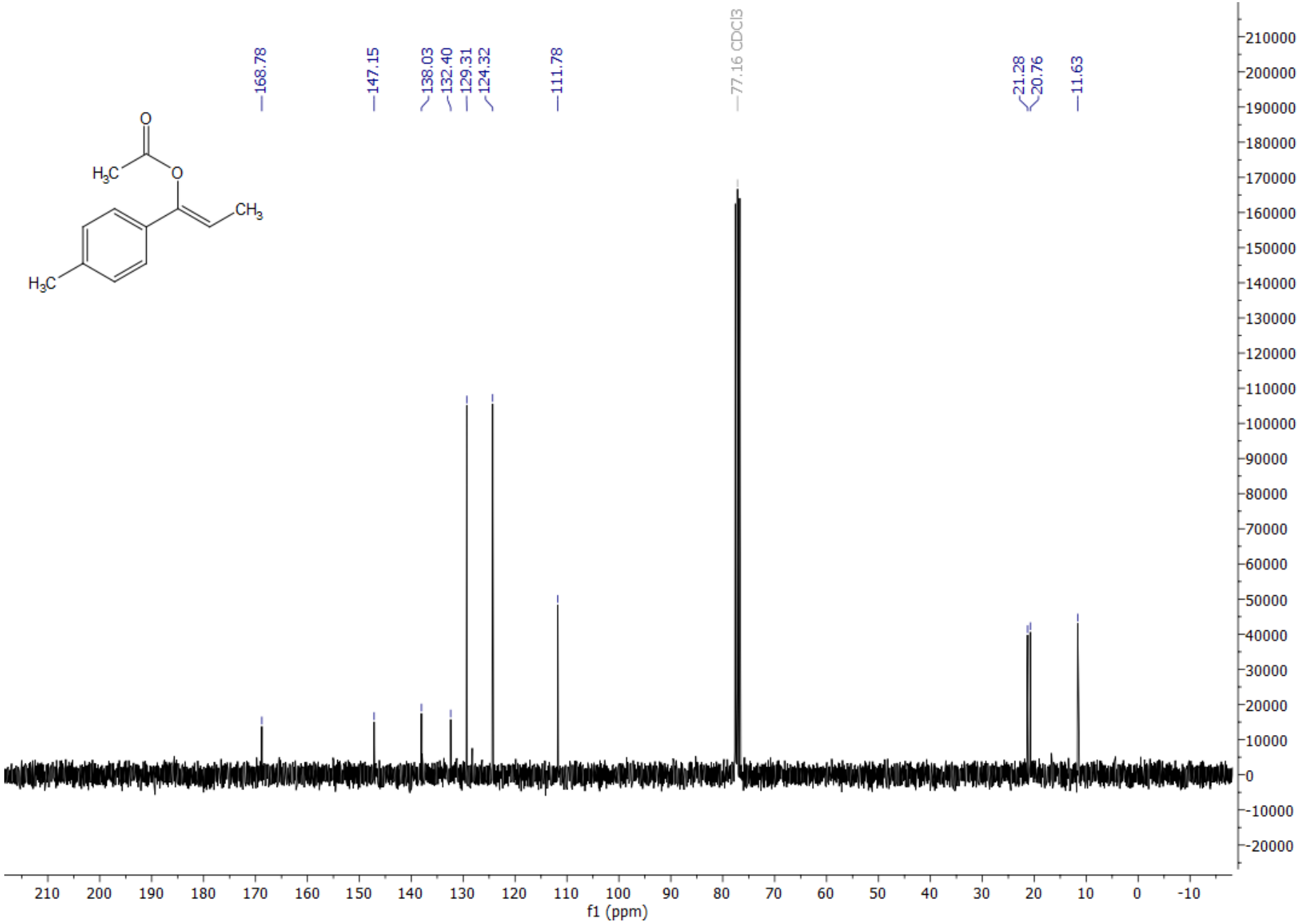
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **(*Z*)-1-Phenylpent-1-en-1-yl acetate, 1n**



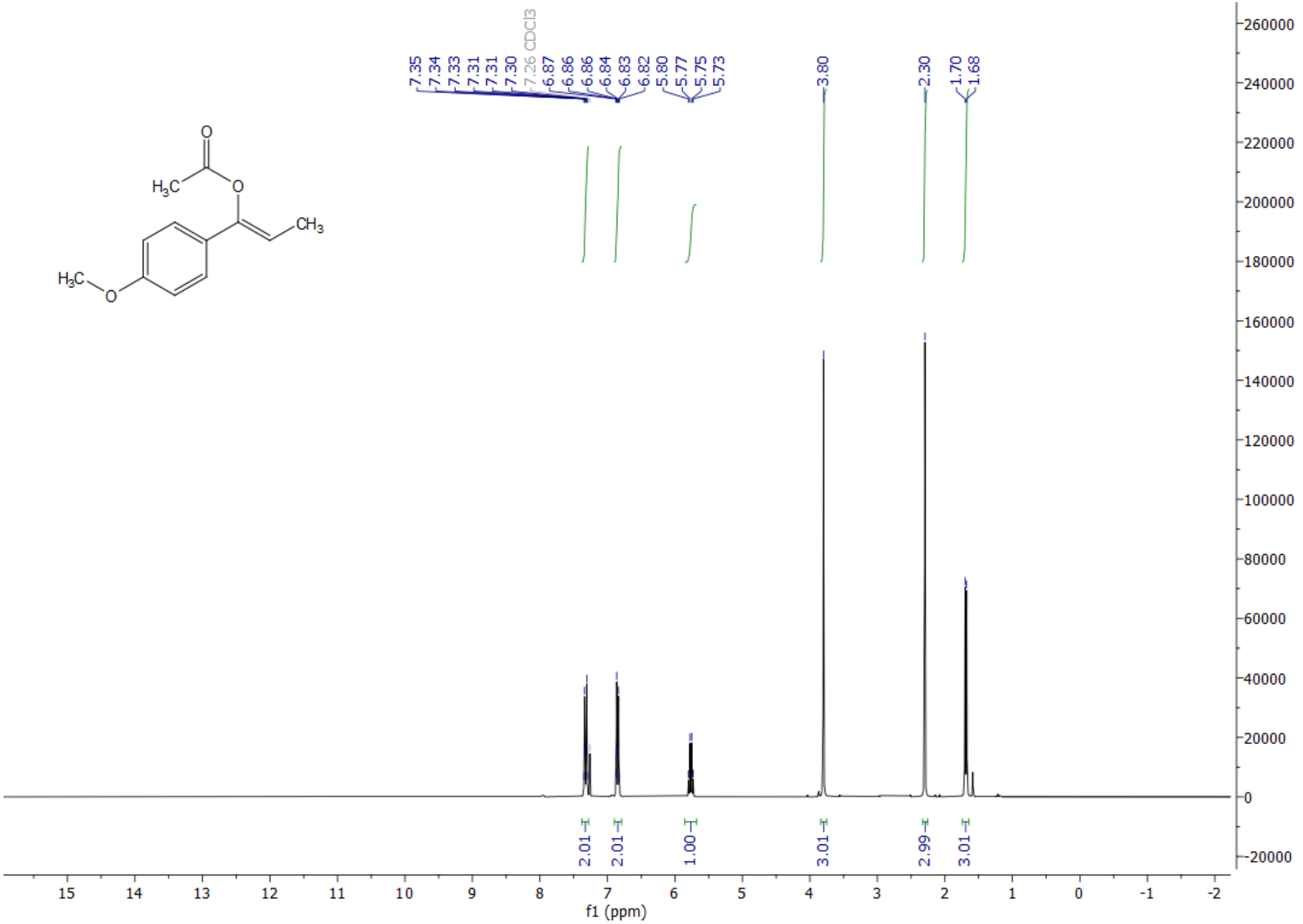
^{1}H NMR (300.13 MHz, $CDCl_3$), **(*Z*)-1-(*p*-Tolyl)prop-1-en-1-yl acetate, 1o**



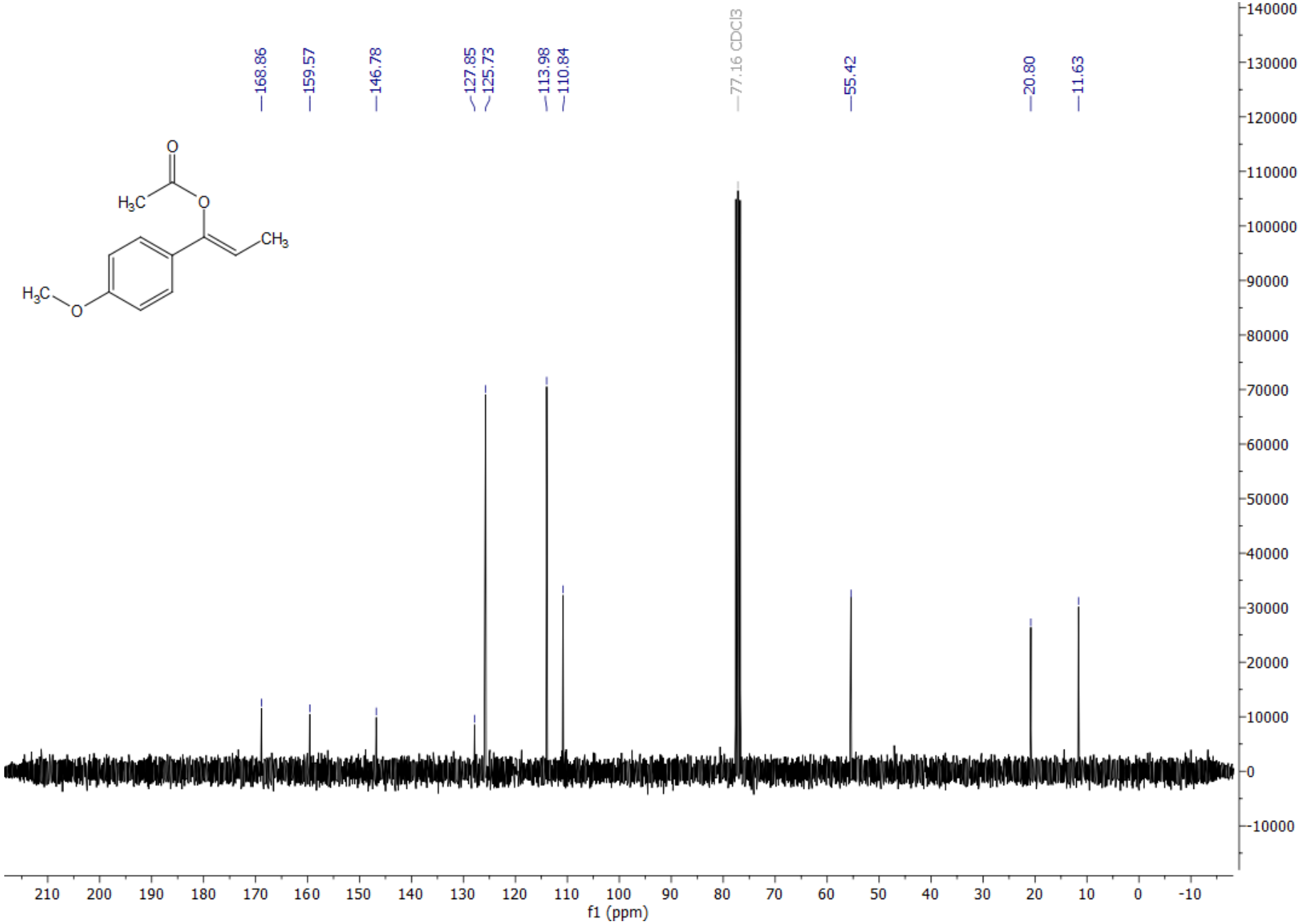
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **(*Z*)-1-(*p*-Tolyl)prop-1-en-1-yl acetate, 1o**



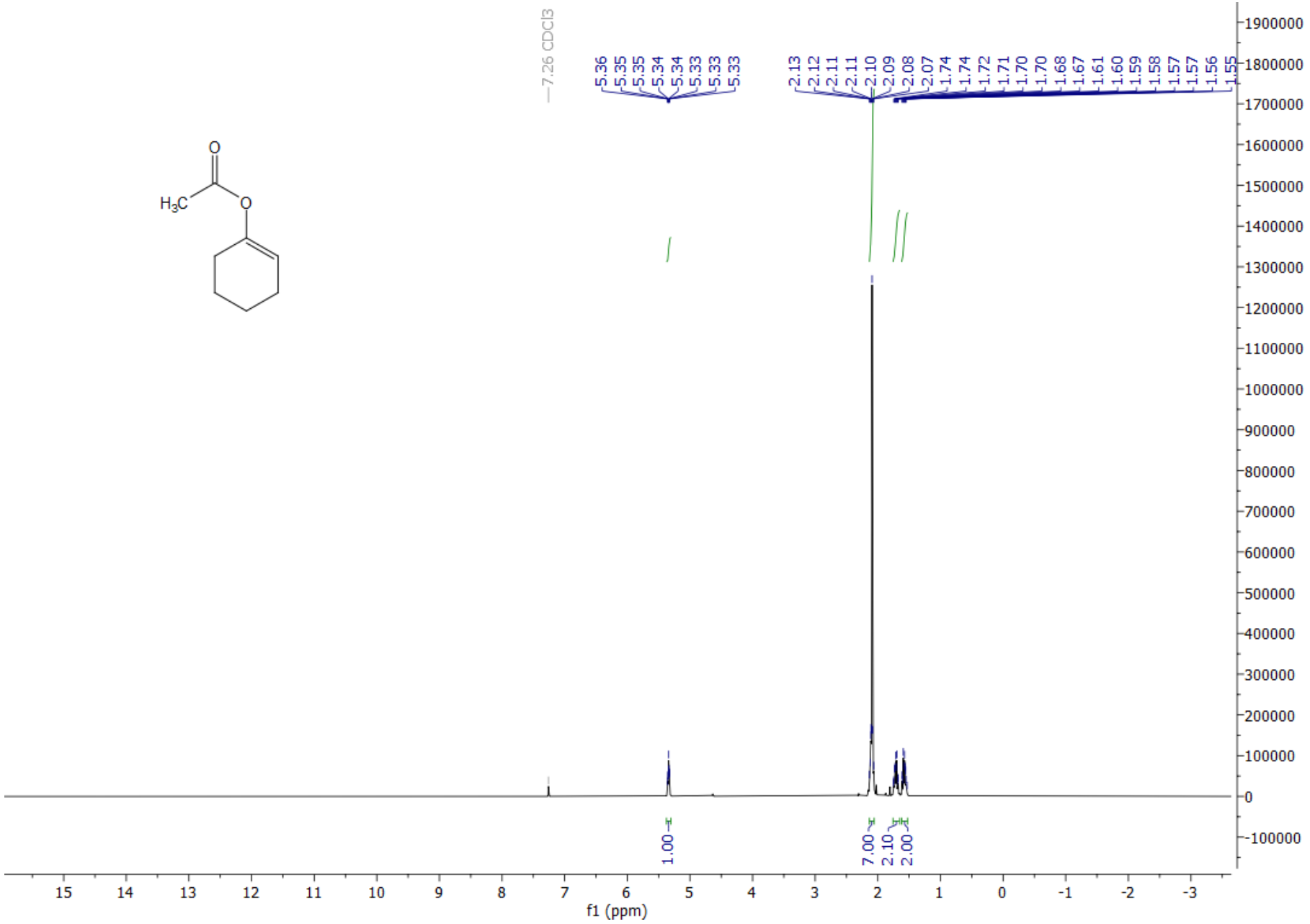
^{1}H NMR (300.13 MHz, $CDCl_3$), **(*Z*)-1-(4-Methoxyphenyl)prop-1-en-1-yl acetate, 1p**



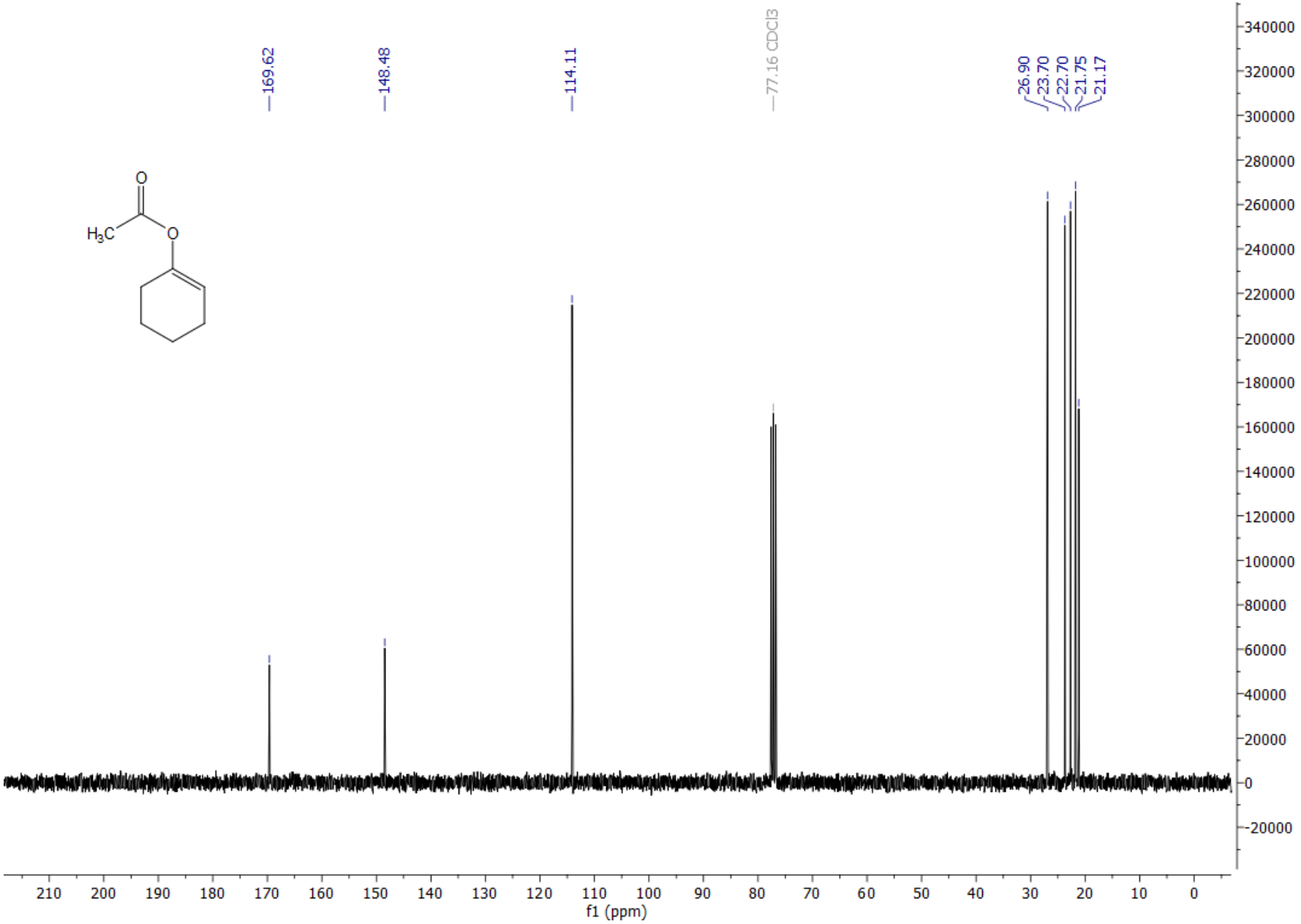
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **(*Z*)-1-(4-Methoxyphenyl)prop-1-en-1-yl acetate, 1p**



^{1}H NMR (300.13 MHz, $CDCl_3$), **Cyclohex-1-en-1-yl acetate**

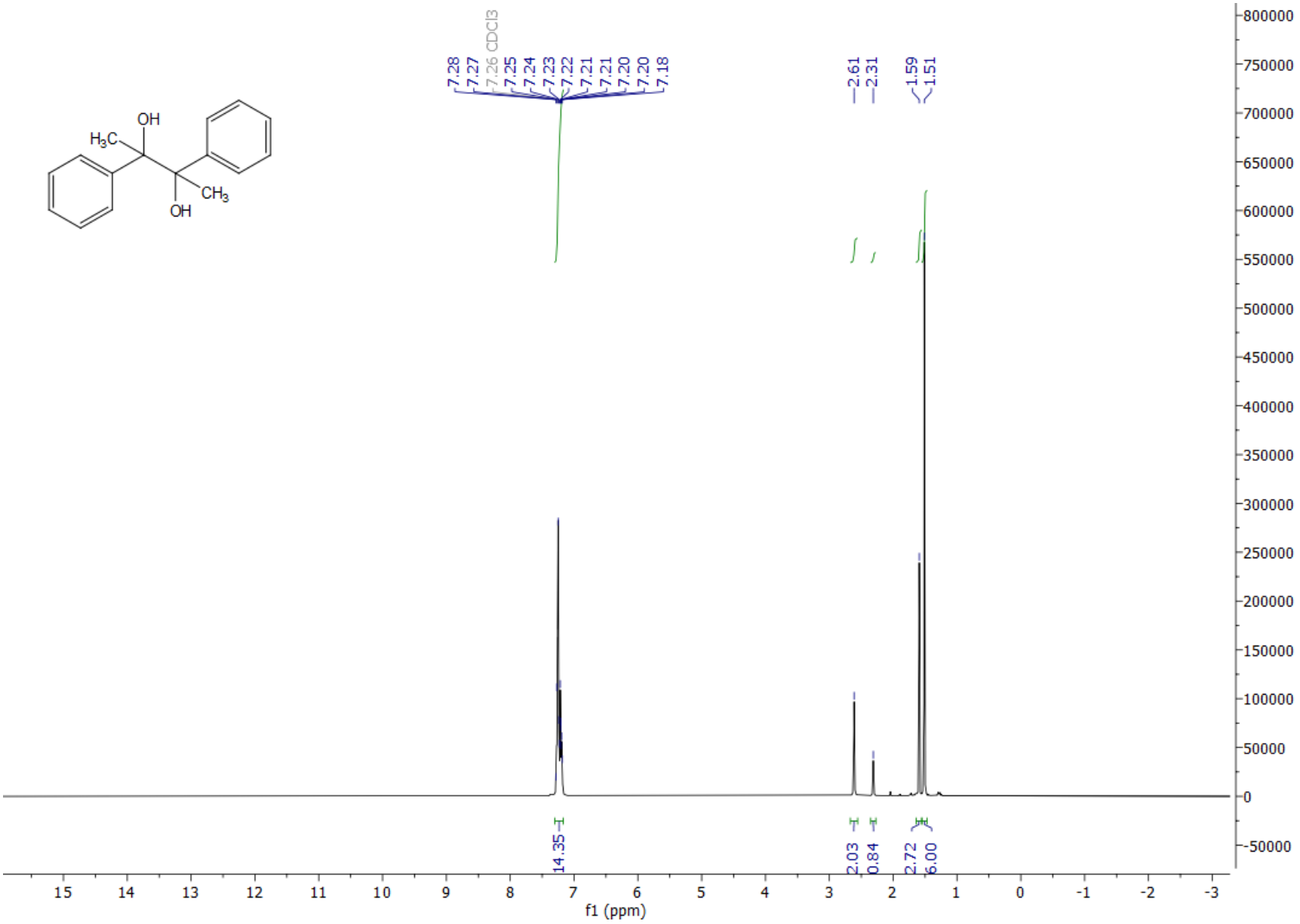


$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **Cyclohex-1-en-1-yl acetate**

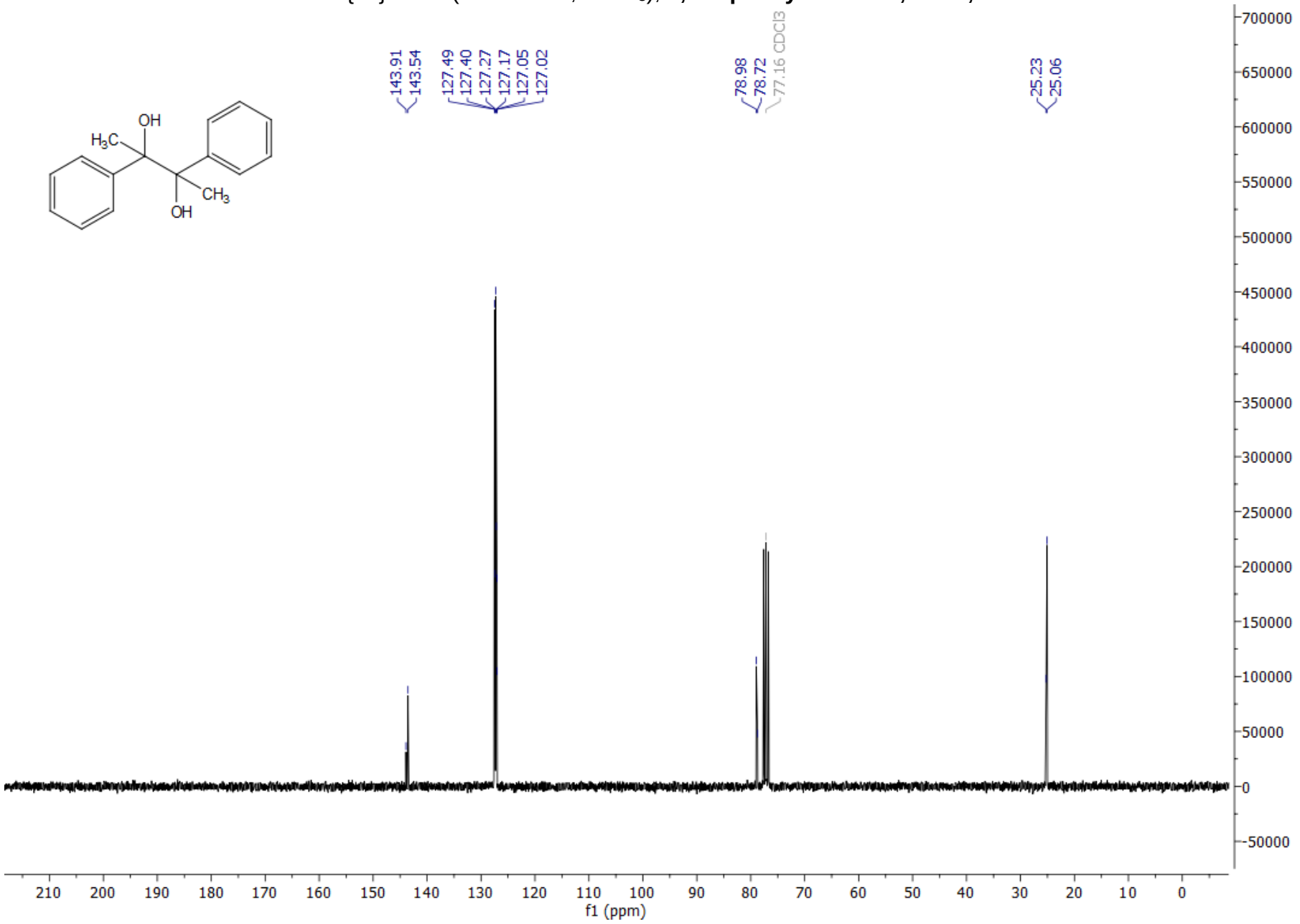


NMR spectra of the obtained products

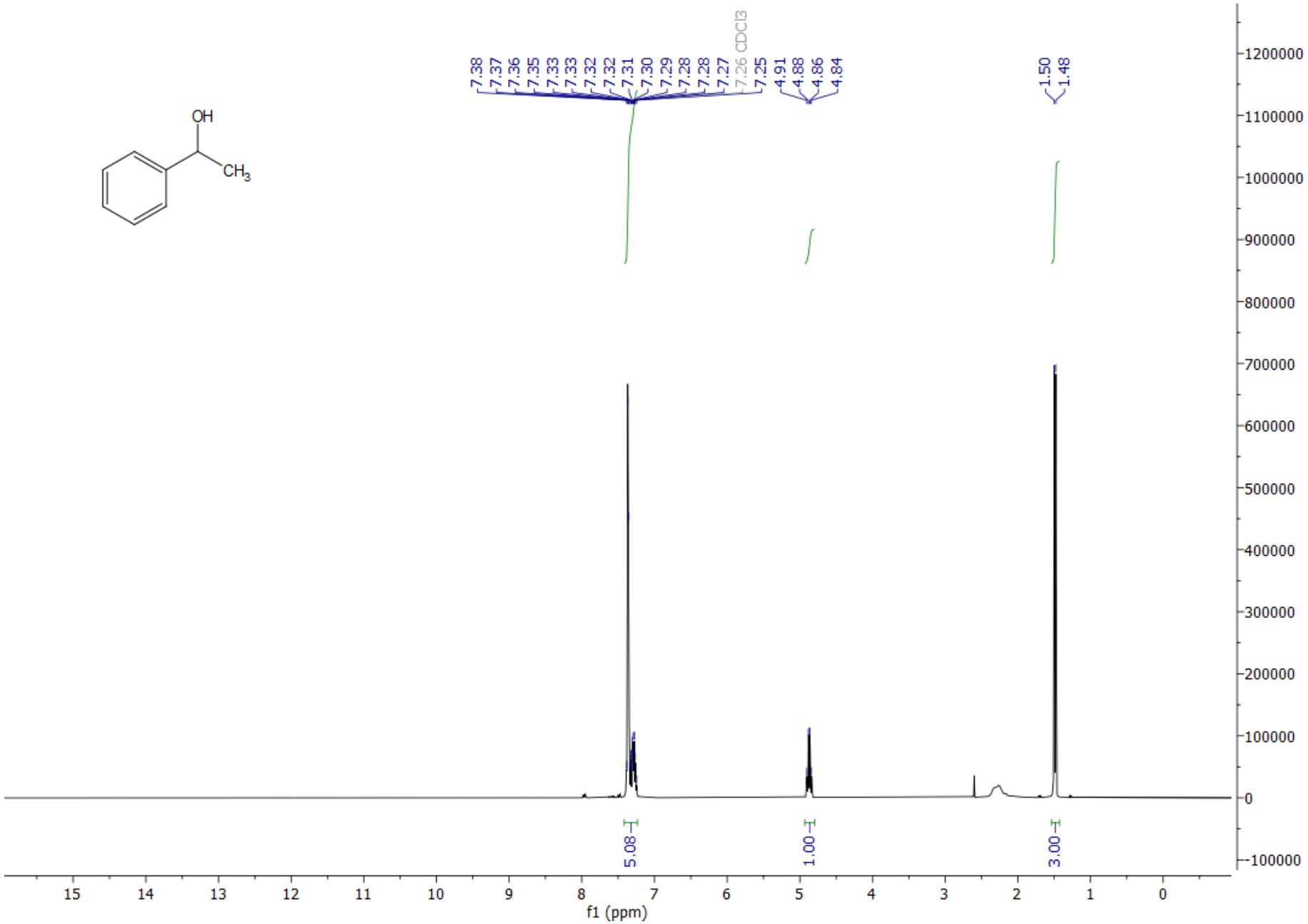
^{1}H NMR (300.13 MHz, $CDCl_3$), **2,3-Diphenylbutane-2,3-diol, P2**



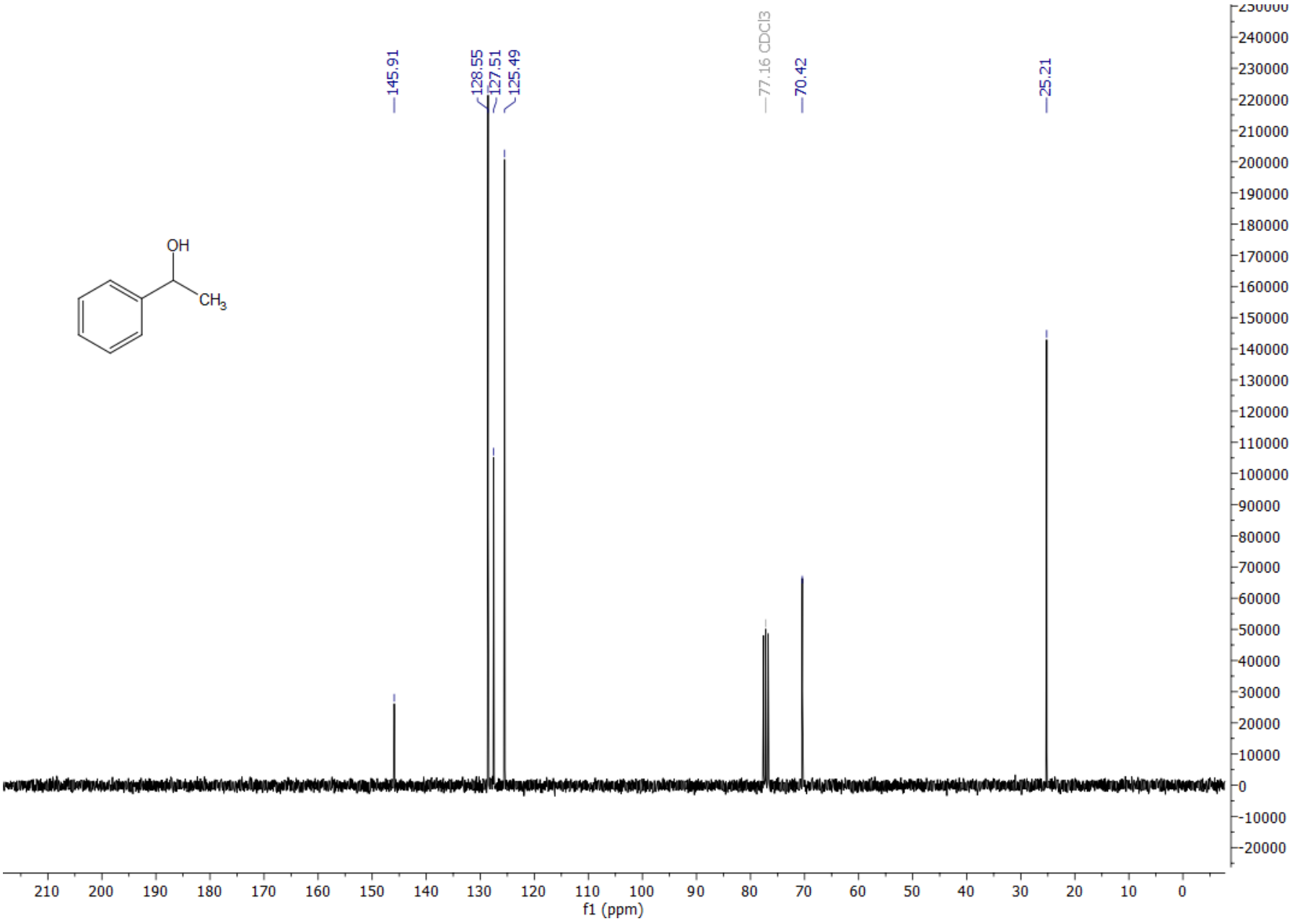
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **2,3-Diphenylbutane-2,3-diol, P2**



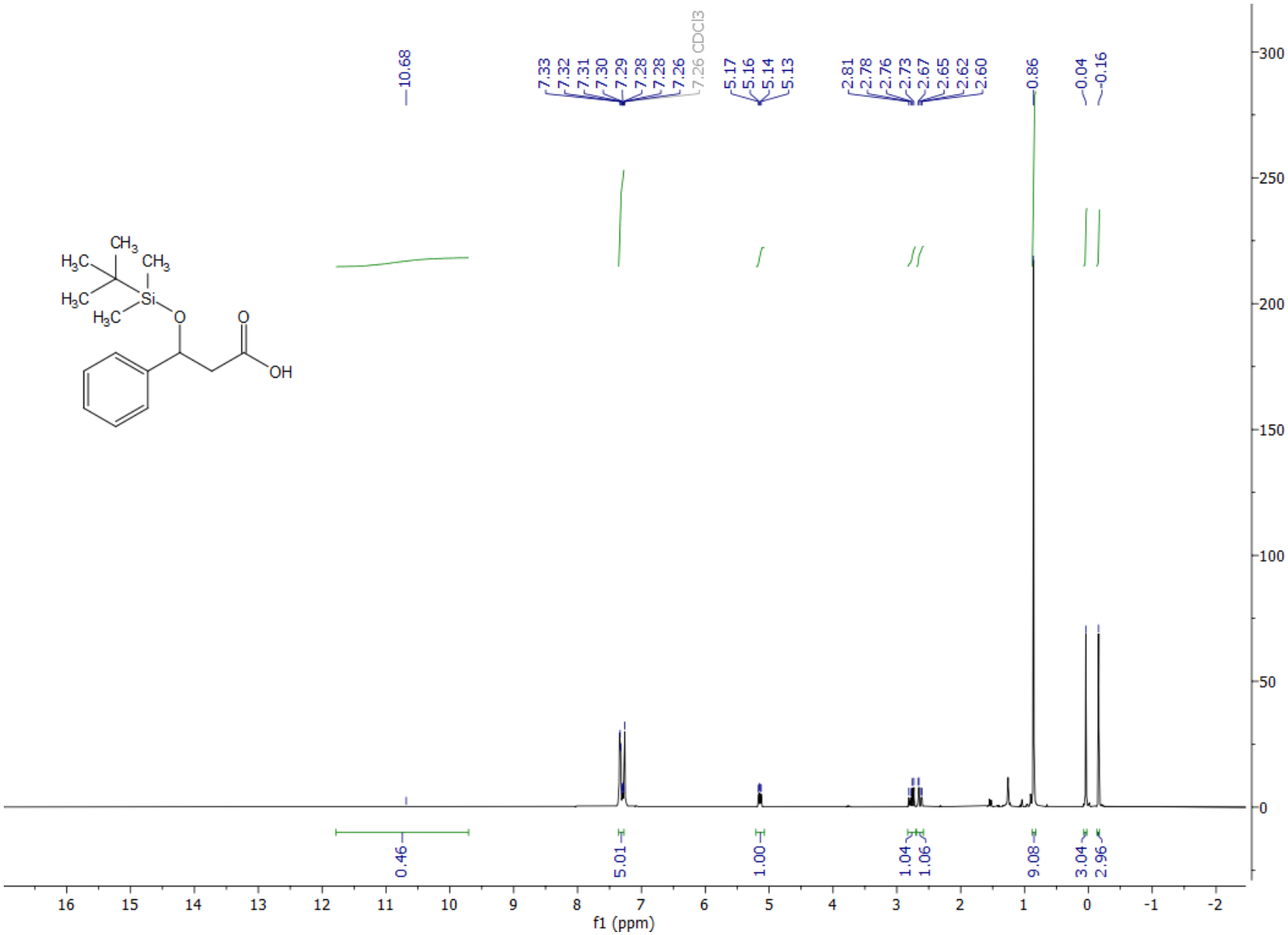
^{1}H NMR (300.13 MHz, $CDCl_3$), **1-Phenylethanol, P3**



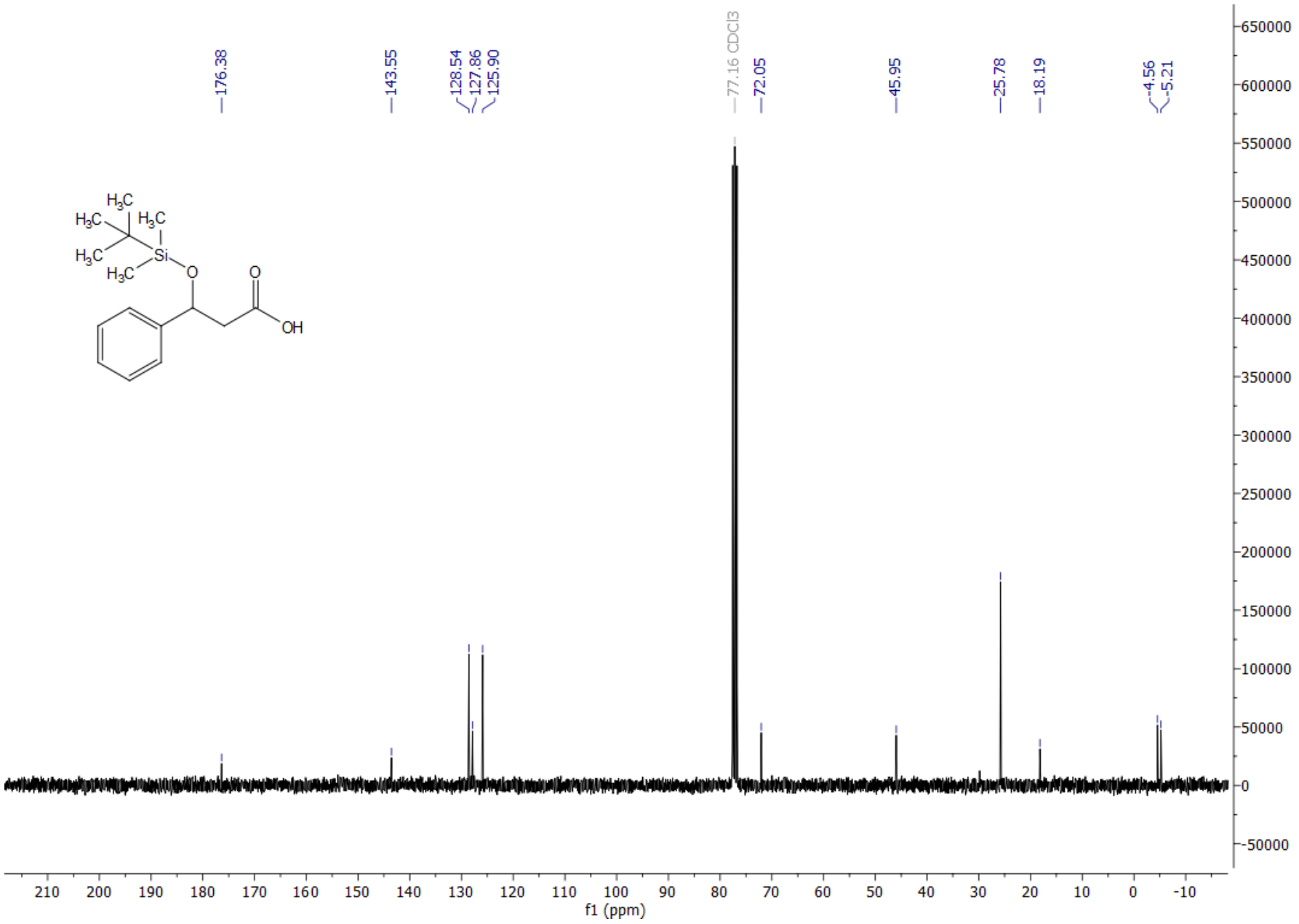
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **1-Phenylethanol, P3**



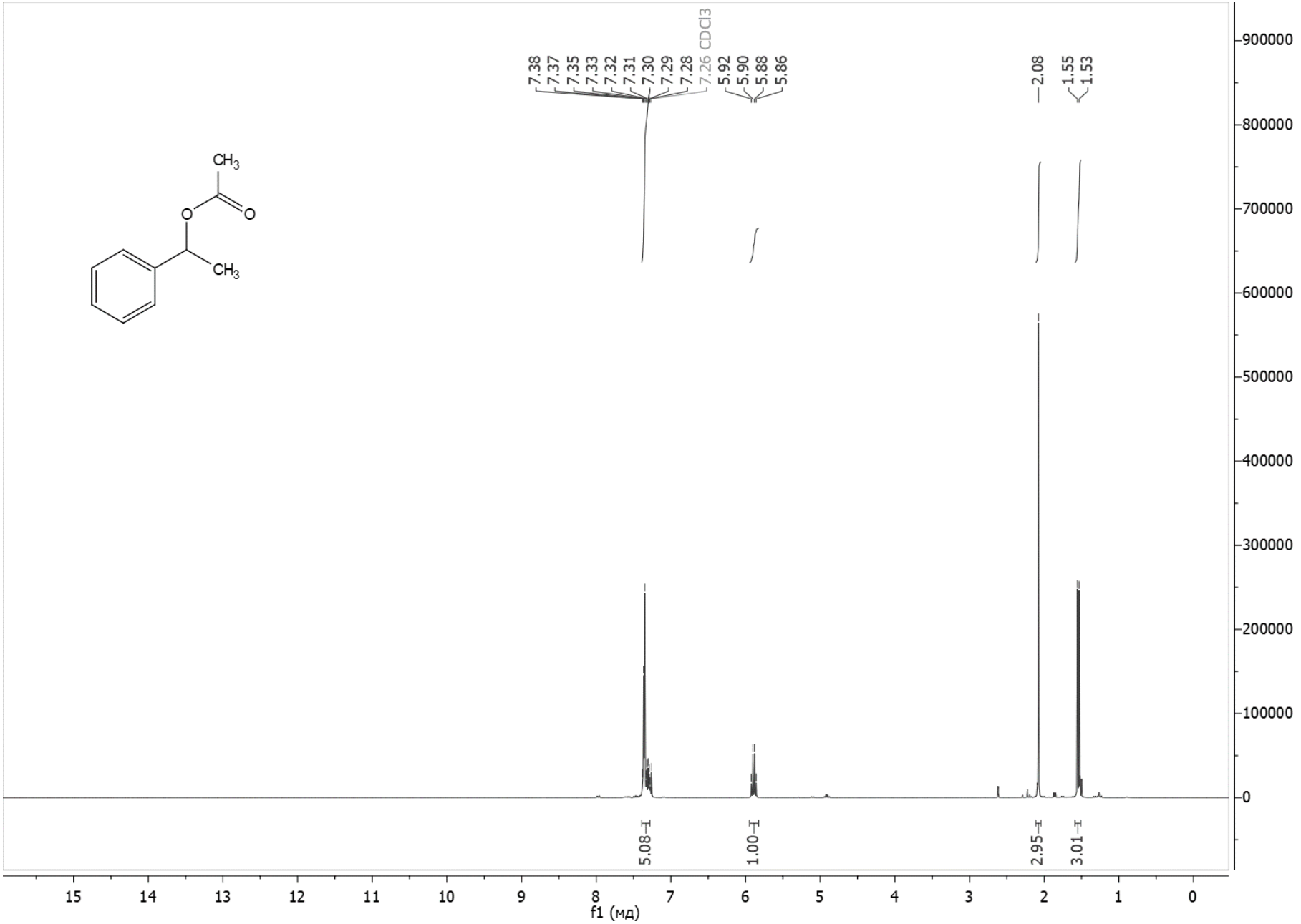
^{1}H NMR (300.13 MHz, $CDCl_3$), **3-((*Tert*-butyldimethylsilyl)oxy)-3-phenylpropanoic acid, P4**



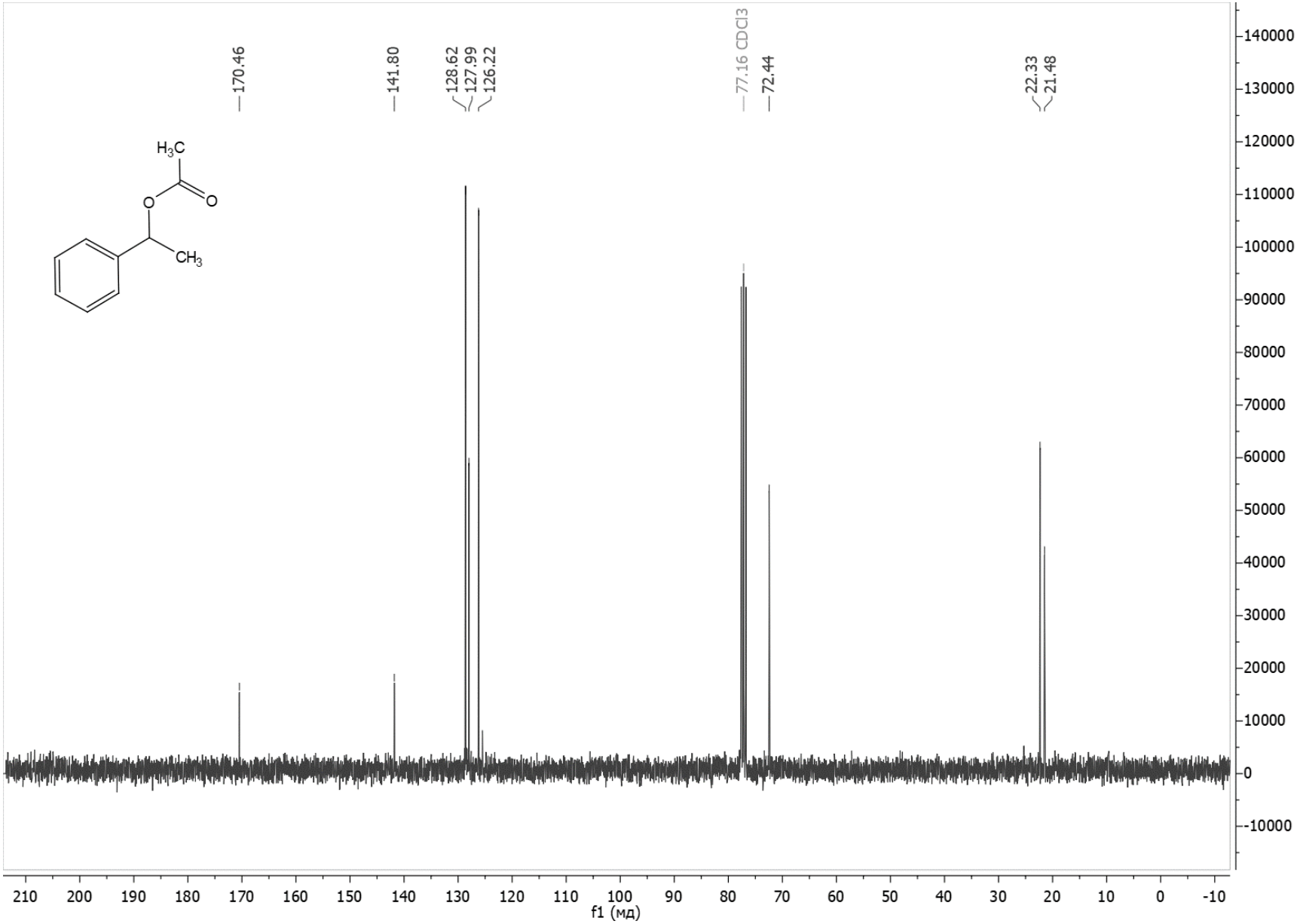
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **3-((*Tert*-butyldimethylsilyl)oxy)-3-phenylpropanoic acid**, **P4**



^{1}H NMR (300.13 MHz, $CDCl_3$), **1-Phenylethyl acetate, P6**

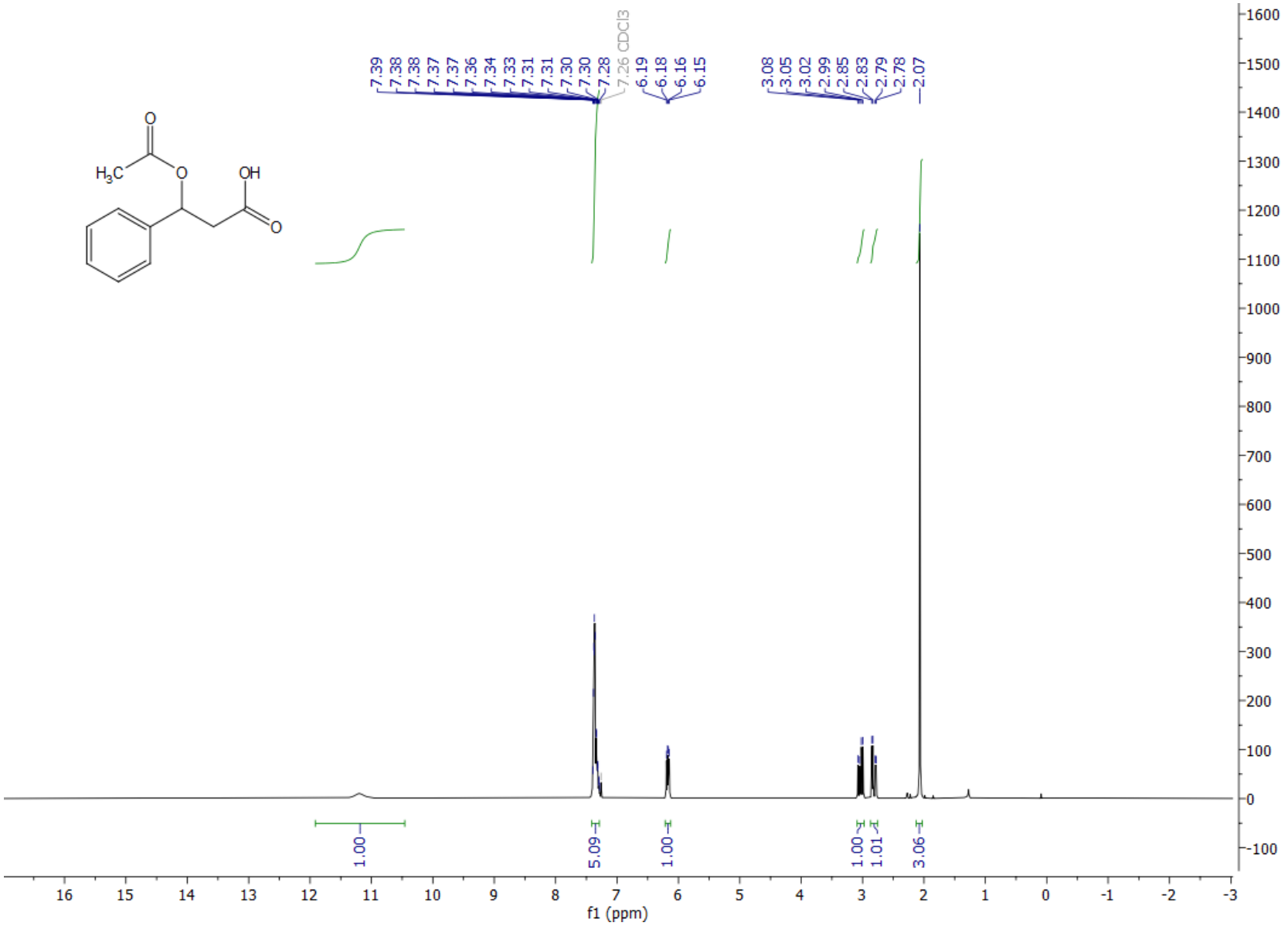


$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **1-Phenylethyl acetate, P6**

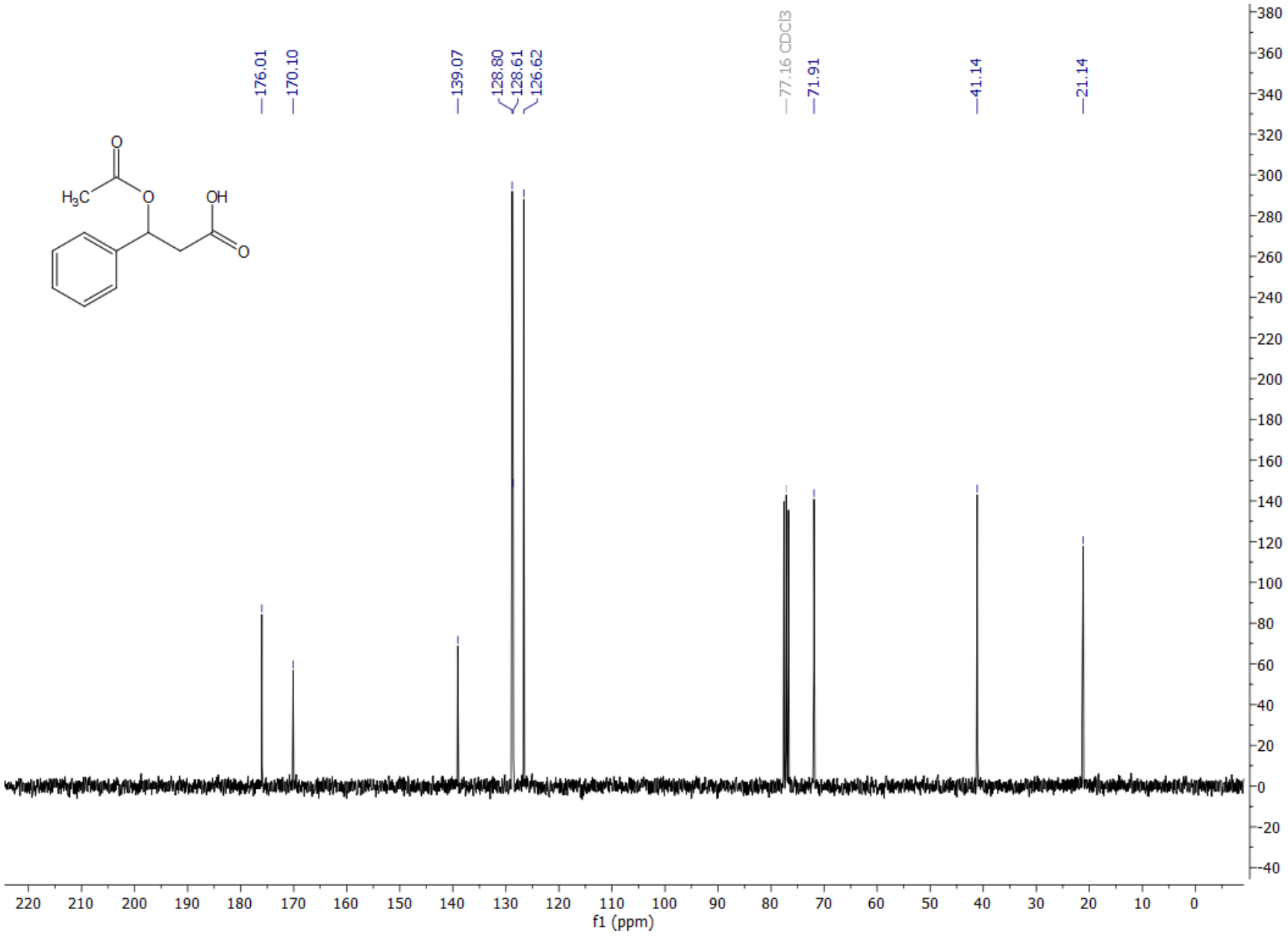


NMR spectra of products 2a-p

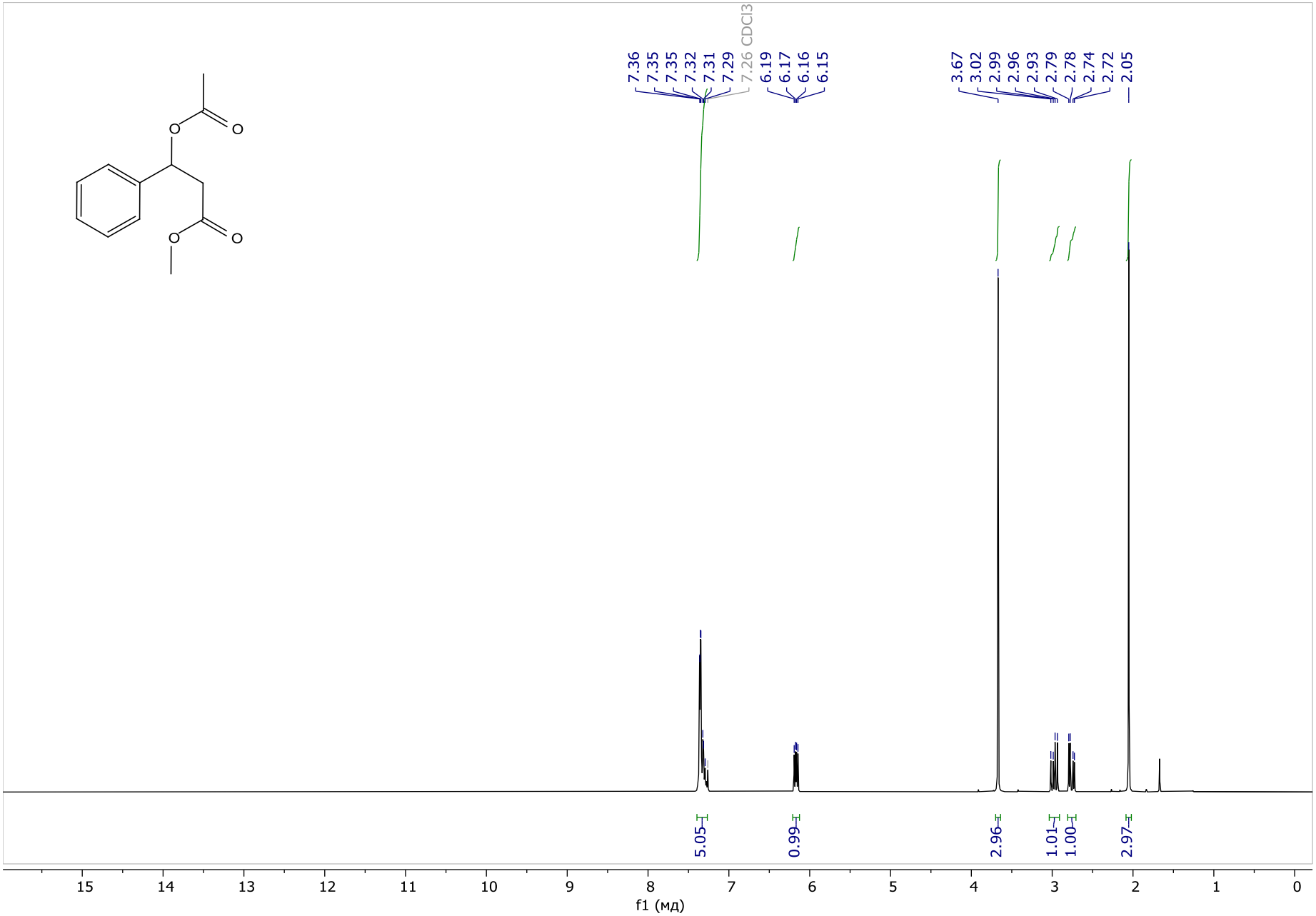
^{1}H NMR (300.13 MHz, $CDCl_3$), **3-Acetoxy-3-phenylpropanoic acid, 2a**



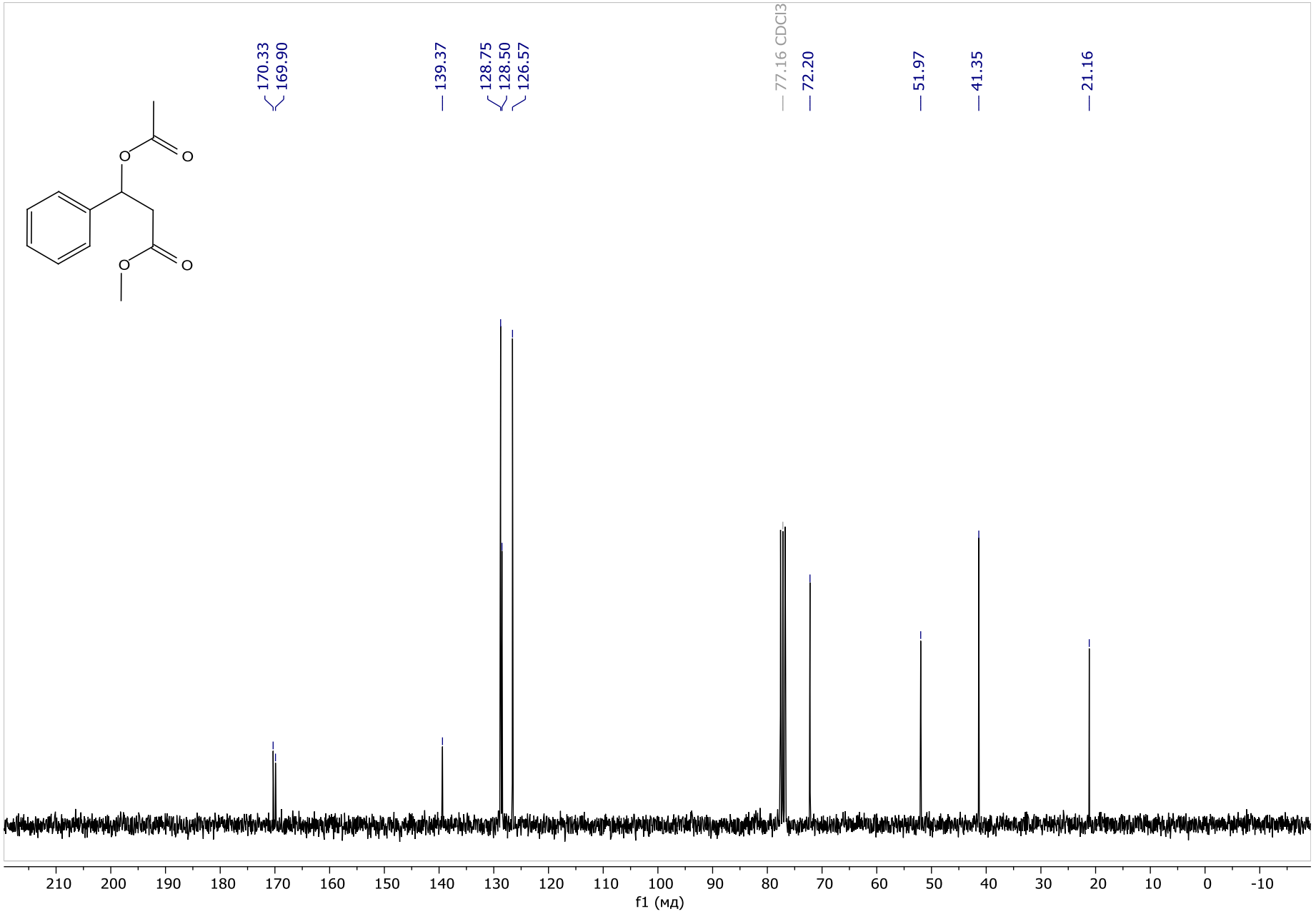
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **3-Acetoxy-3-phenylpropanoic acid, 2a**



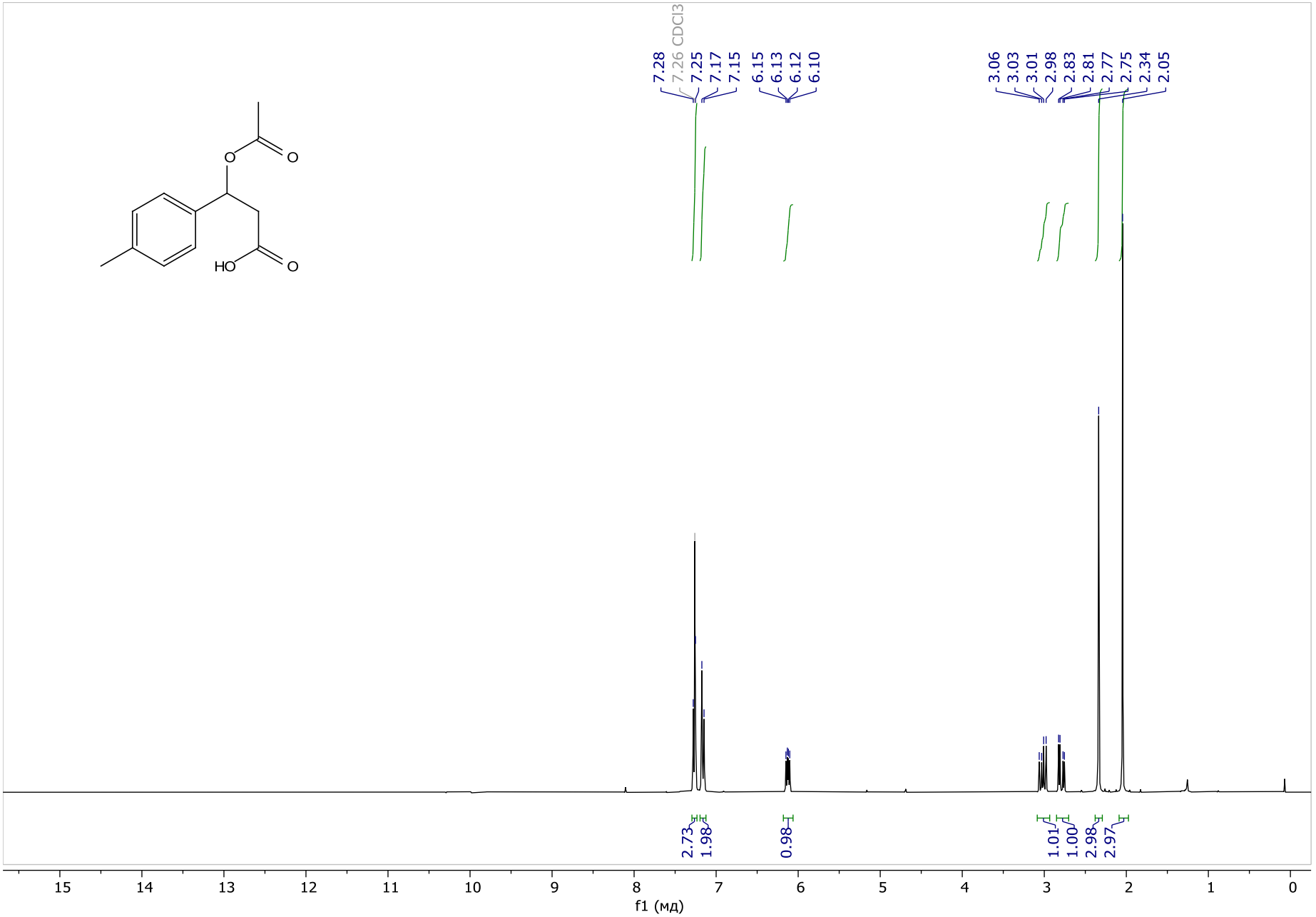
^{1}H NMR (300.13 MHz, $CDCl_3$), **Methyl 3-acetoxy-3-phenylpropanoate, 2a'**



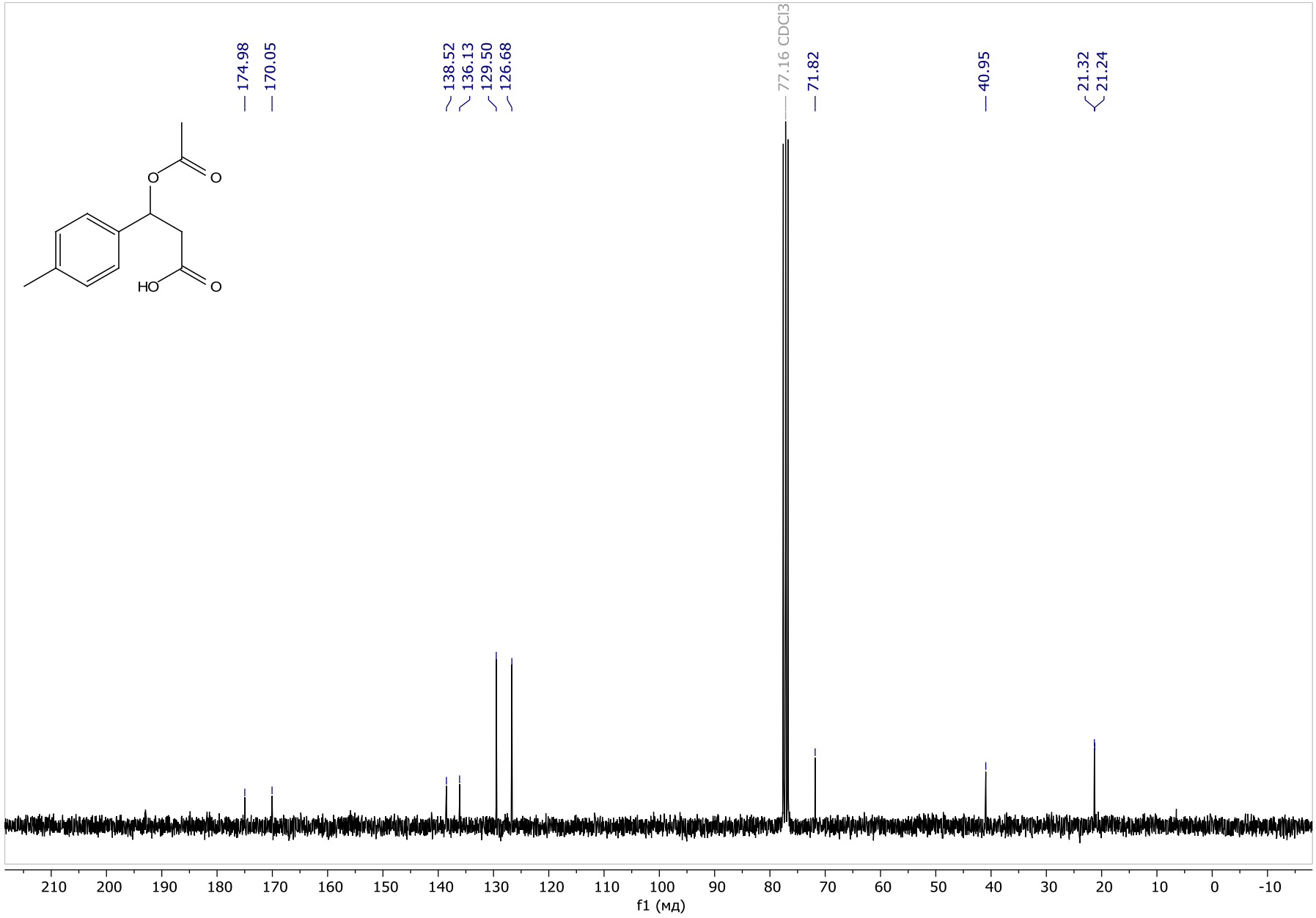
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **Methyl 3-acetoxy-3-phenylpropanoate, 2a'**



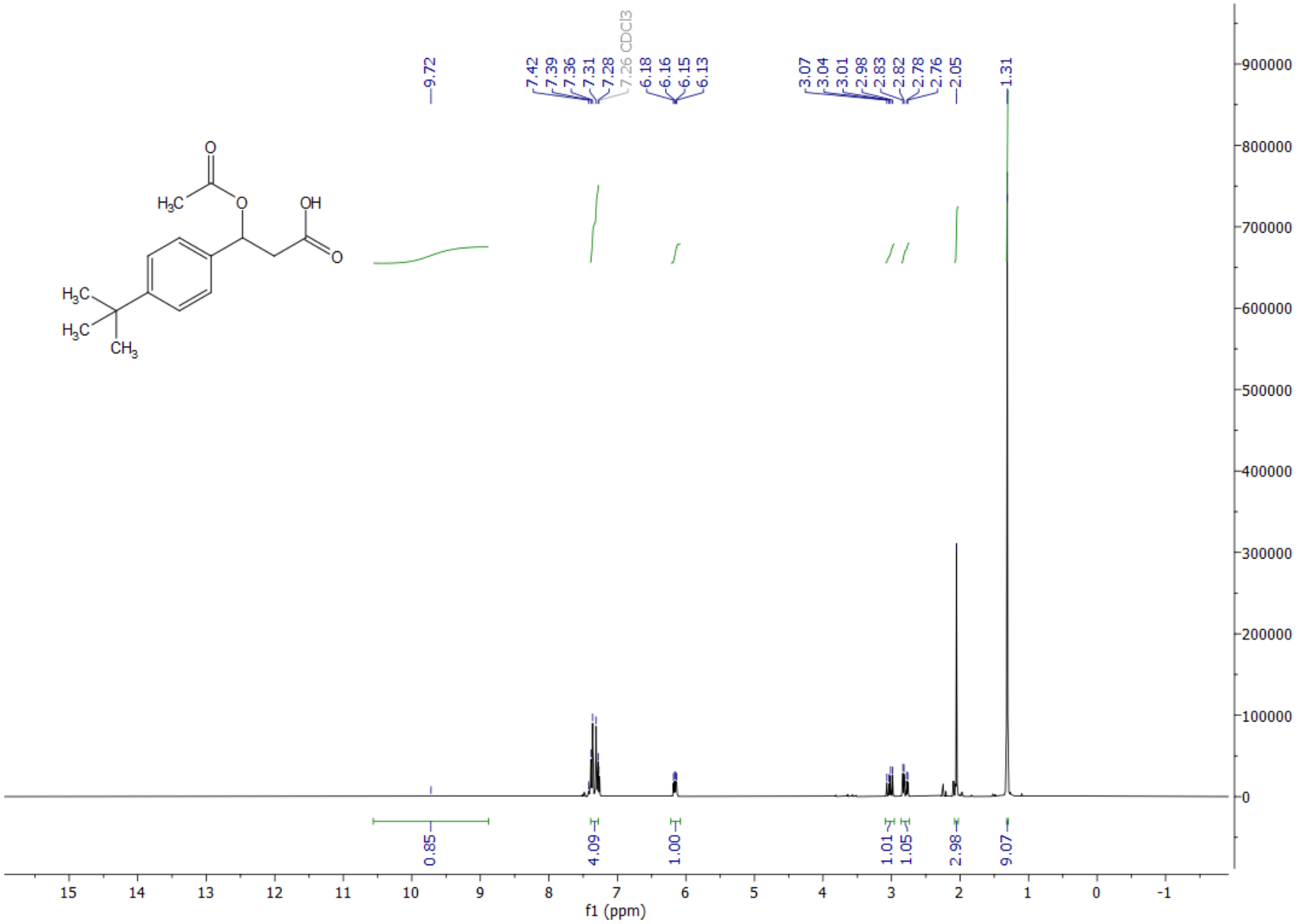
^{1}H NMR (300.13 MHz, $CDCl_3$), **3-Acetoxy-3-(*p*-tolyl)propanoic acid, 2b**



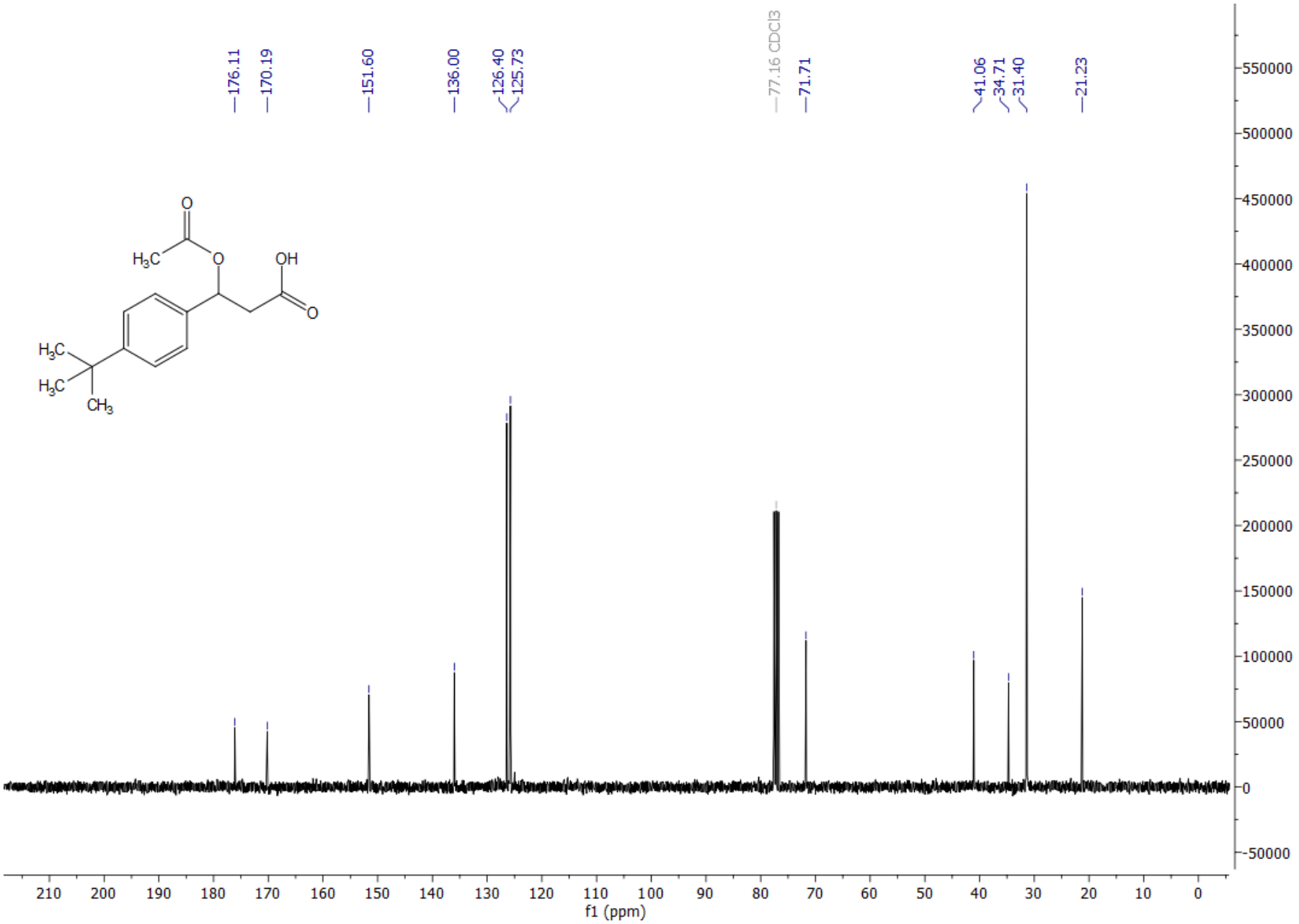
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$),**3-Acetoxy-3-(*p*-tolyl)propanoic acid, 2b**



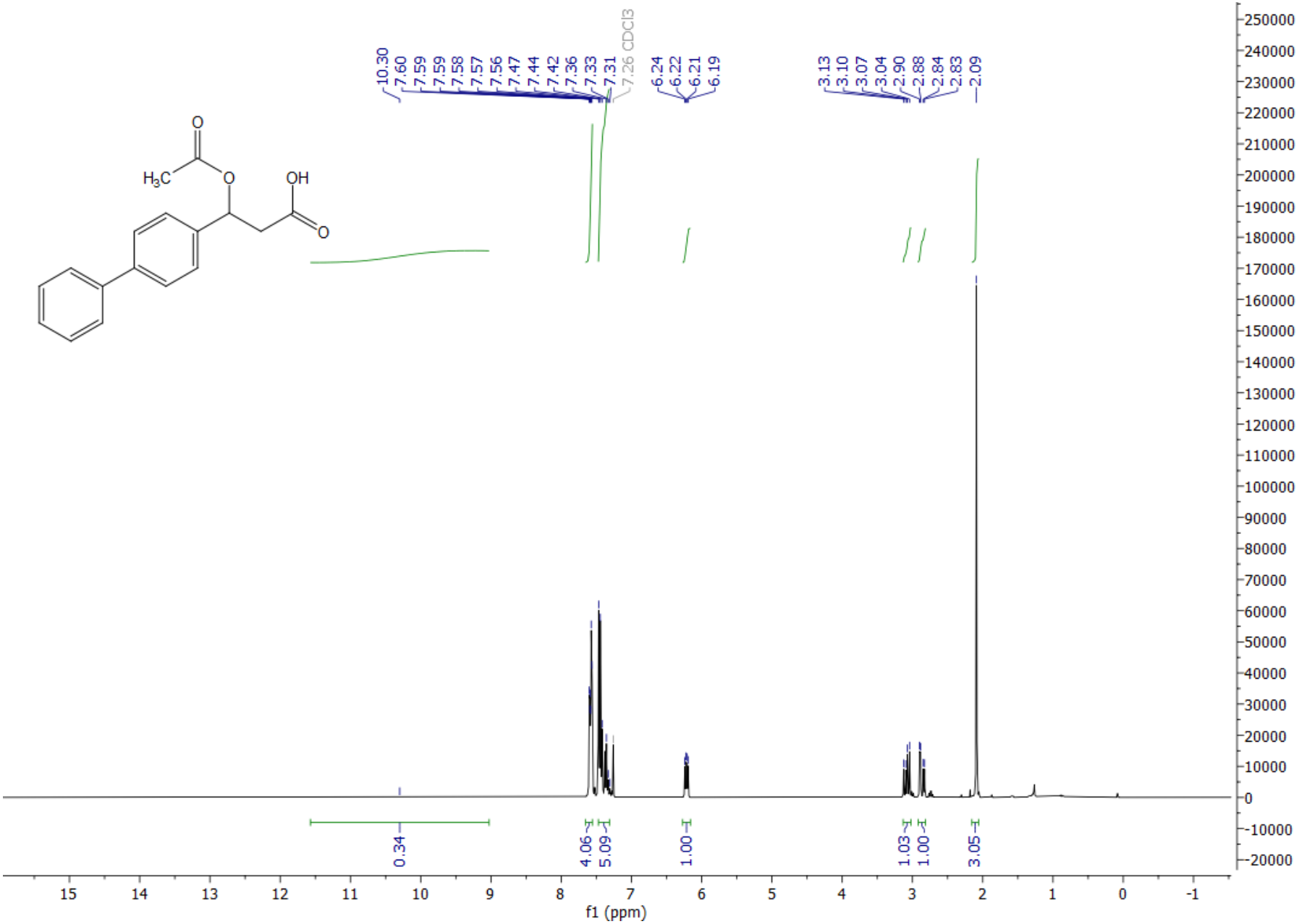
^{1}H NMR (300.13 MHz, $CDCl_3$), **3-Acetoxy-3-(4-(*tert*-butyl)phenyl)propanoic acid, 2c**



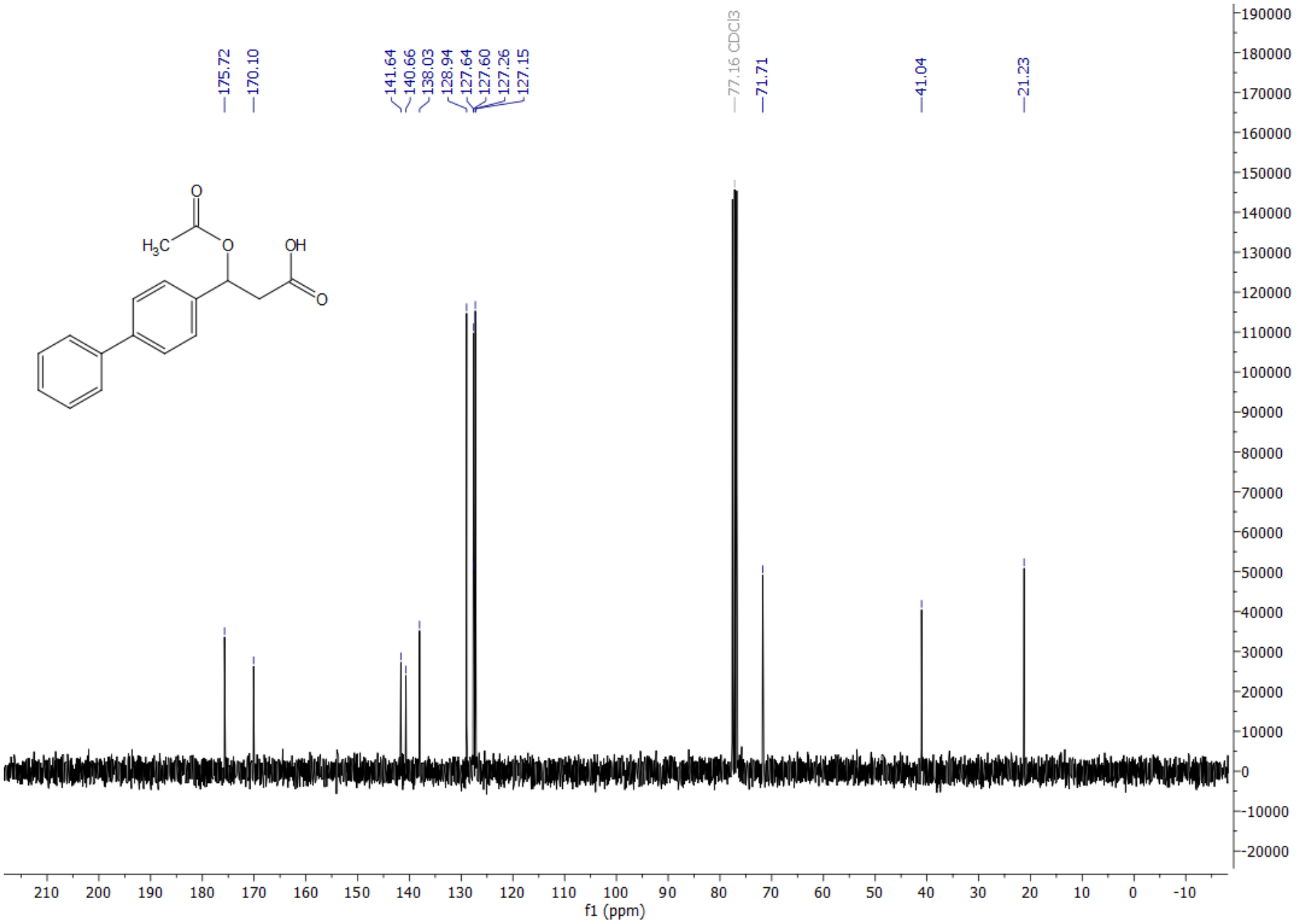
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **3-Acetoxy-3-(4-(*tert*-butyl)phenyl)propanoic acid, 2c**



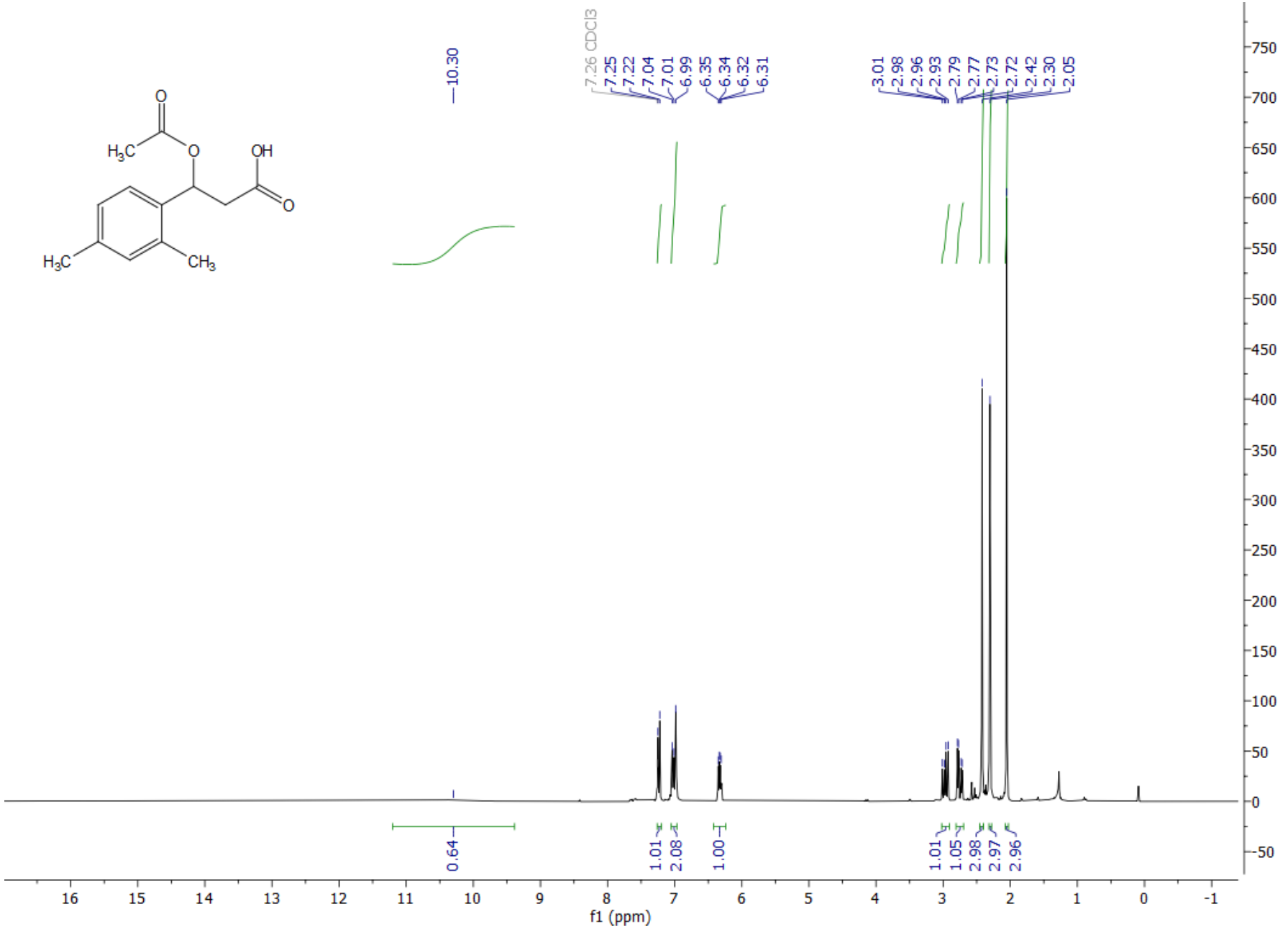
^{1}H NMR (300.13 MHz, $CDCl_3$), **3-([1,1'-Biphenyl]-4-yl)-3-acetoxypropanoic acid, 2d**



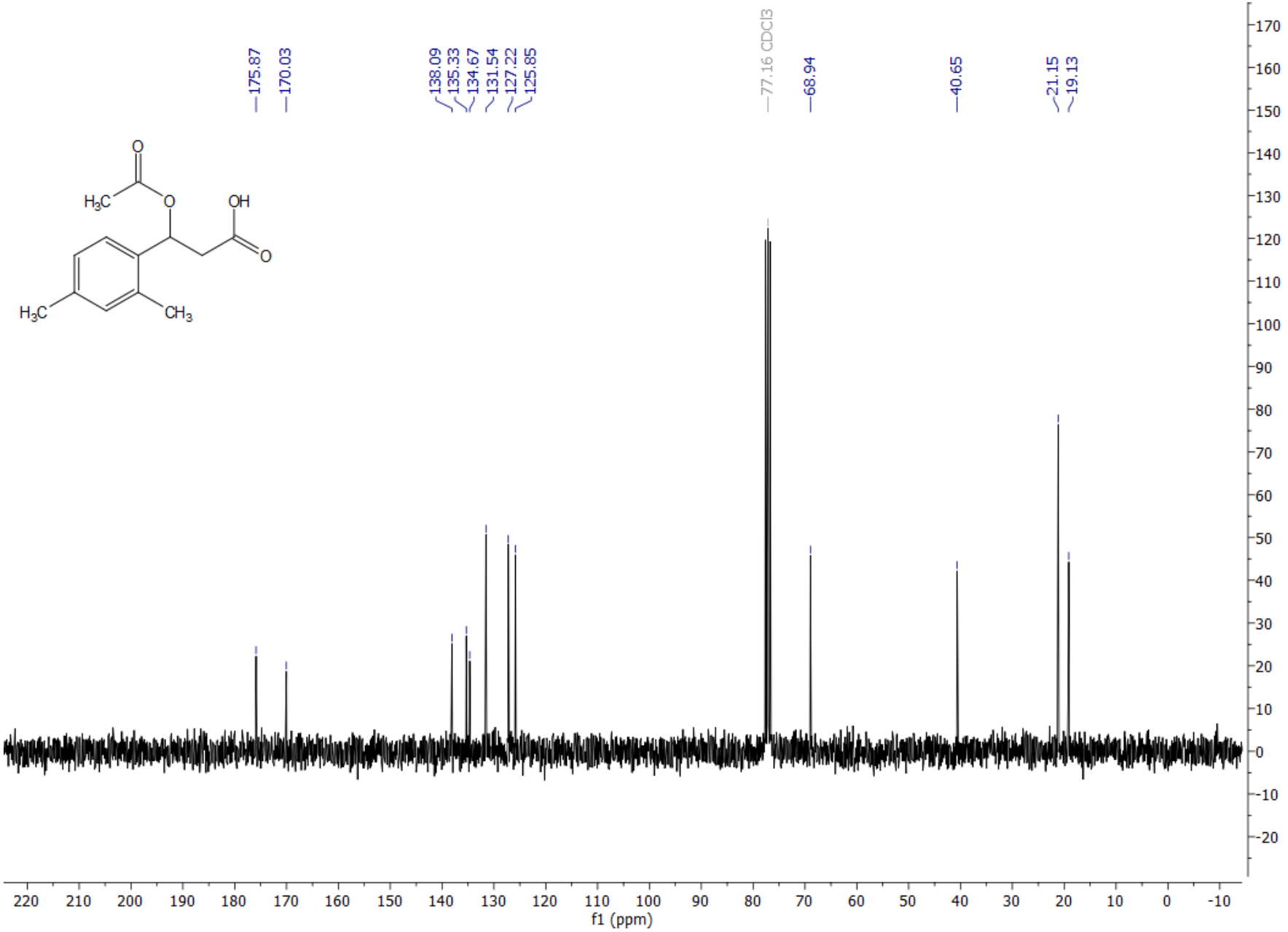
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **3-([1,1'-Biphenyl]-4-yl)-3-acetoxypropanoic acid, 2d**



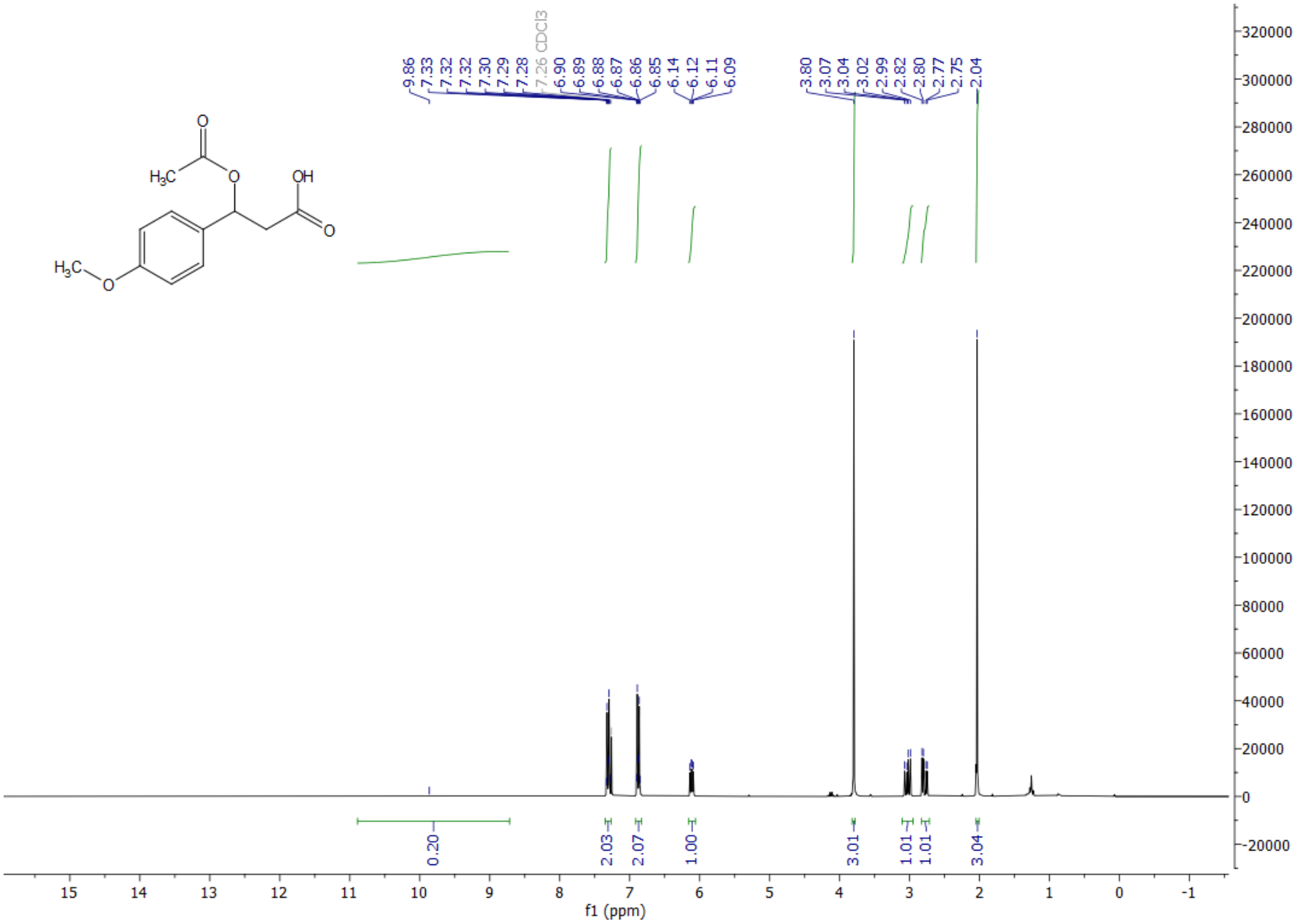
^{1}H NMR (300.13 MHz, $CDCl_3$), **3-Acetoxy-3-(2,4-dimethylphenyl)propanoic acid, 2e**



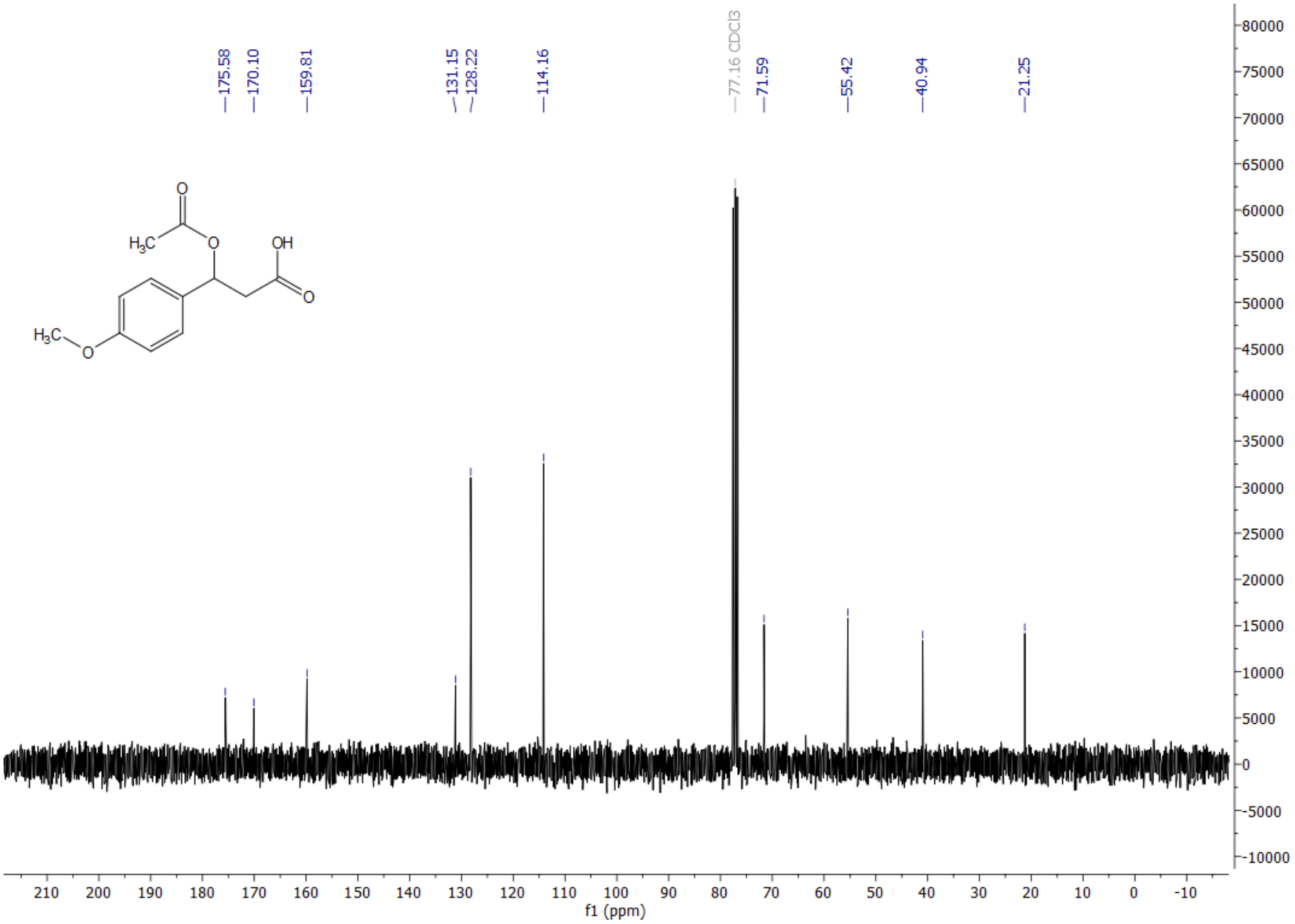
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **3-Acetoxy-3-(2,4-dimethylphenyl)propanoic acid, 2e**



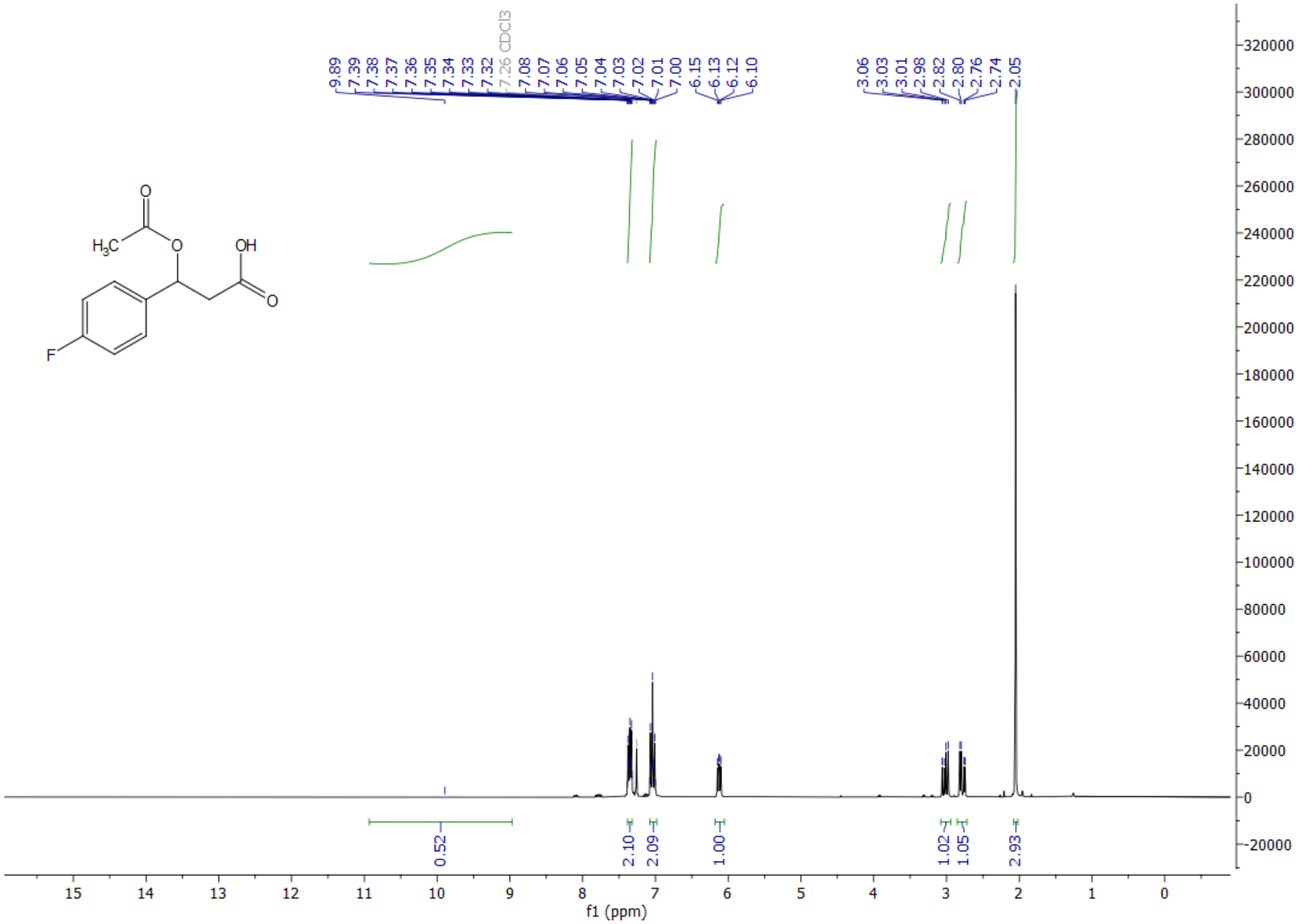
^{1}H NMR (300.13 MHz, $CDCl_3$), **3-Acetoxy-3-(4-methoxyphenyl)propanoic acid, 2f**



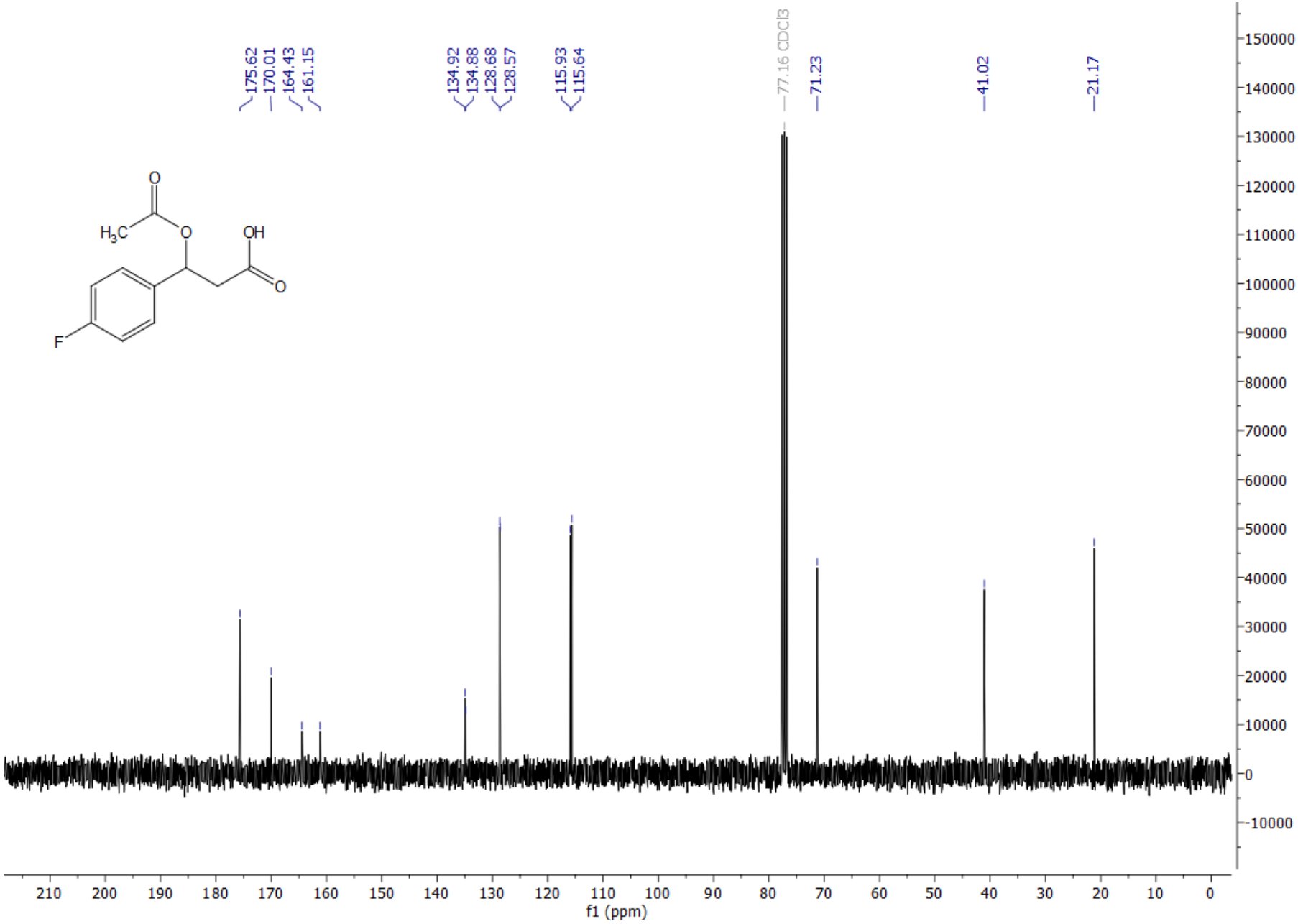
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **3-Acetoxy-3-(4-methoxyphenyl)propanoic acid, 2f**



^{1}H NMR (300.13 MHz, $CDCl_3$), **3-Acetoxy-3-(4-fluorophenyl)propanoic acid, 2g**

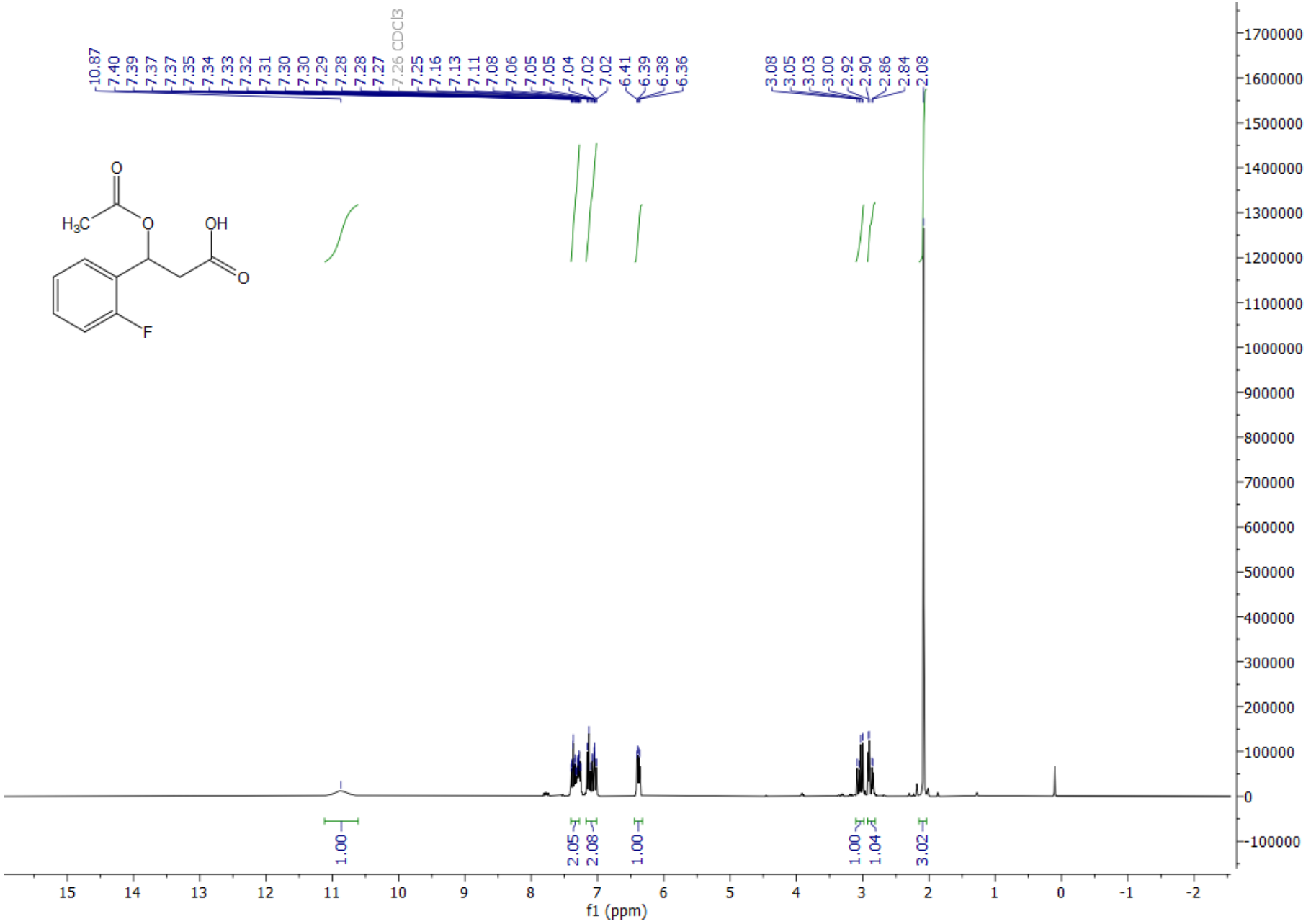


$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **3-Acetoxy-3-(4-fluorophenyl)propanoic acid, 2g**

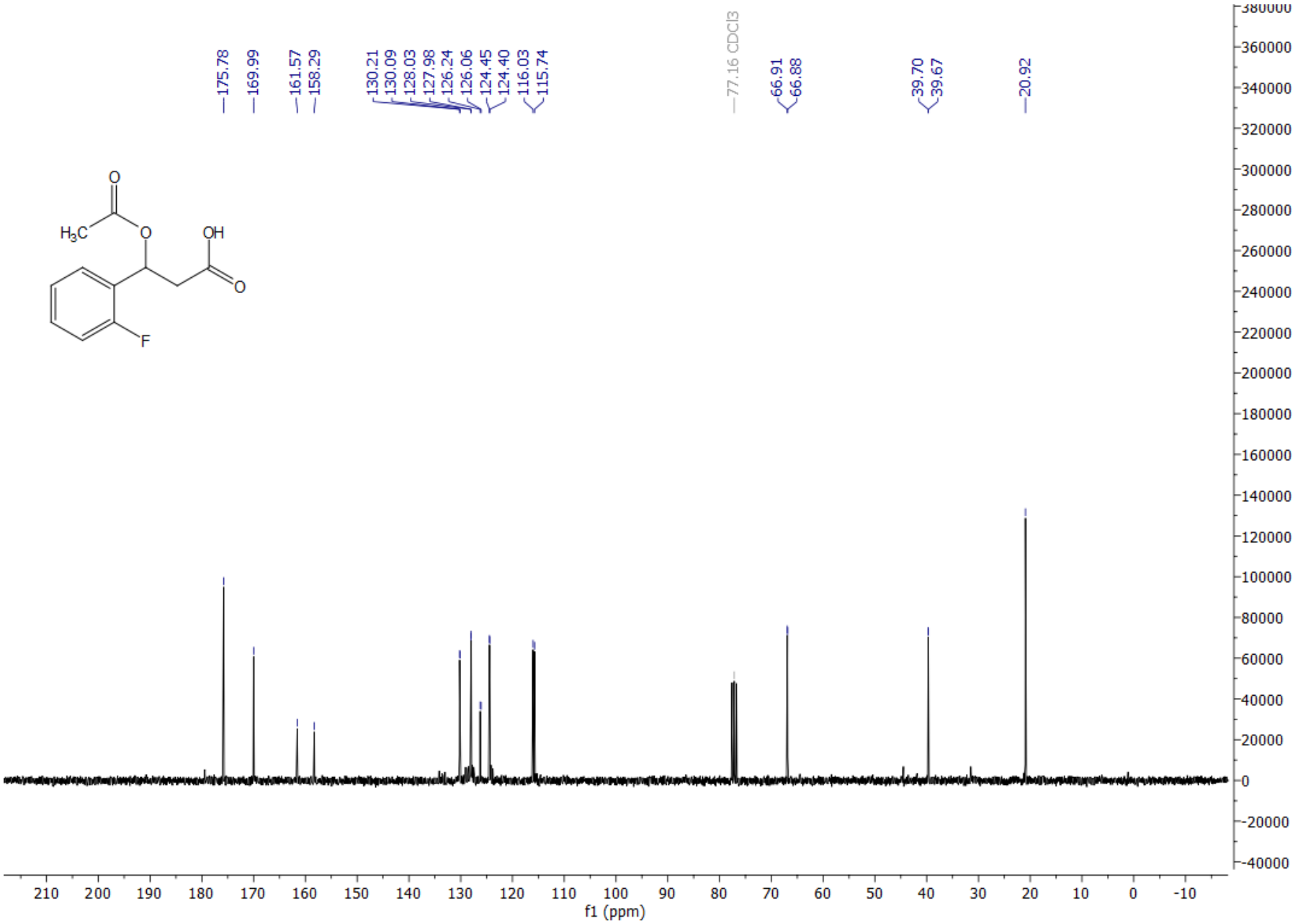


^{19}F NMR (282,5 MHz, $CDCl_3$),**3-Acetoxy-3-(4-fluorophenyl)propanoic acid, 2g**

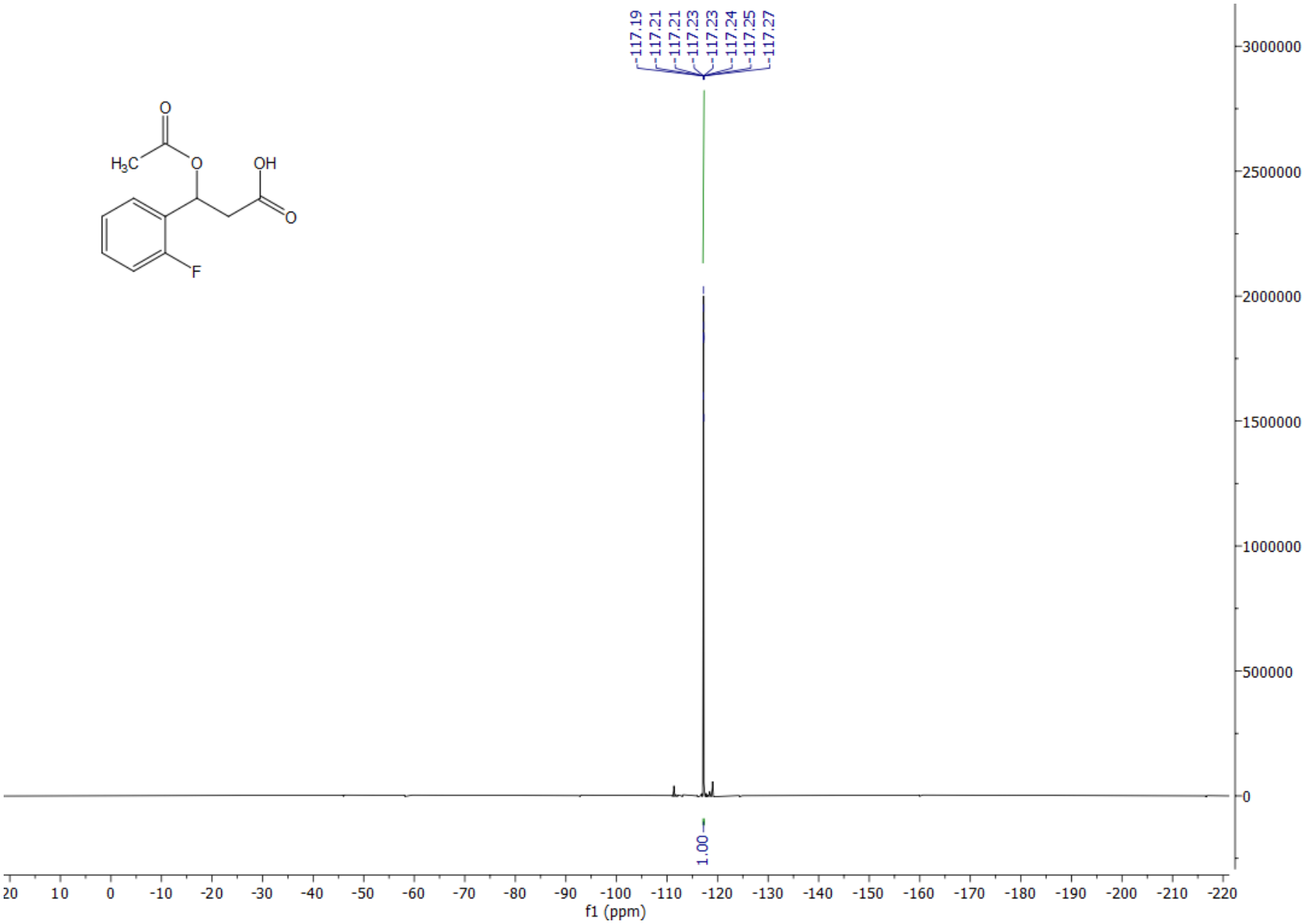
^{1}H NMR (300.13 MHz, $CDCl_3$), **3-Acetoxy-3-(2-fluorophenyl)propanoic acid, 2h**



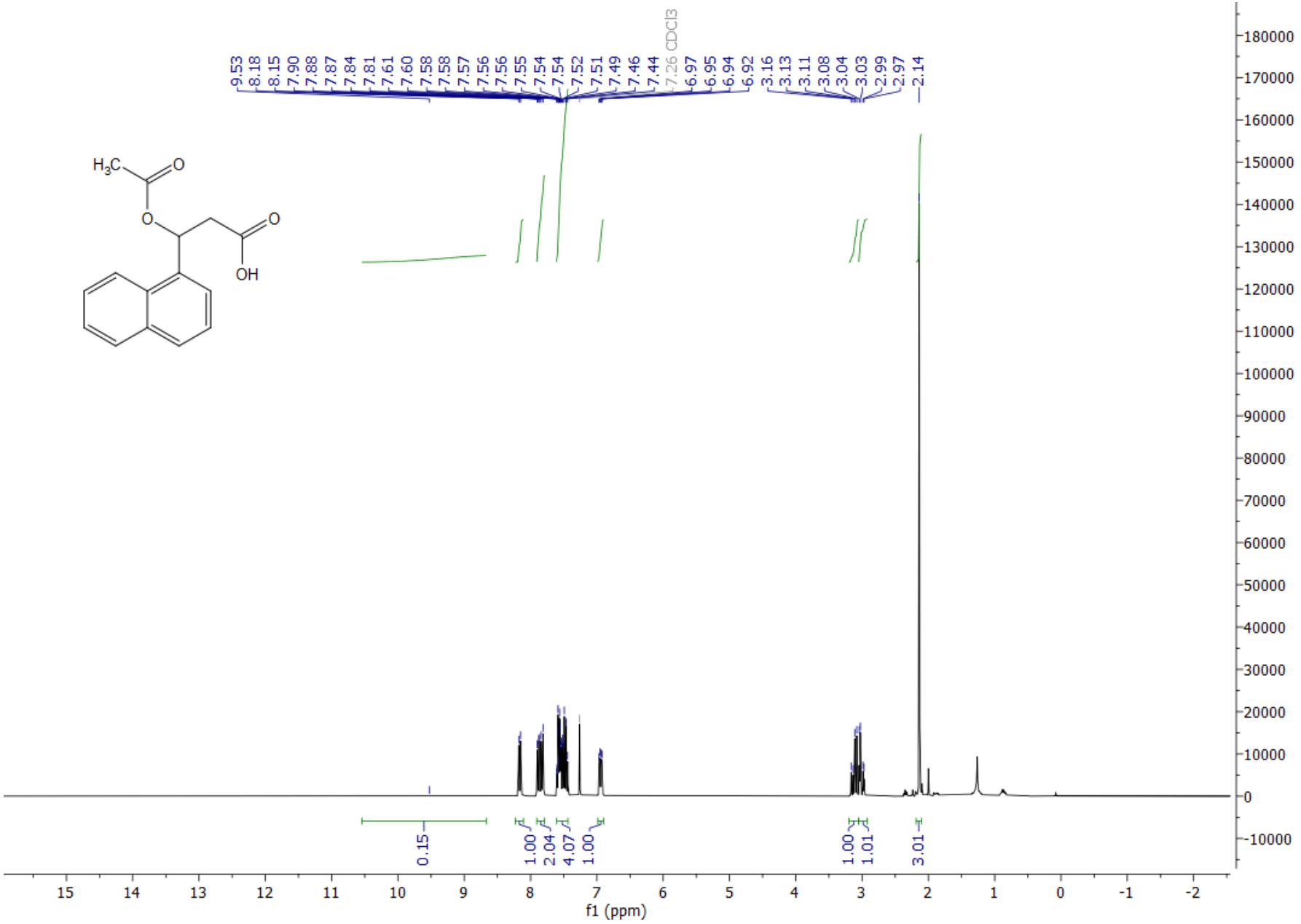
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **3-Acetoxy-3-(2-fluorophenyl)propanoic acid, 2h**



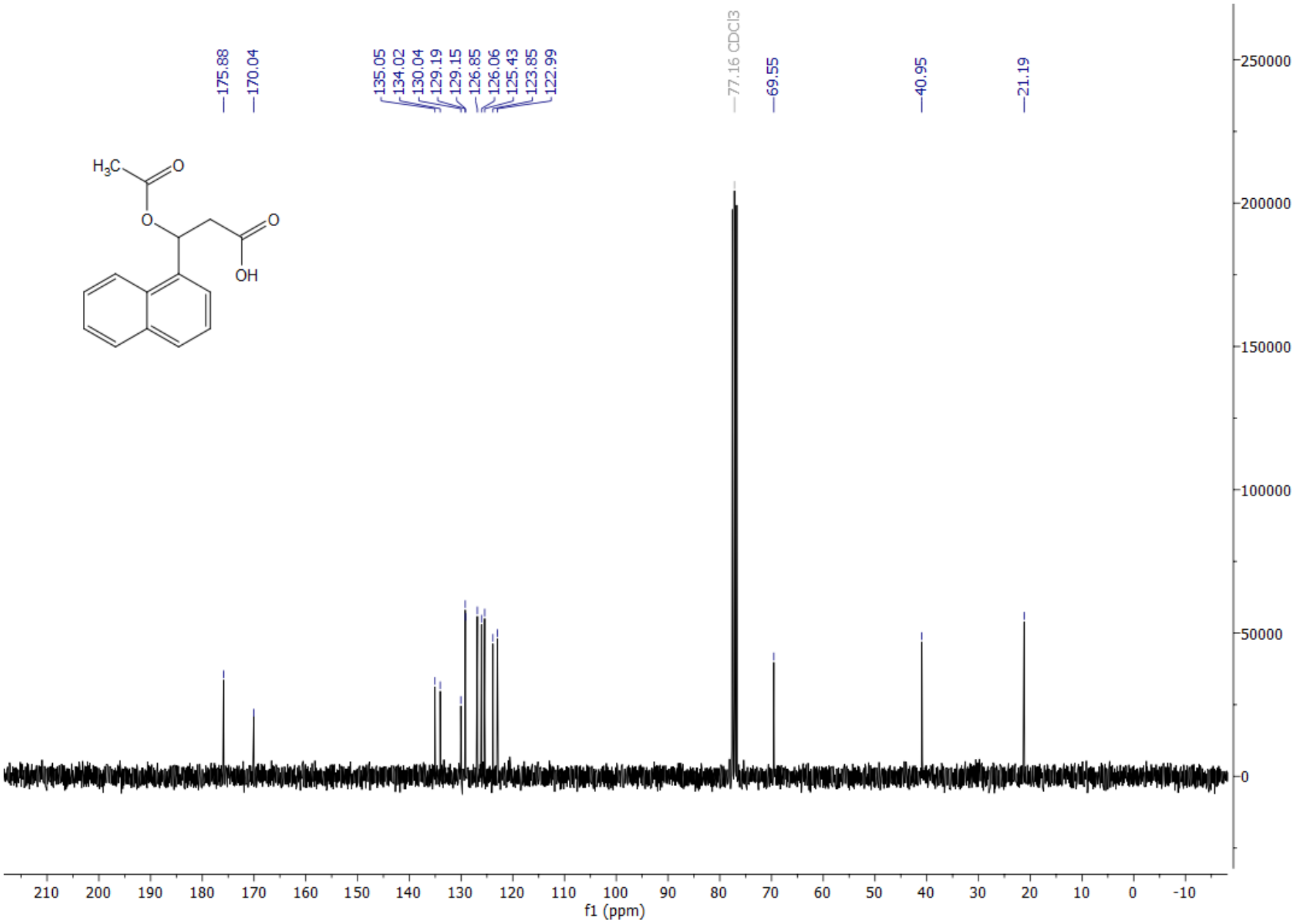
^{19}F NMR (282,5 MHz, $CDCl_3$), **3-Acetoxy-3-(2-fluorophenyl)propanoic acid, 2h**



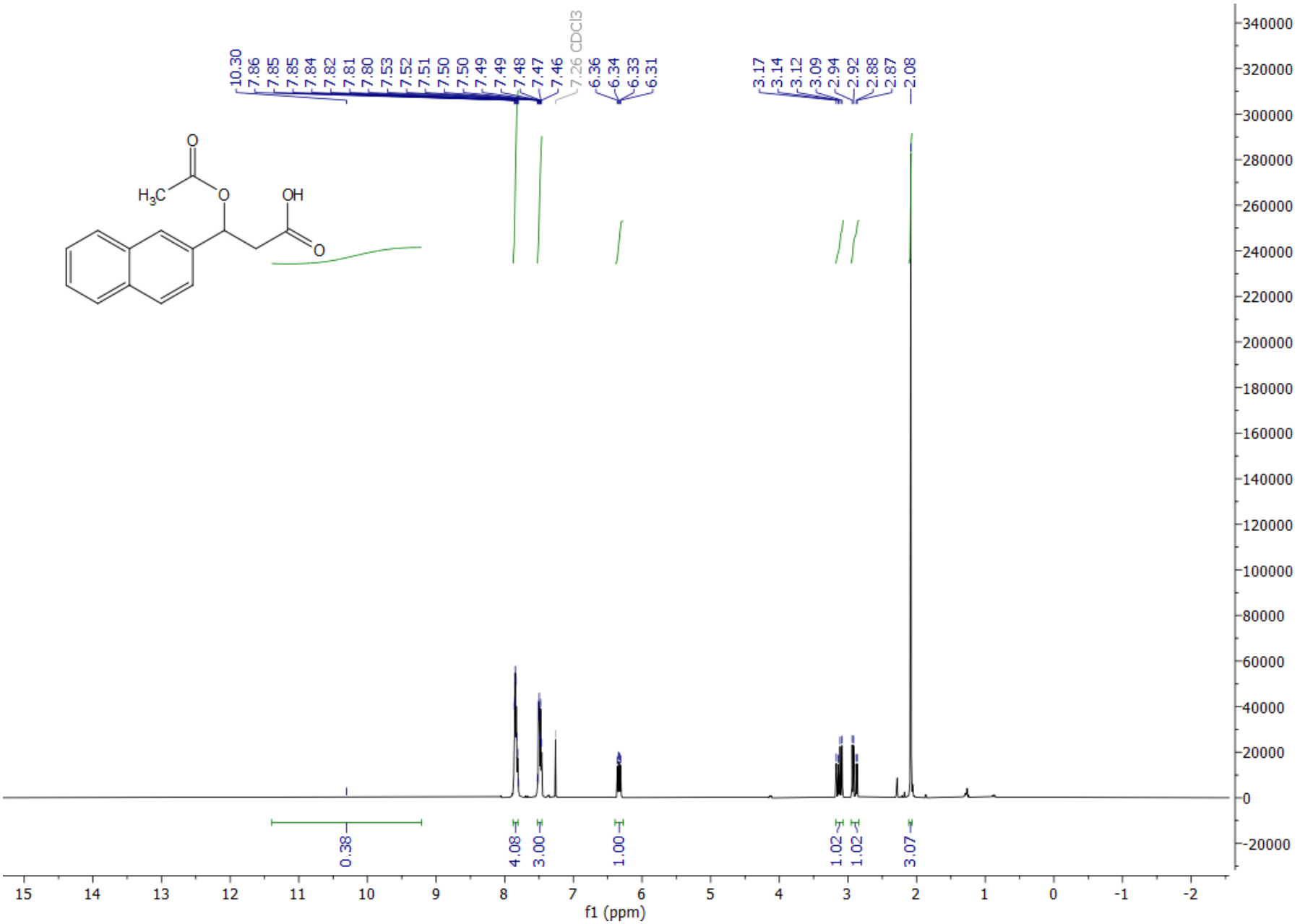
^{1}H NMR (300.13 MHz, $CDCl_3$), **3-Acetoxy-3-(naphthalen-1-yl)propanoic acid, 2i**



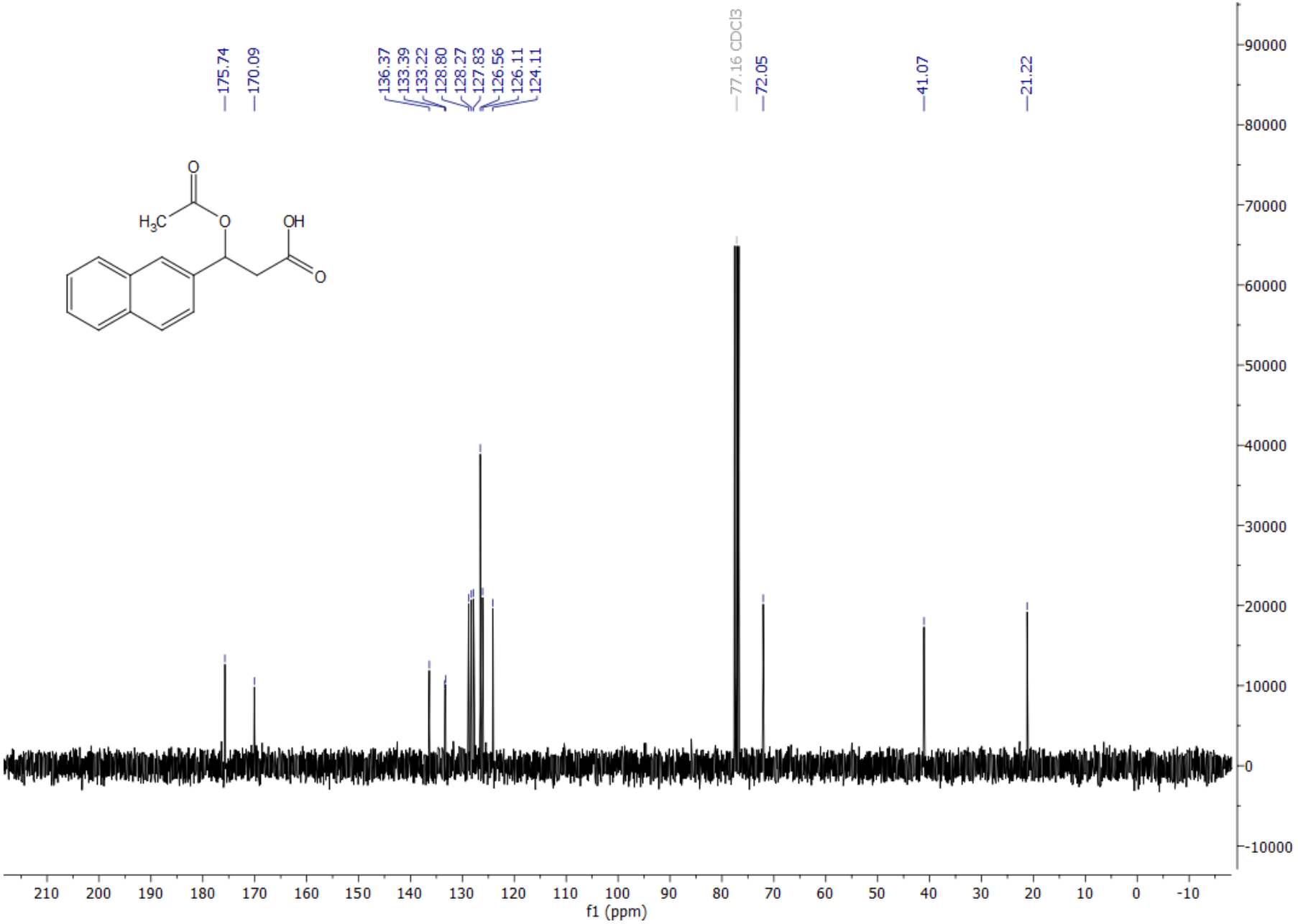
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **3-Acetoxy-3-(naphthalen-1-yl)propanoic acid, 2i**



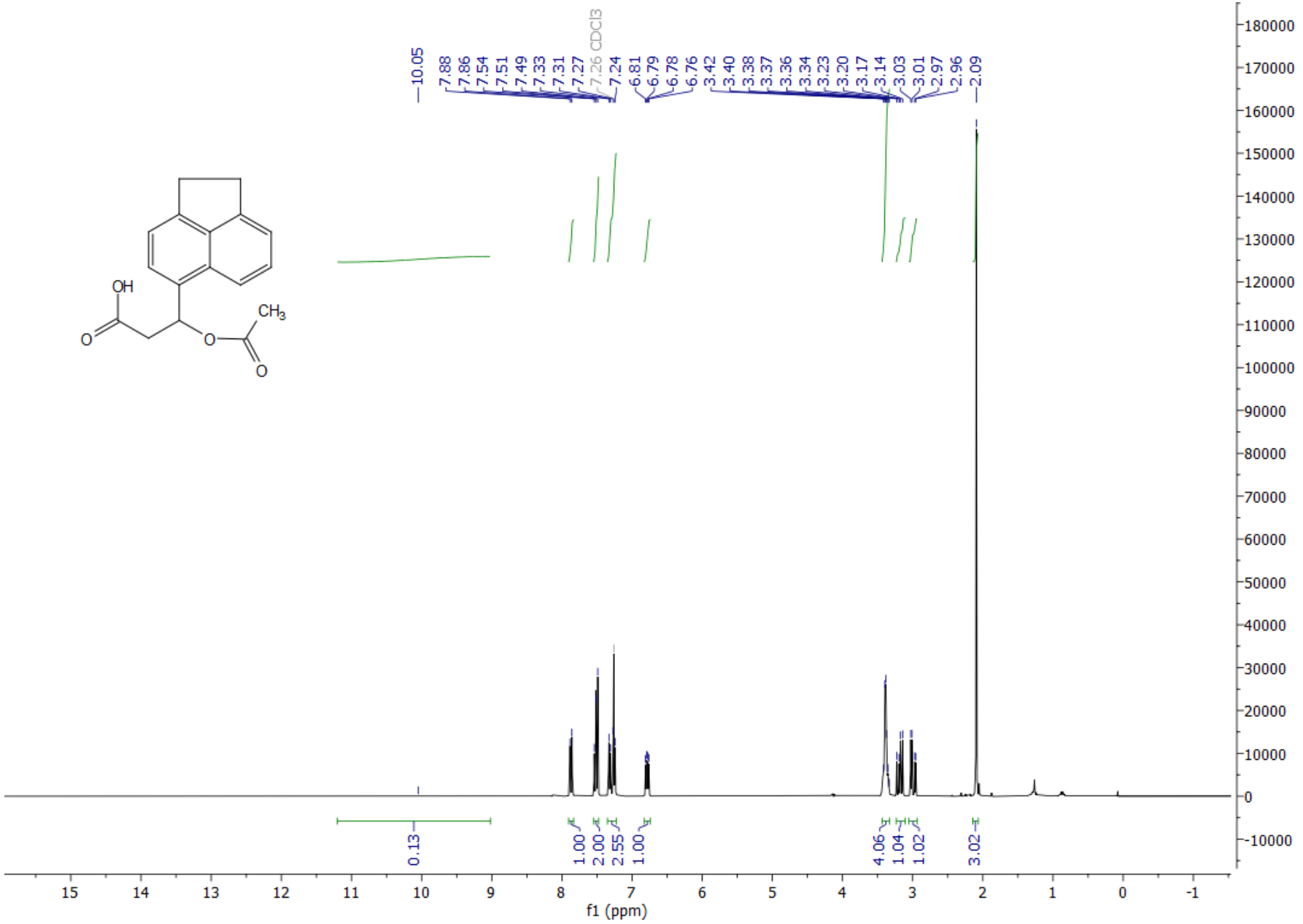
^{1}H NMR (300.13 MHz, $CDCl_3$), **3-Acetoxy-3-(naphthalen-2-yl)propanoic acid, 2j**



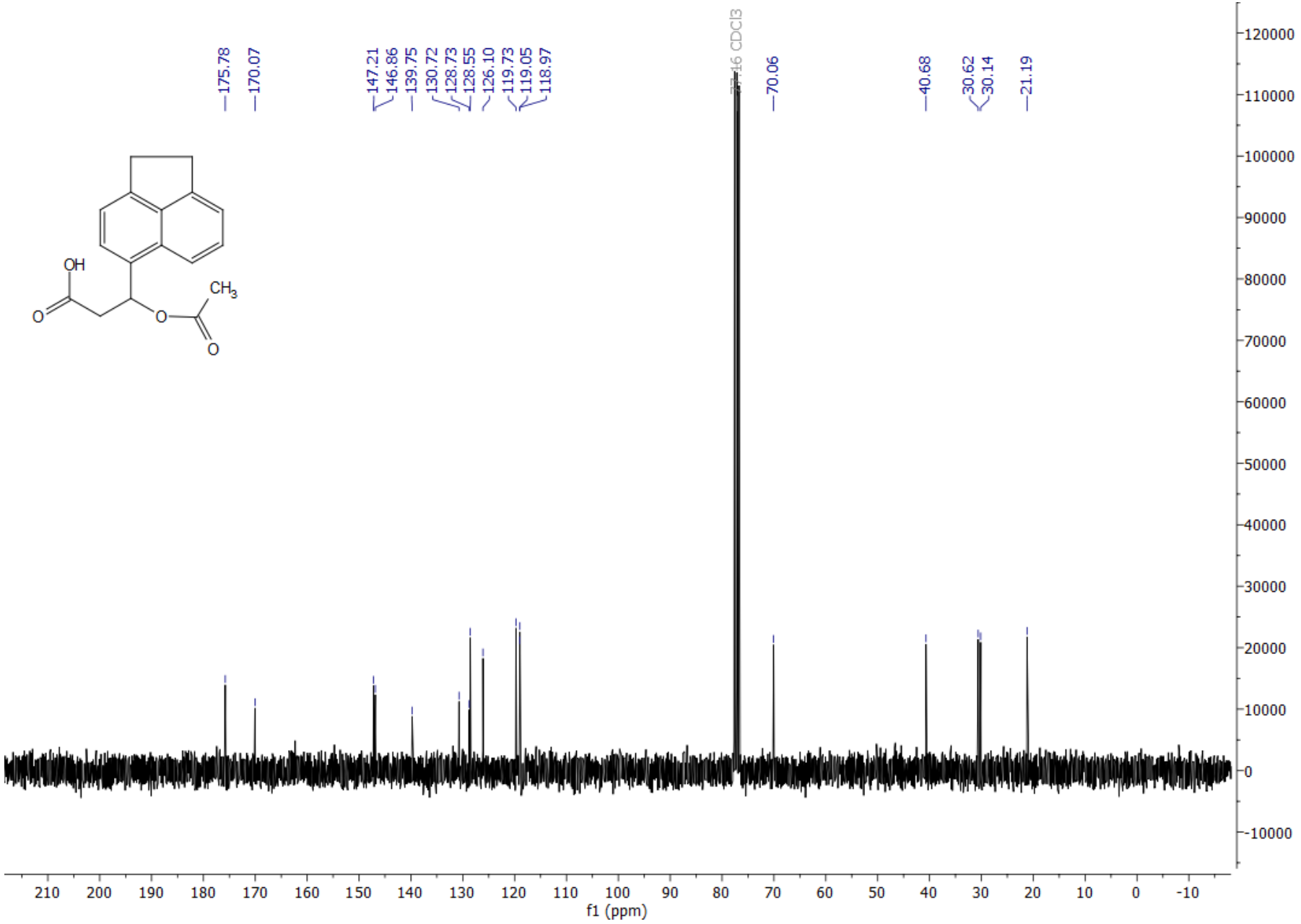
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **3-Acetoxy-3-(naphthalen-2-yl)propanoic acid, 2j**



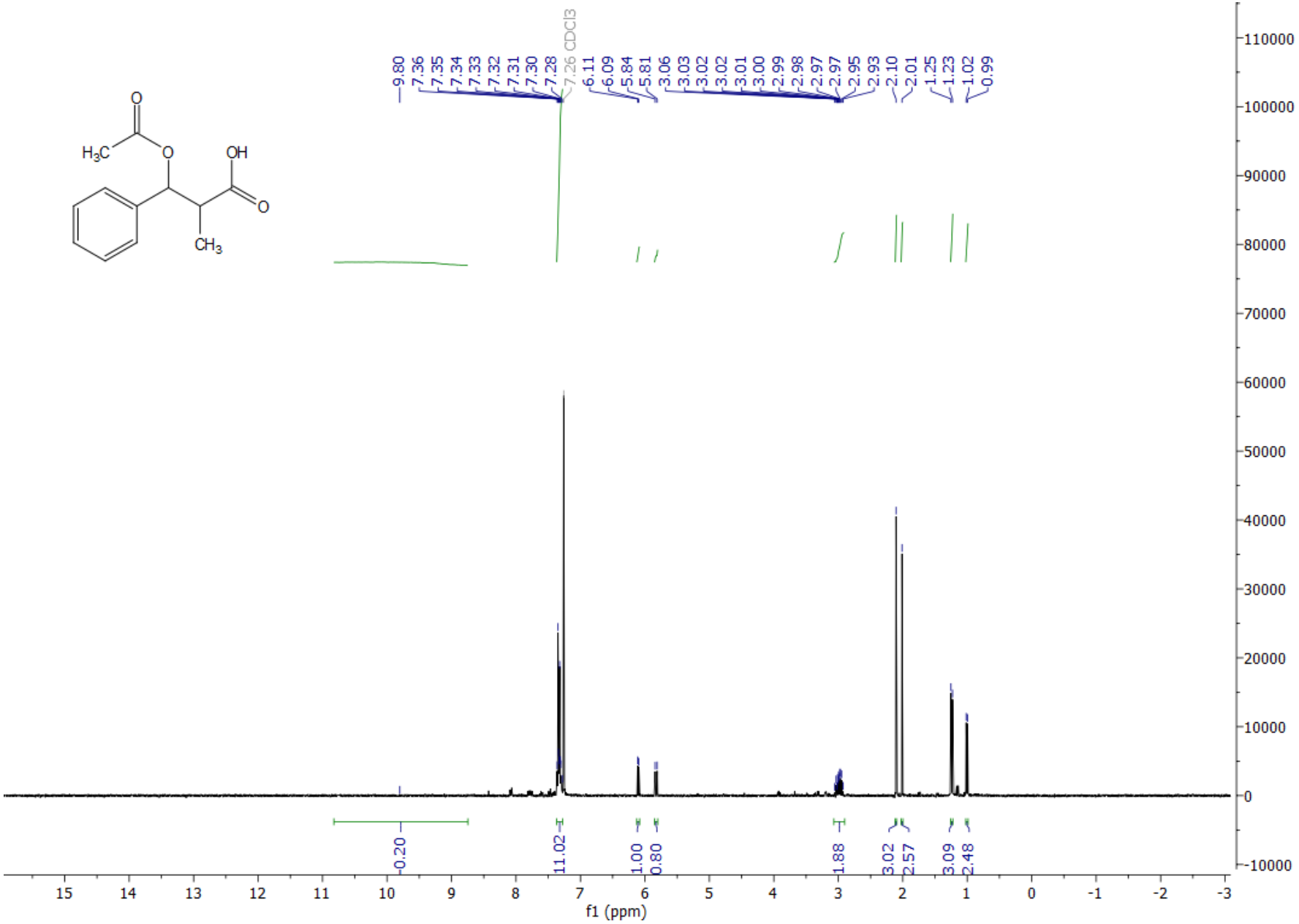
^{1}H NMR (300.13 MHz, $CDCl_3$), **3-Acetoxy-3-(1,2-dihydroacenaphthylen-5-yl)propanoic acid, 2k**



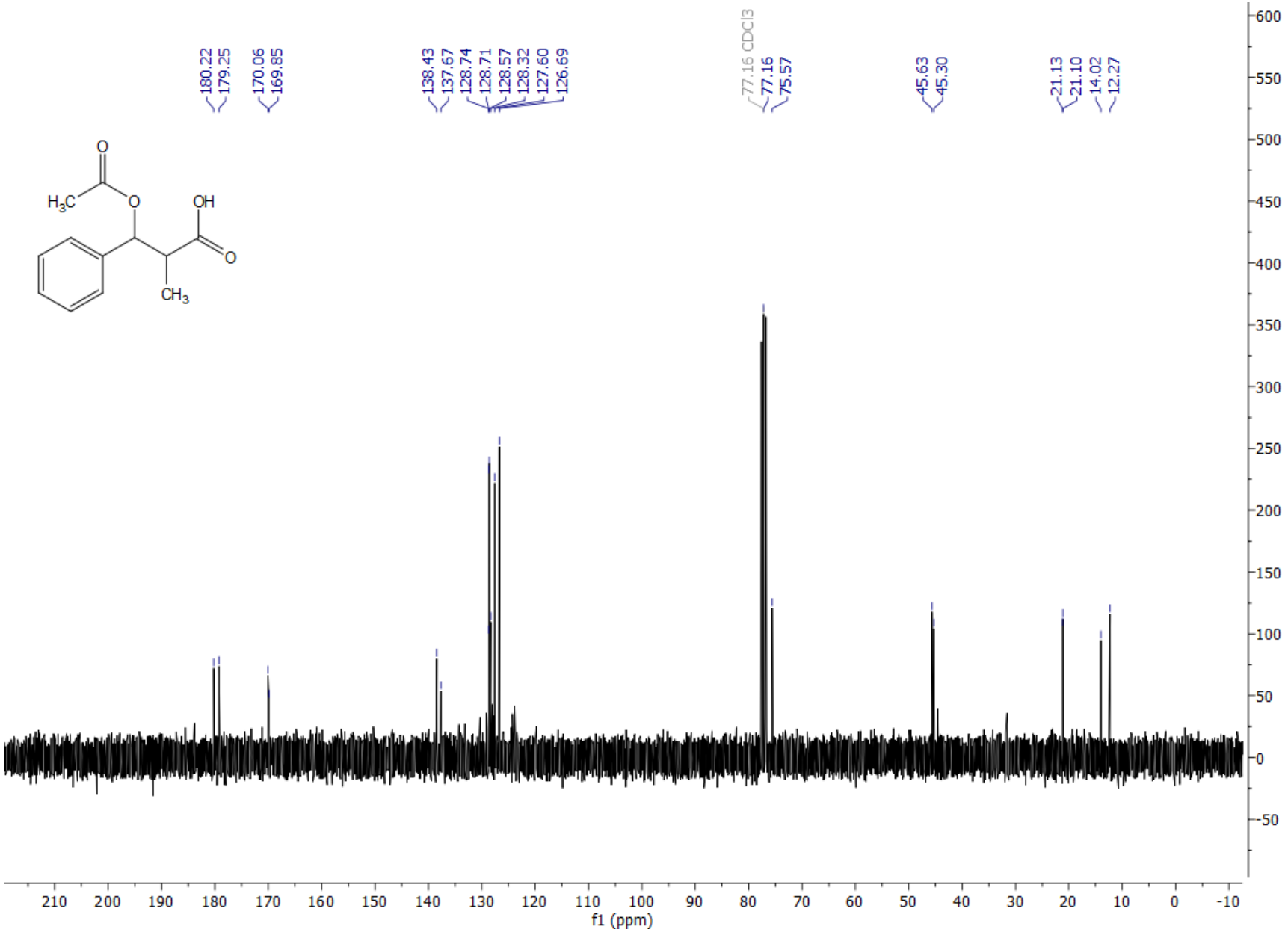
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **3-Acetoxy-3-(1,2-dihydroacenaphthylen-5-yl)propanoic acid, 2k**



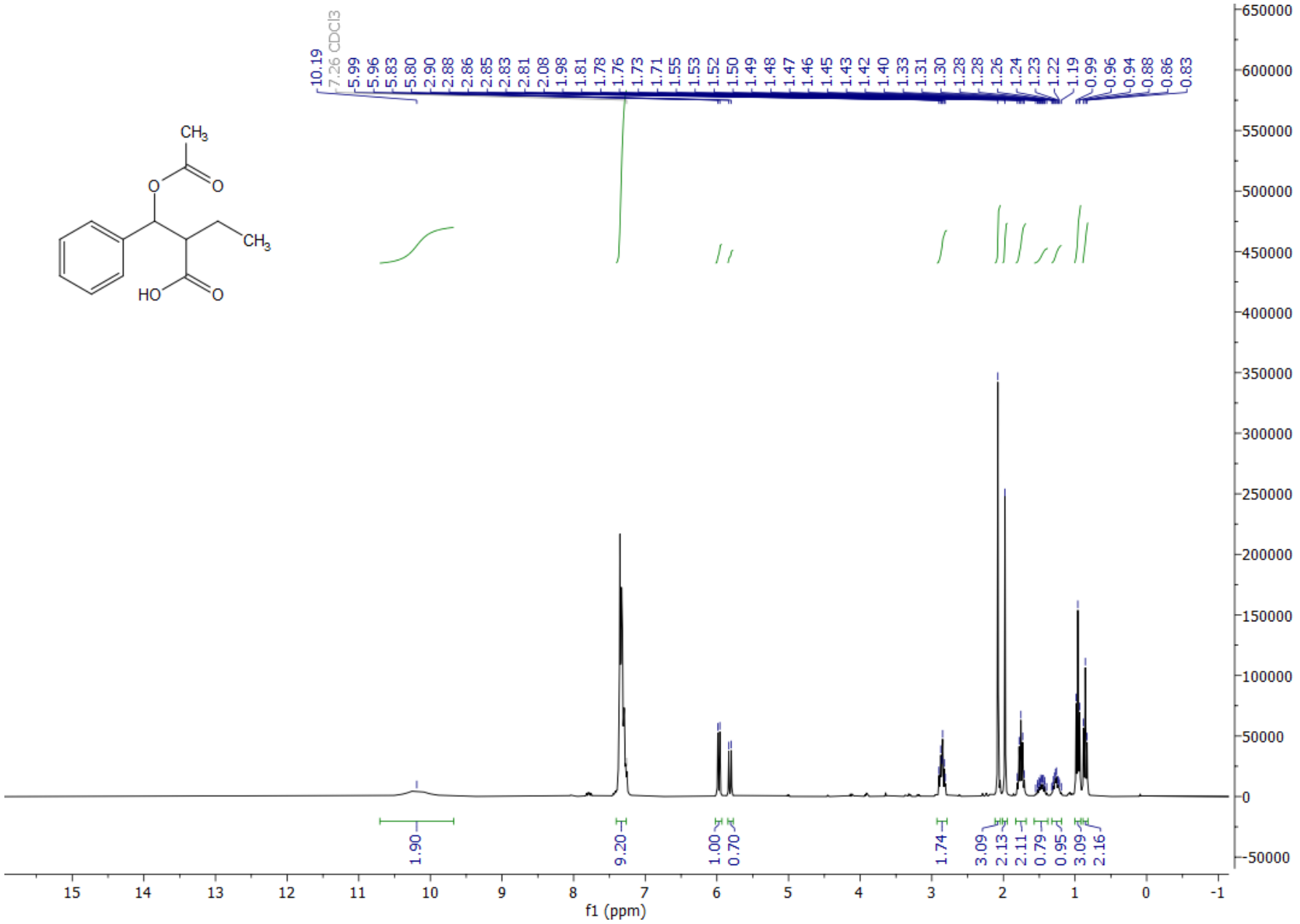
^{1}H NMR (300.13 MHz, $CDCl_3$), **3-Acetoxy-2-methyl-3-phenylpropanoic acid, 2l**



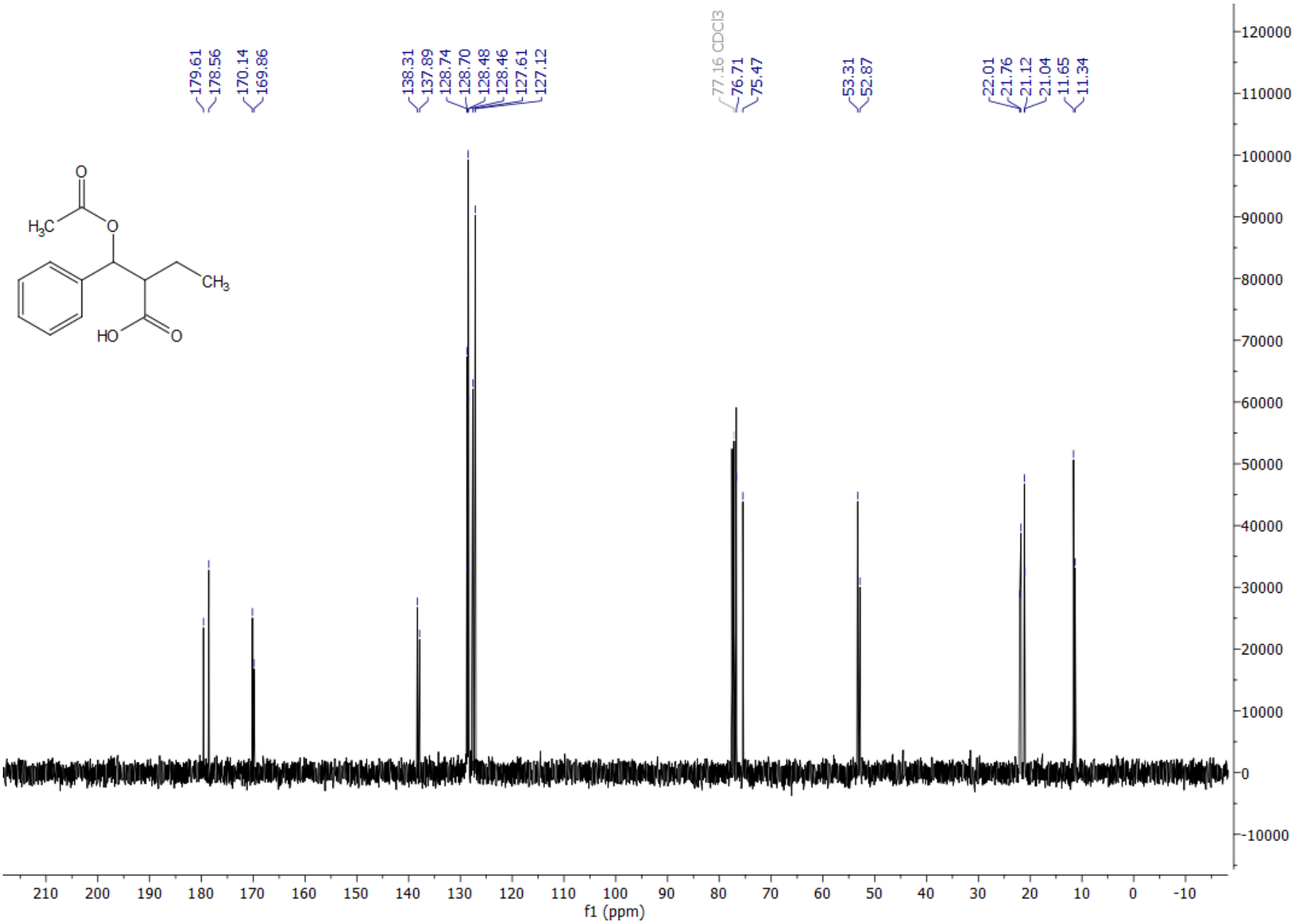
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **3-Acetoxy-2-methyl-3-phenylpropanoic acid, 2l**



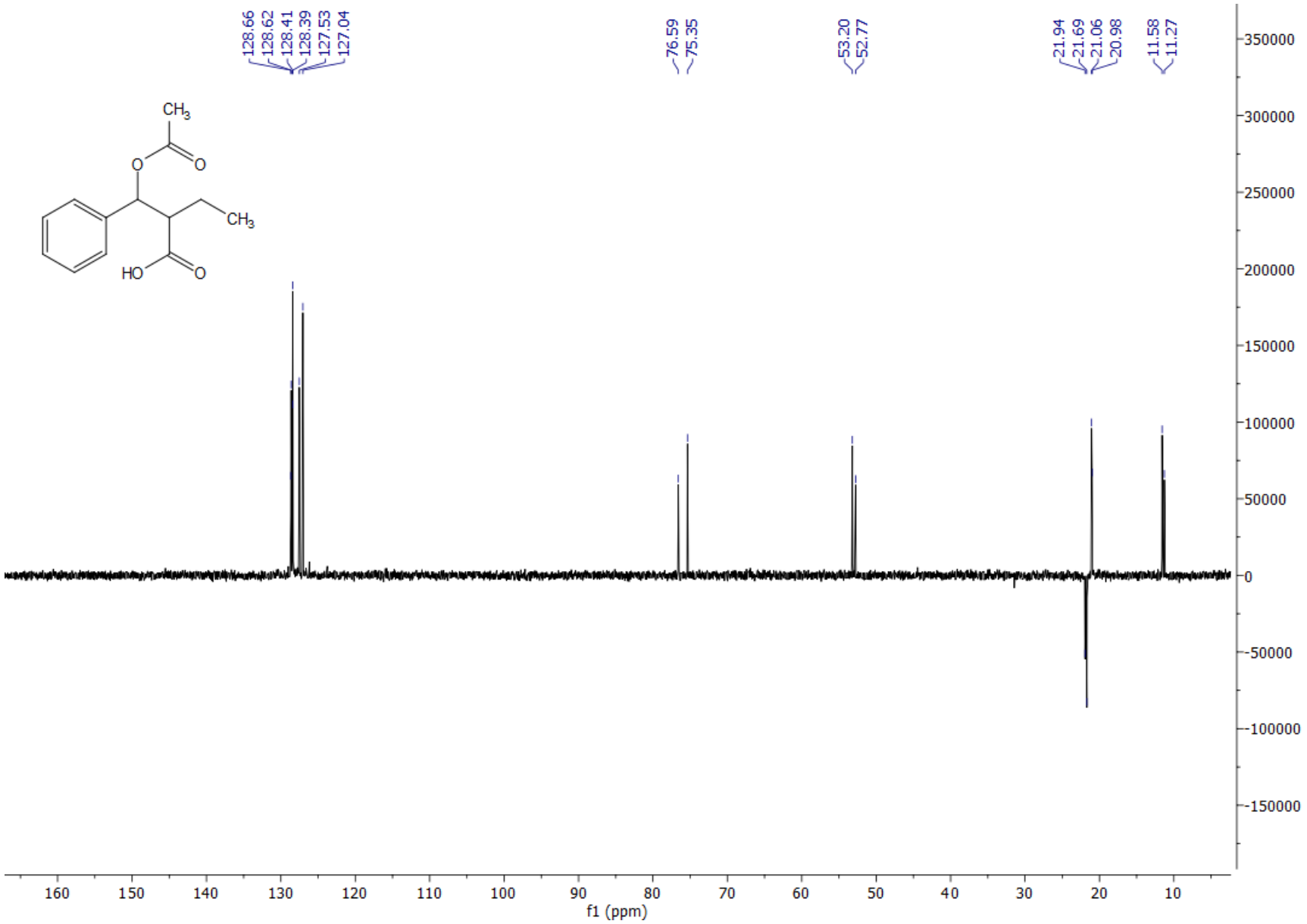
^{1}H NMR (300.13 MHz, $CDCl_3$),**2-(Acetoxy(phenyl)methyl)butanoic acid, 2m**



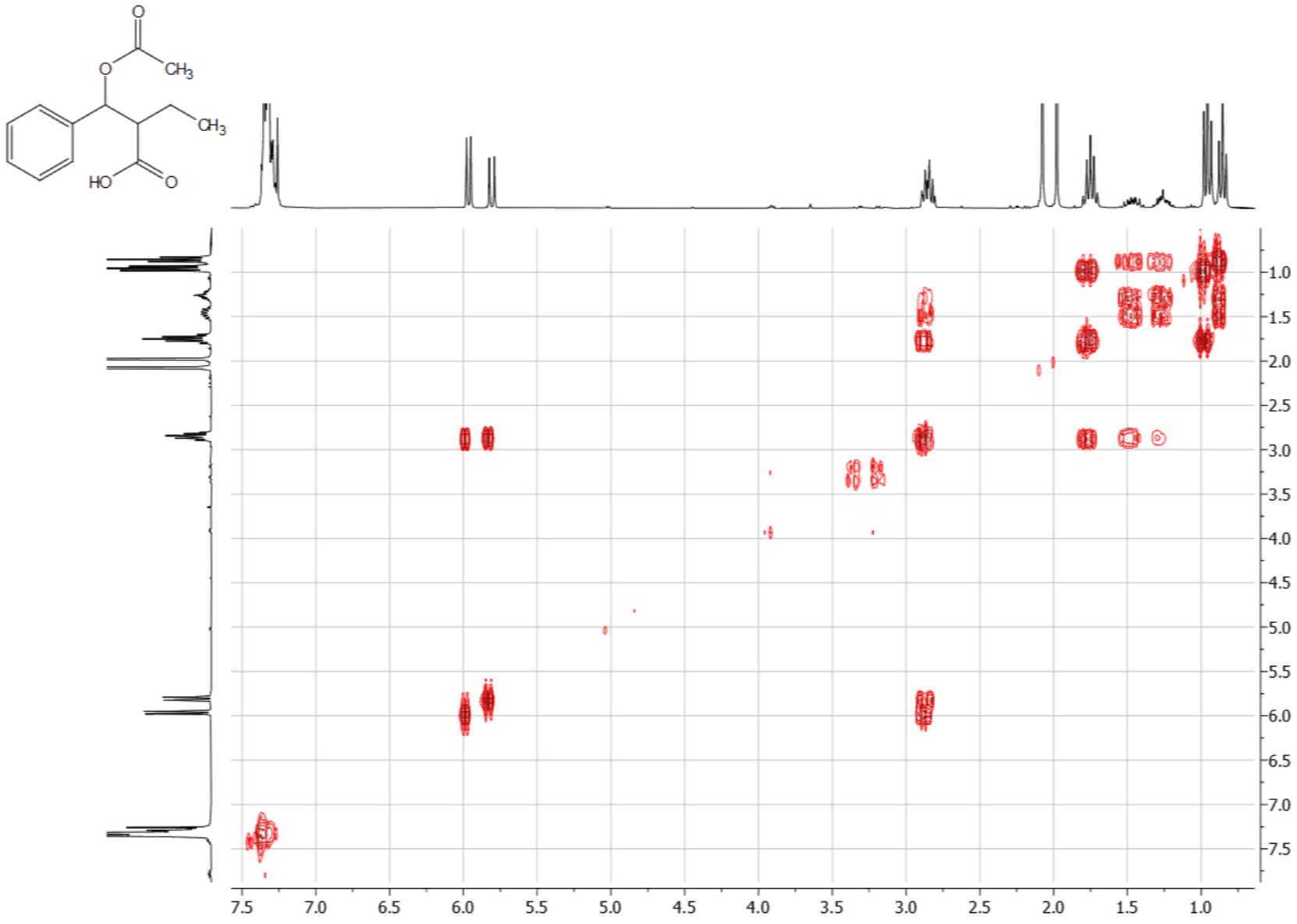
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **2-(Acetoxy(phenyl)methyl)butanoic acid, 2m**



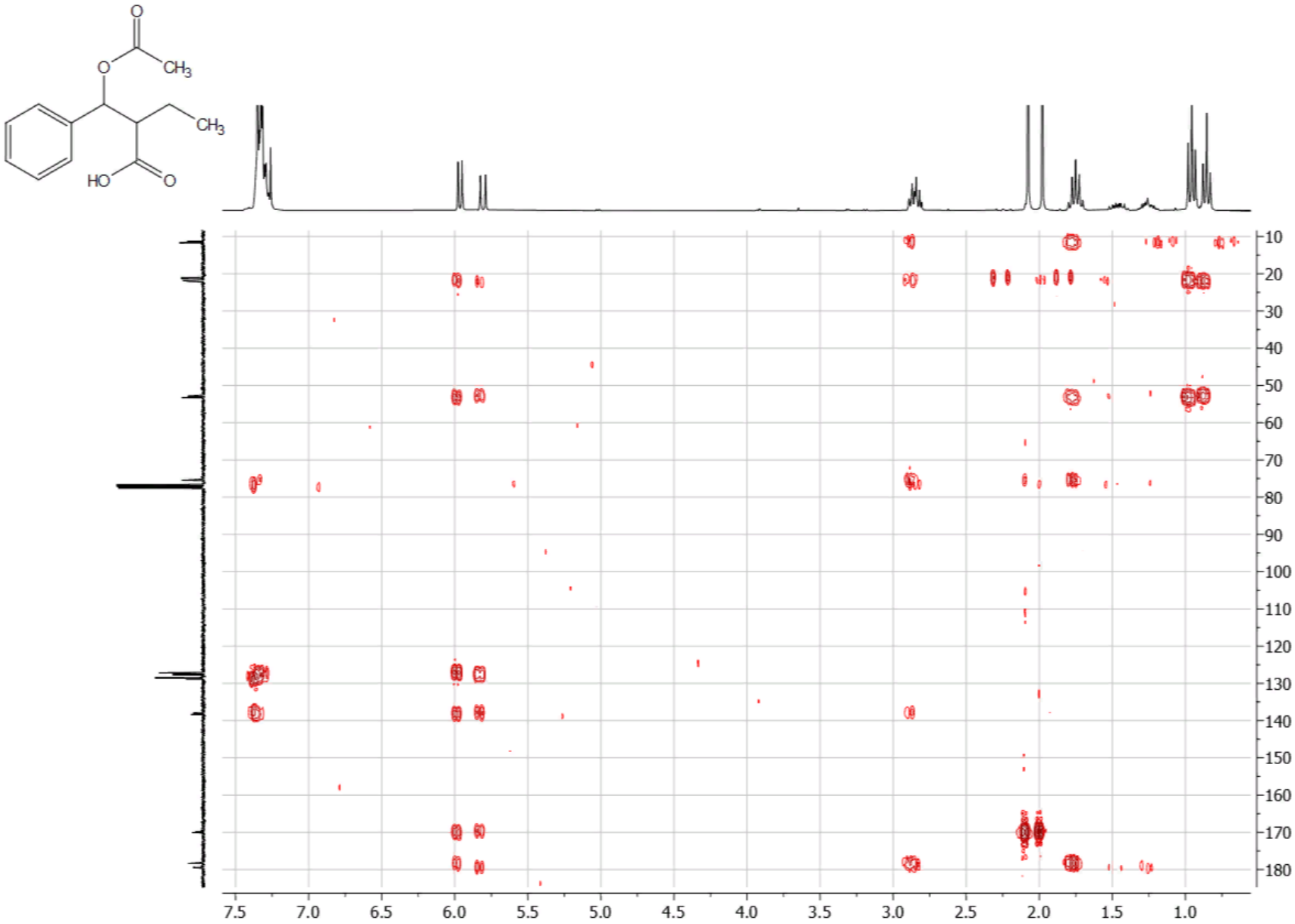
^{13}C DEPT135, **2-(Acetoxy(phenyl)methyl)butanoic acid, 2m**



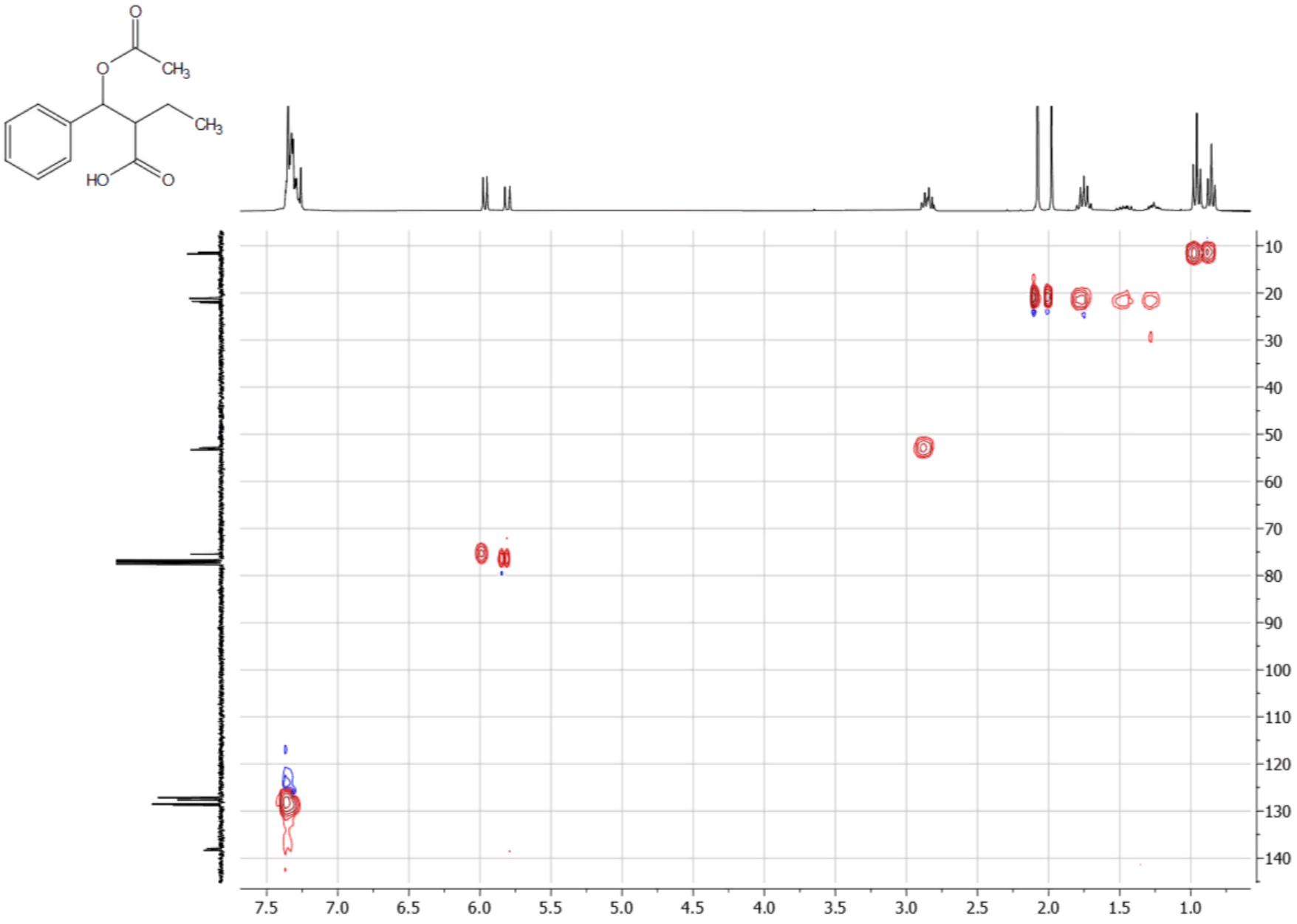
{^{1}H - ^{1}H} COSY of **2-(Acetoxy(phenyl)methyl)butanoic acid, 2m**



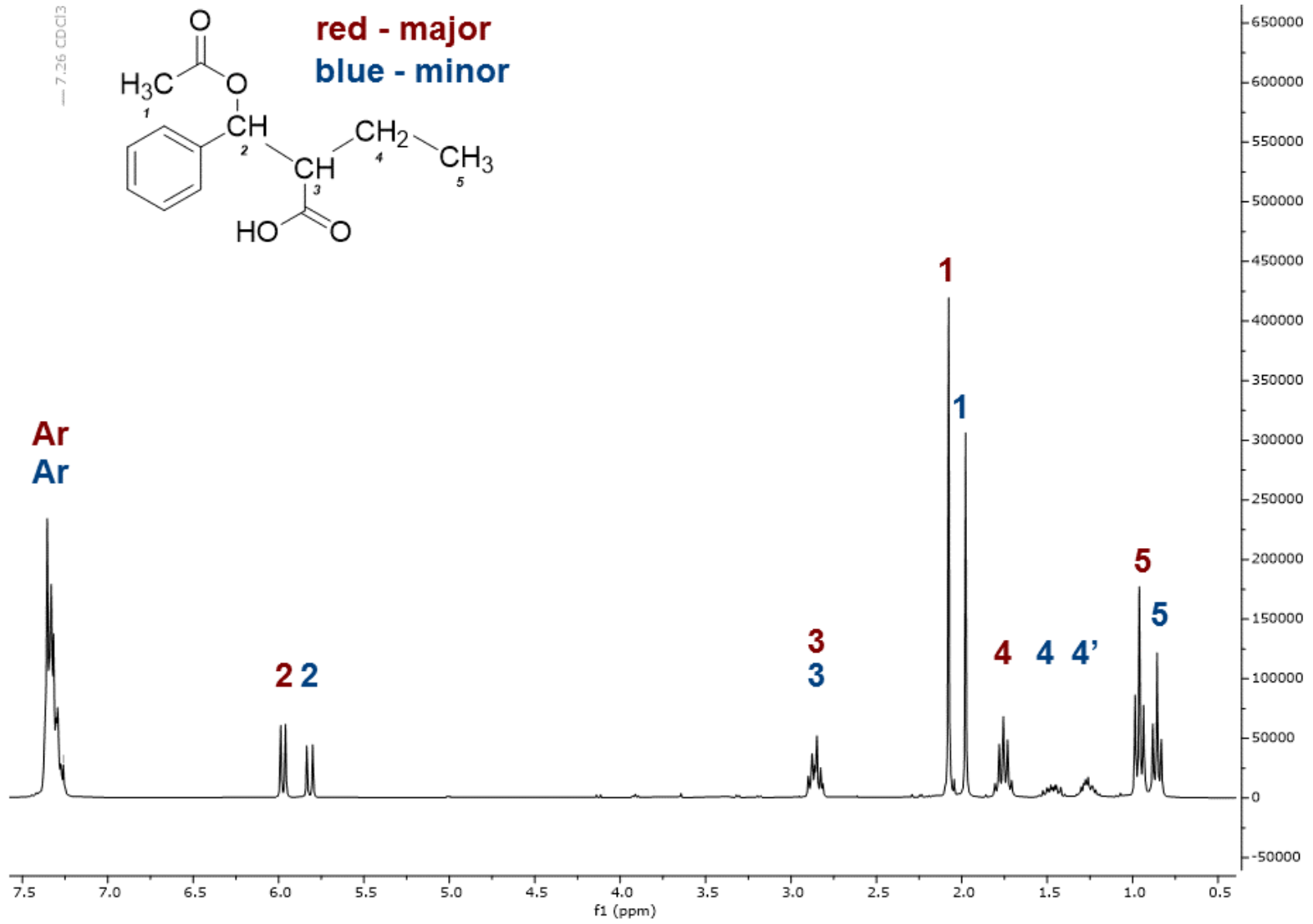
{^{1}H - ^{13}C} HMBC of **2-(Acetoxy(phenyl)methyl)butanoic acid, 2m**



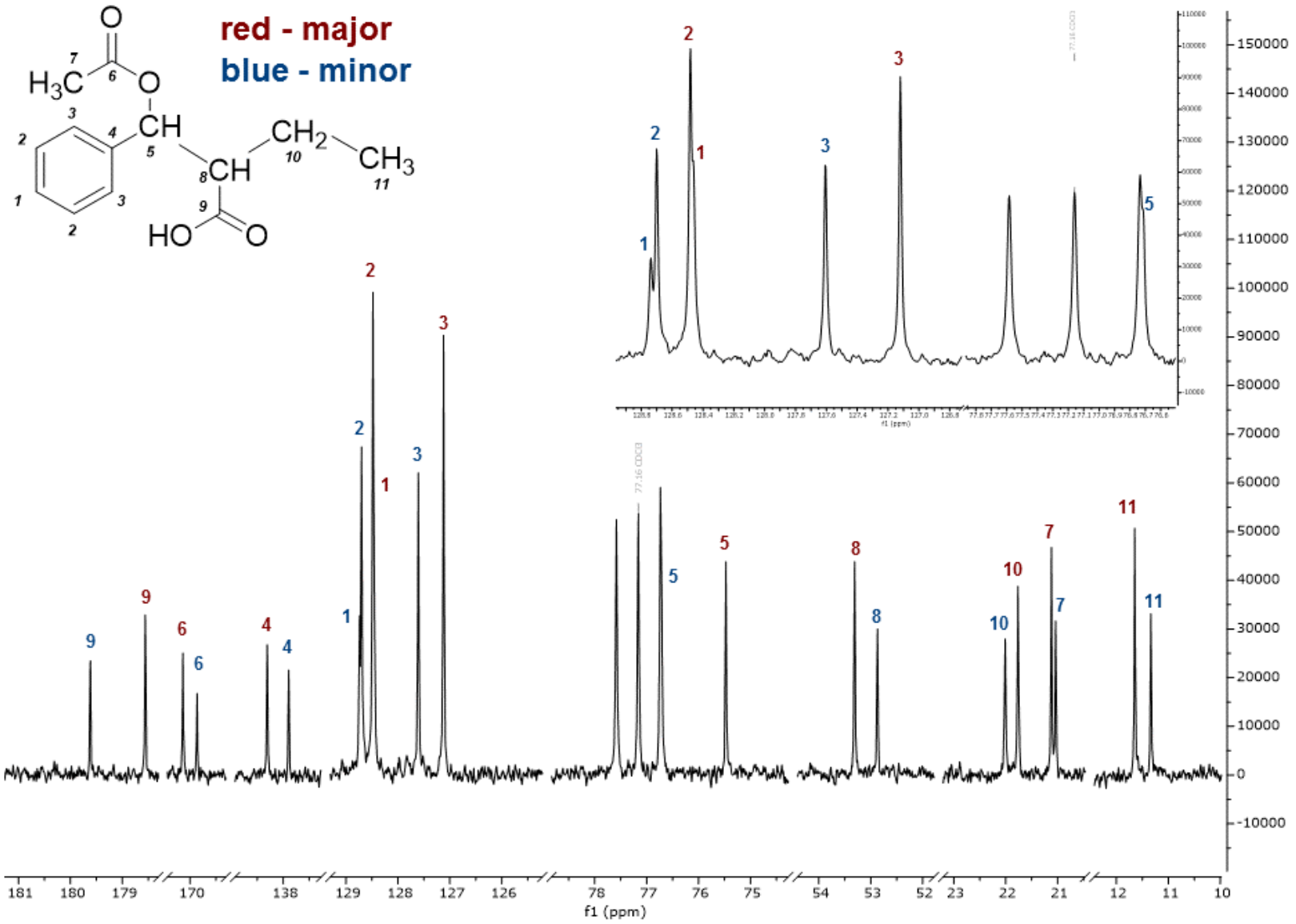
{^{1}H - ^{13}C} HSQC of **2-(Acetoxy(phenyl)methyl)butanoic acid, 2m**



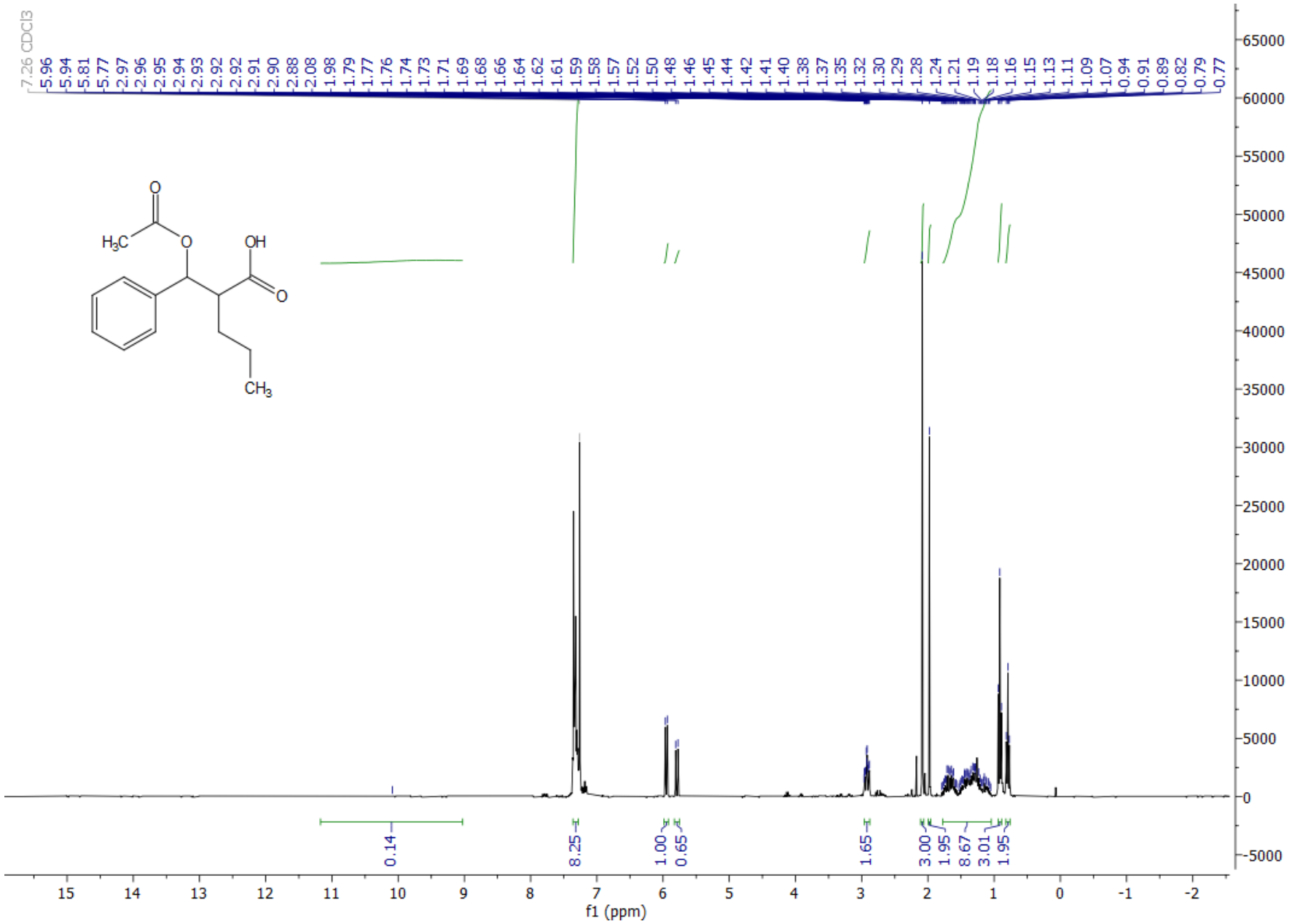
Correlation ^{1}H NMR (300.13 MHz, $CDCl_3$), **2-(Acetoxy(phenyl)methyl)butanoic acid, 2m**



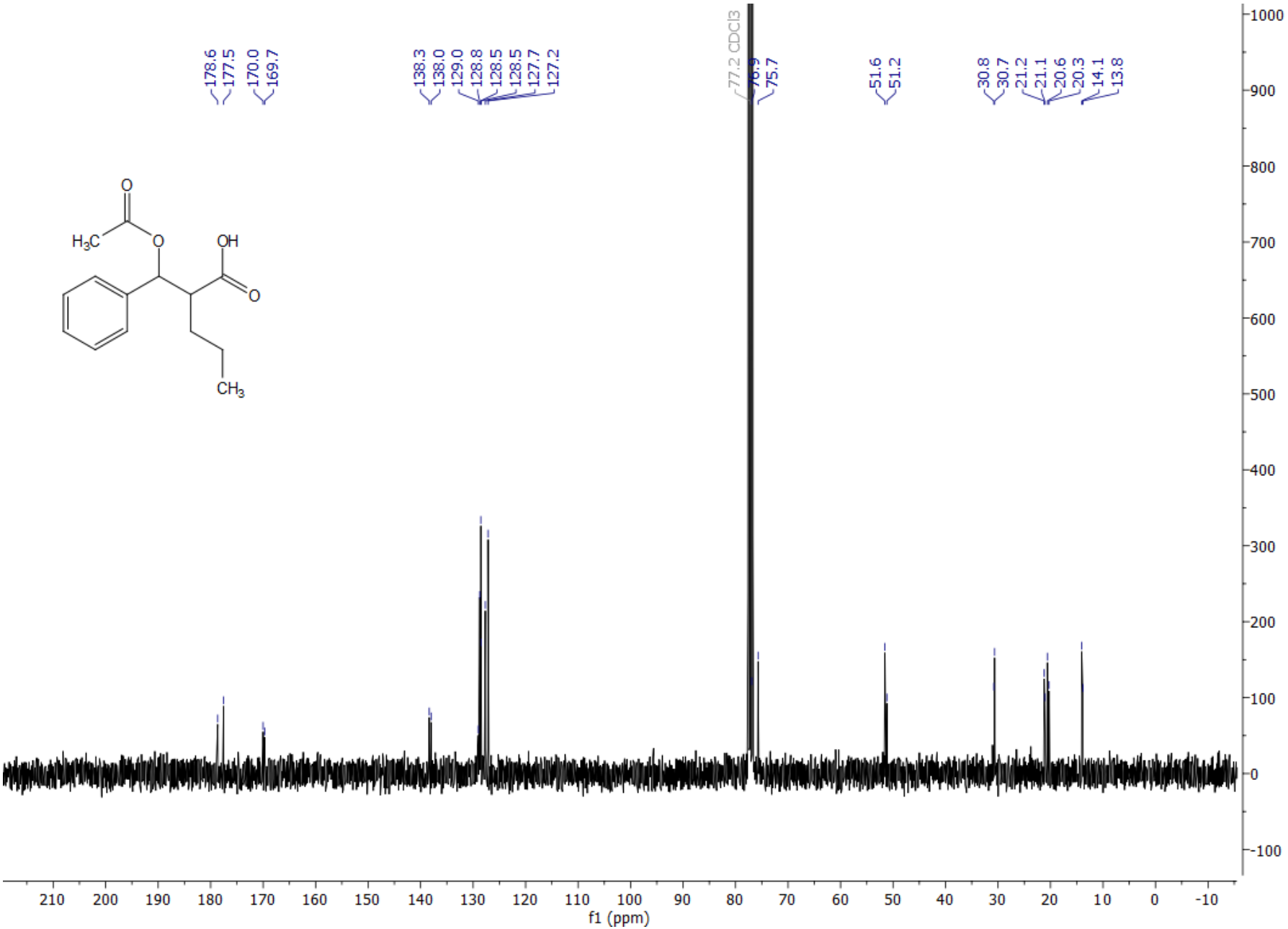
Correlation $^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **2-(Acetoxy(phenyl)methyl)butanoic acid, 2m**



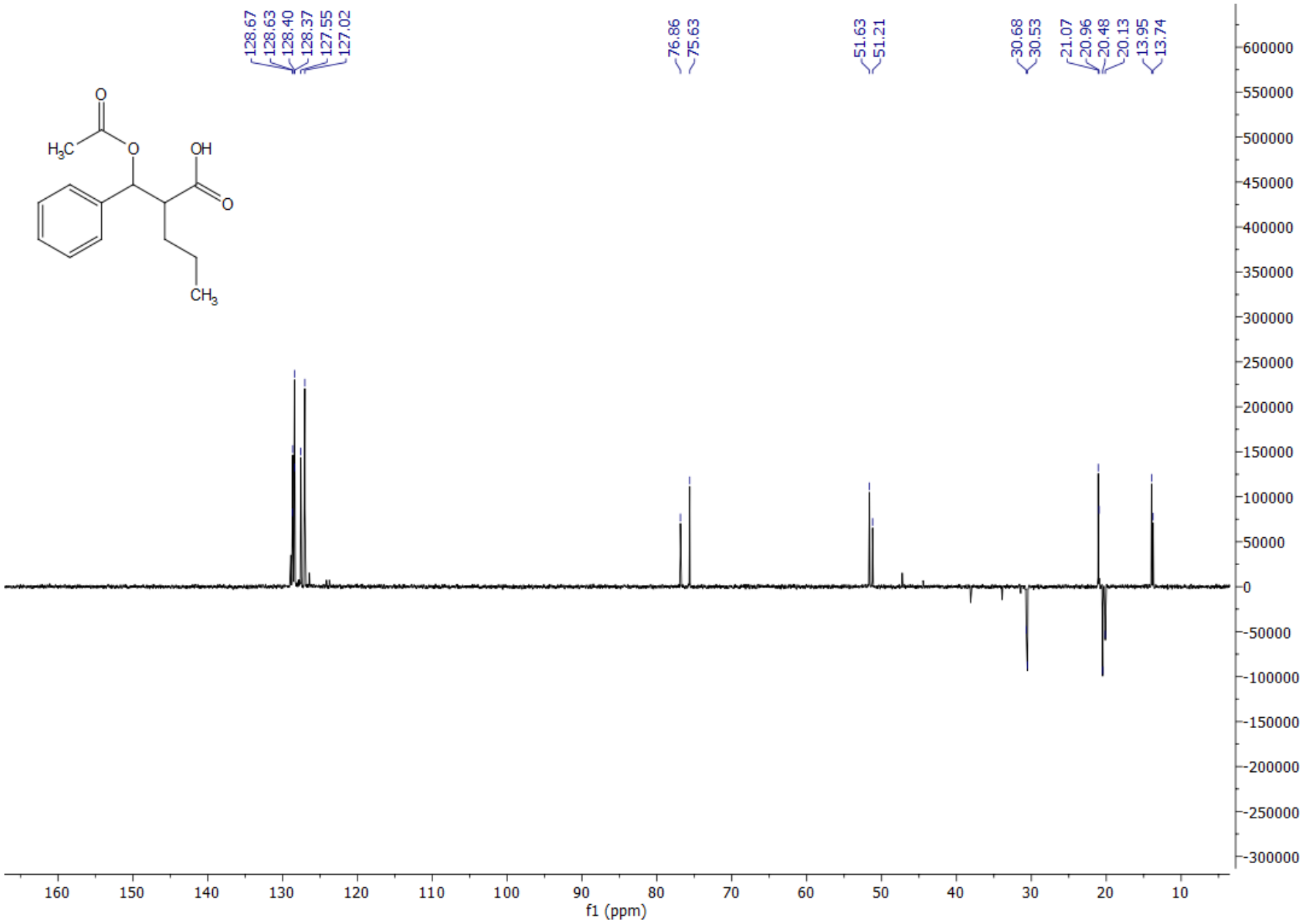
^{1}H NMR (300.13 MHz, $CDCl_3$), **2-(Acetoxy(phenyl)methyl)pentanoic acid, 2n**



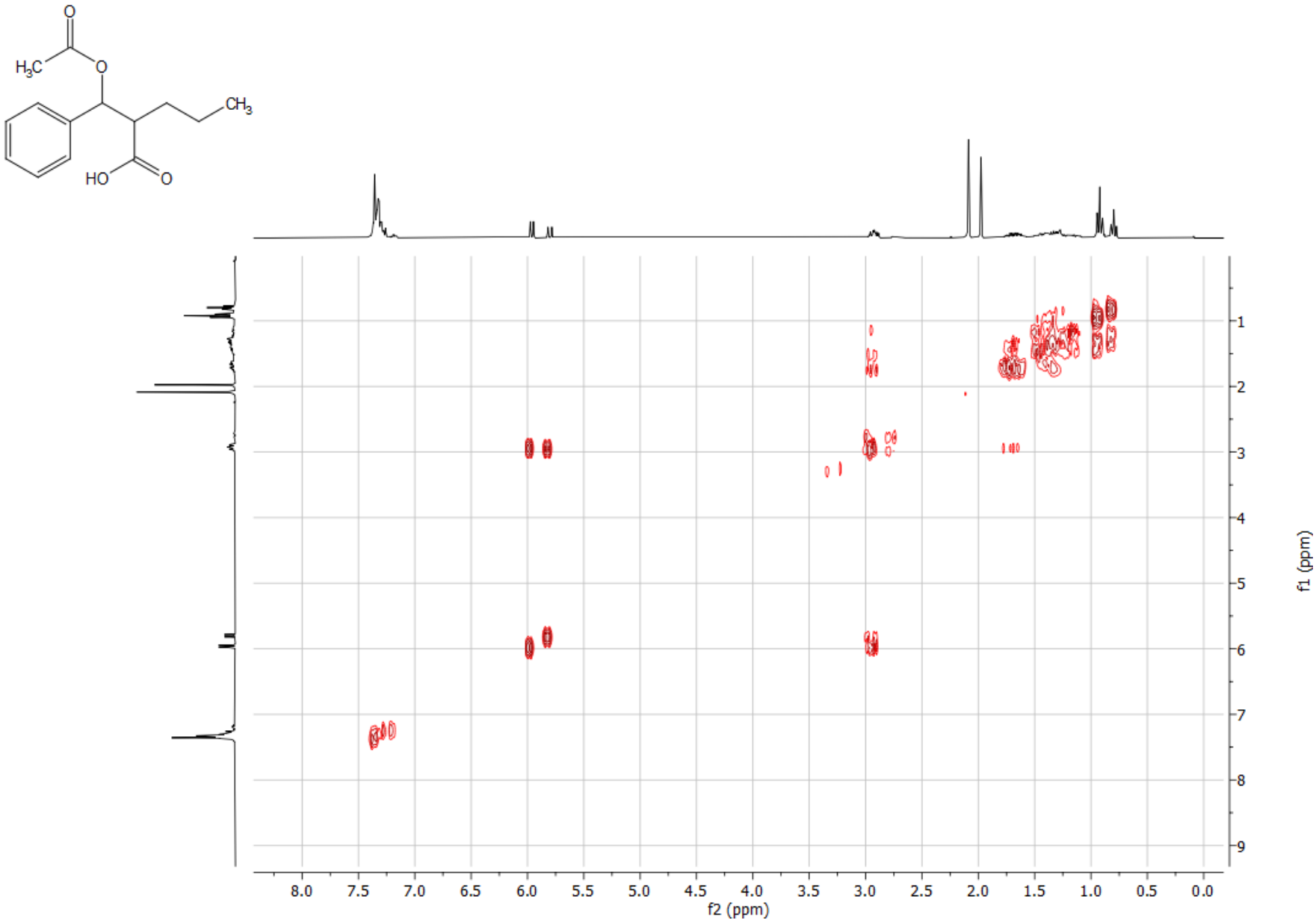
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$),**2-(Acetoxy(phenyl)methyl)pentanoic acid, 2n**



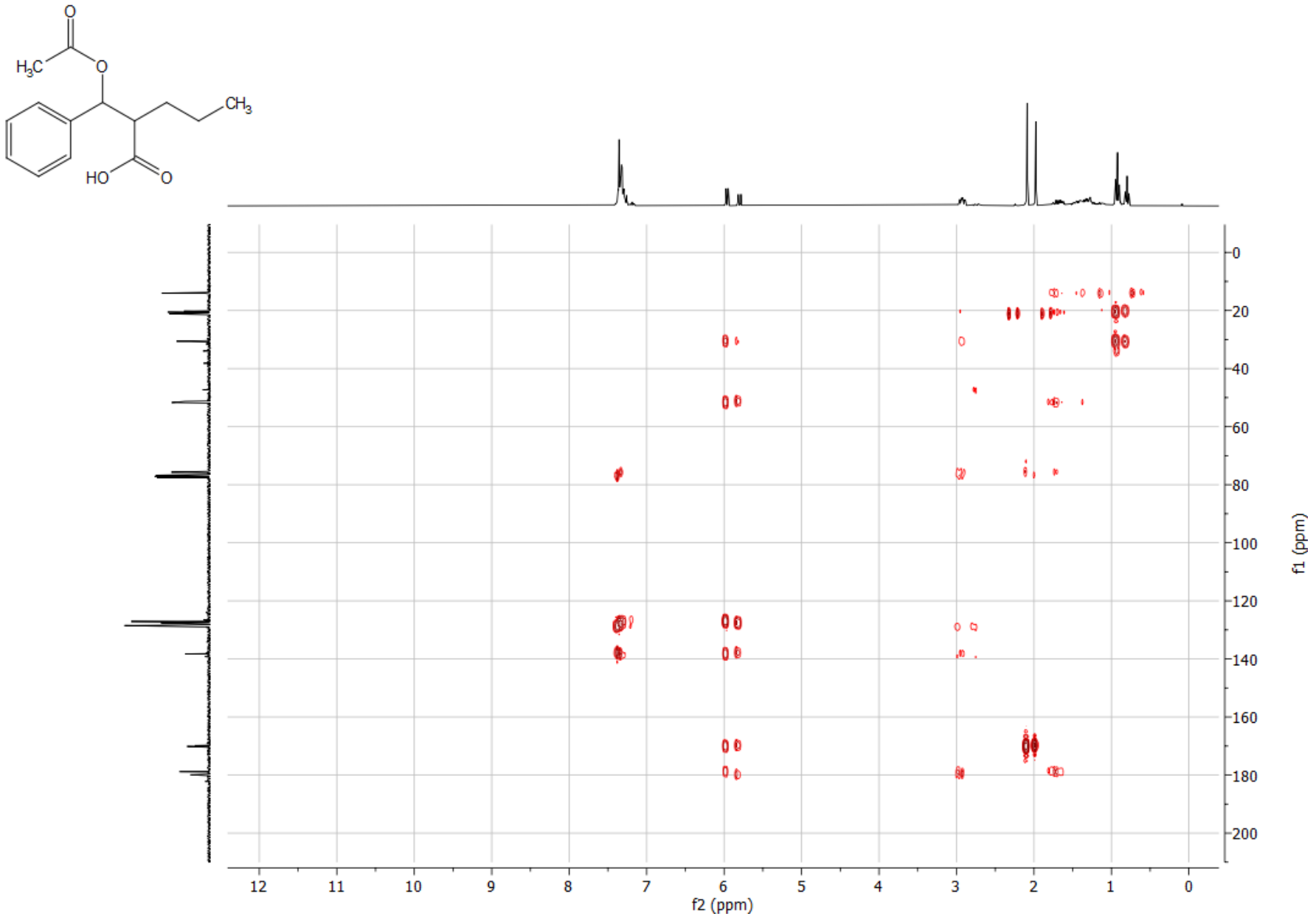
^{13}C DEPT135, **2-(Acetoxy(phenyl)methyl)pentanoic acid, 2n**



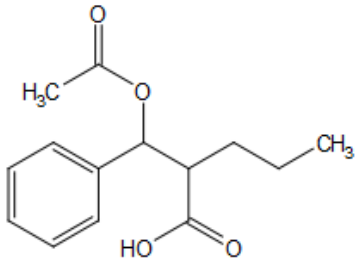
{^{1}H - ^{1}H} COSY of **2-(Acetoxy(phenyl)methyl)pentanoic acid, 2n**

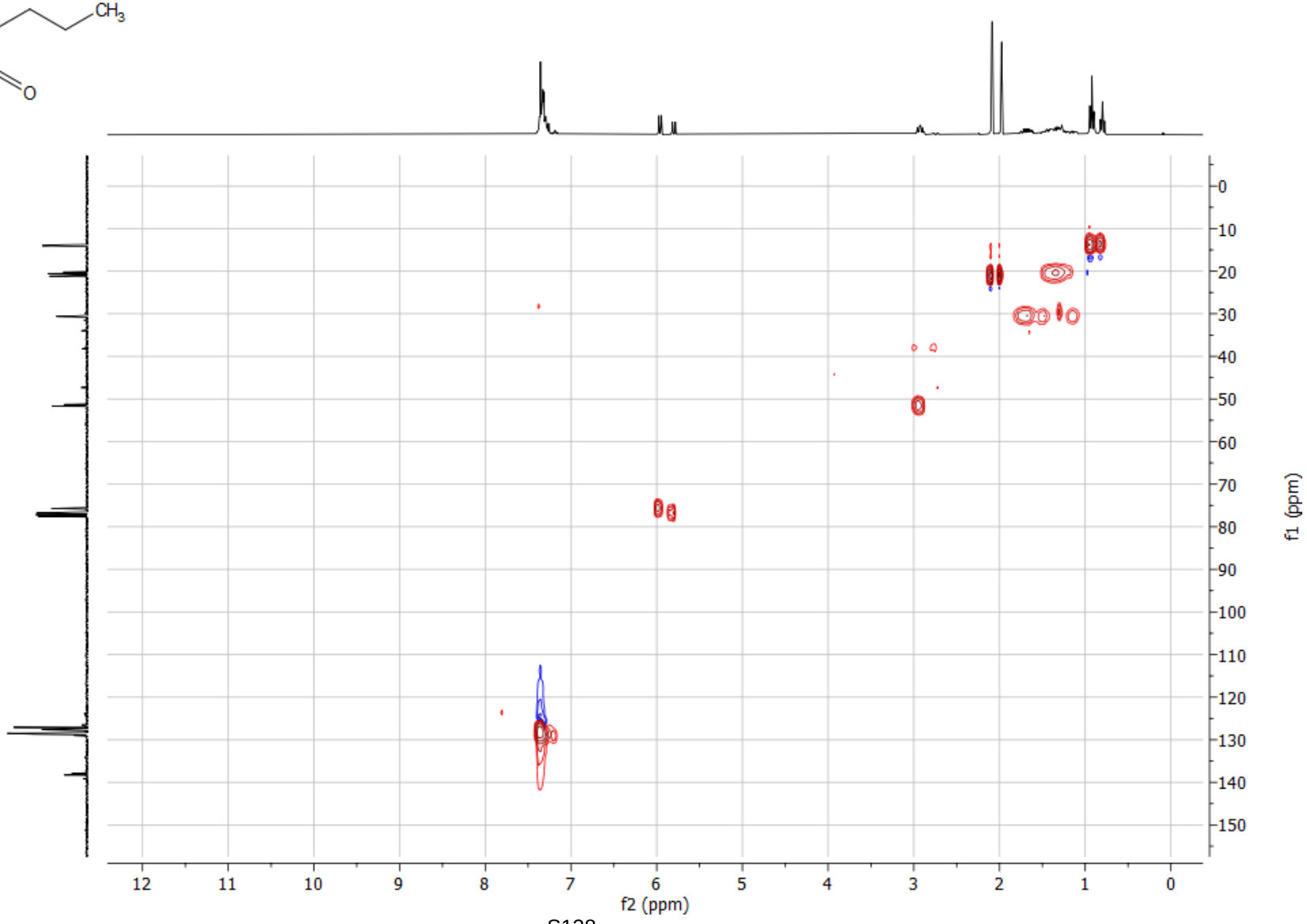


{^{1}H - ^{13}C} HMBC of **2-(Acetoxy(phenyl)methyl)pentanoic acid, 2n**

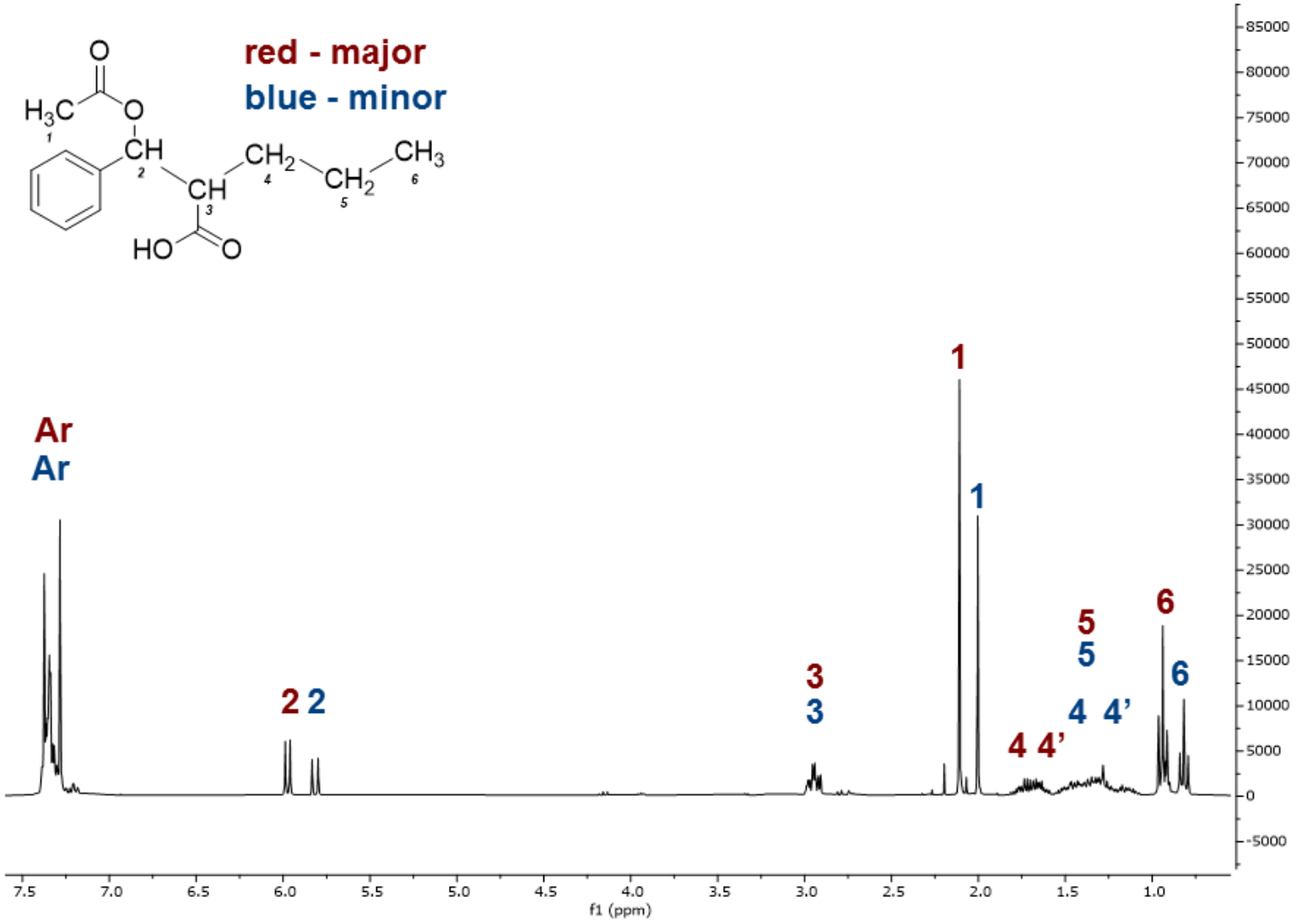


{^{1}H - ^{13}C} HSQC of **2-(Acetoxy(phenyl)methyl)pentanoic acid, 2n**

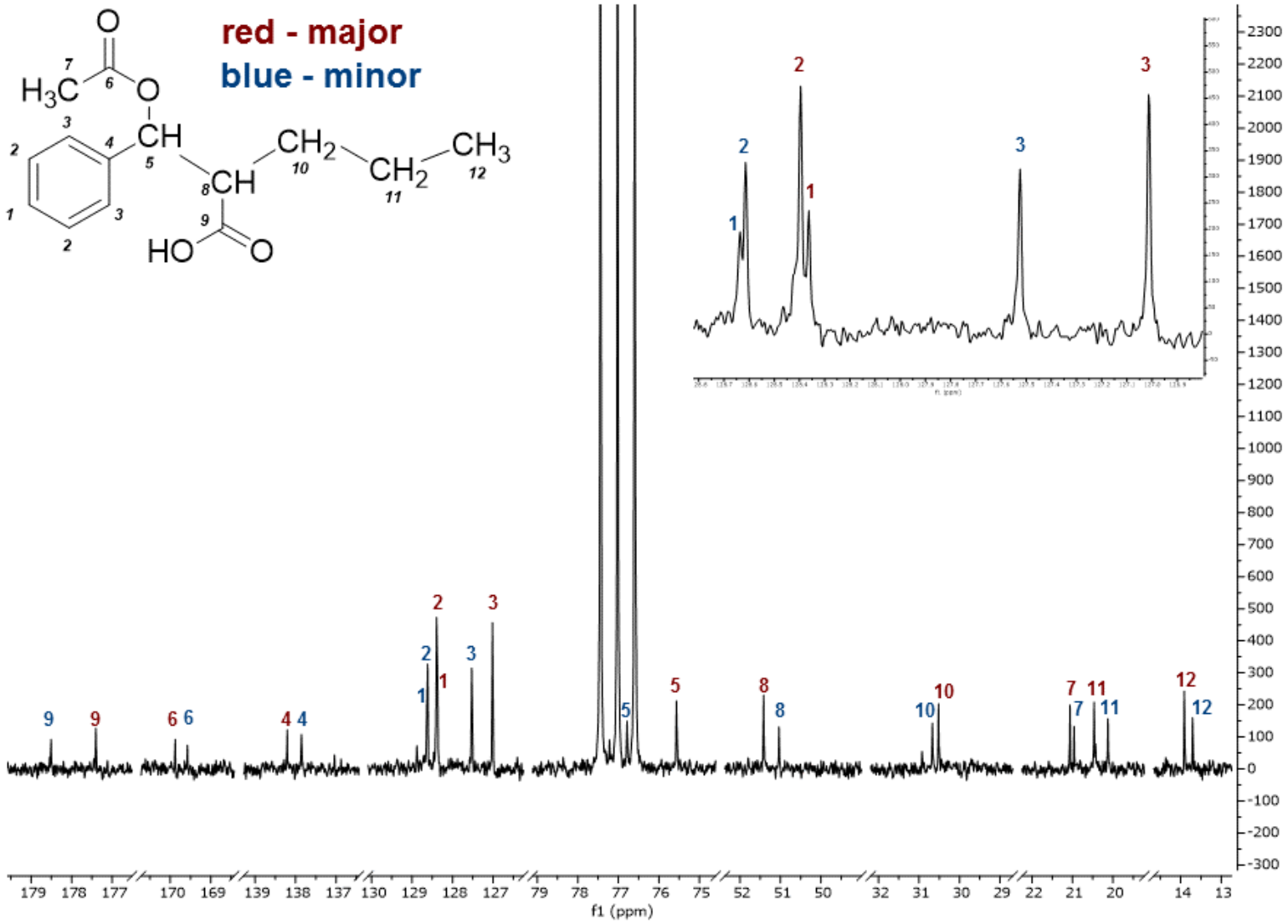




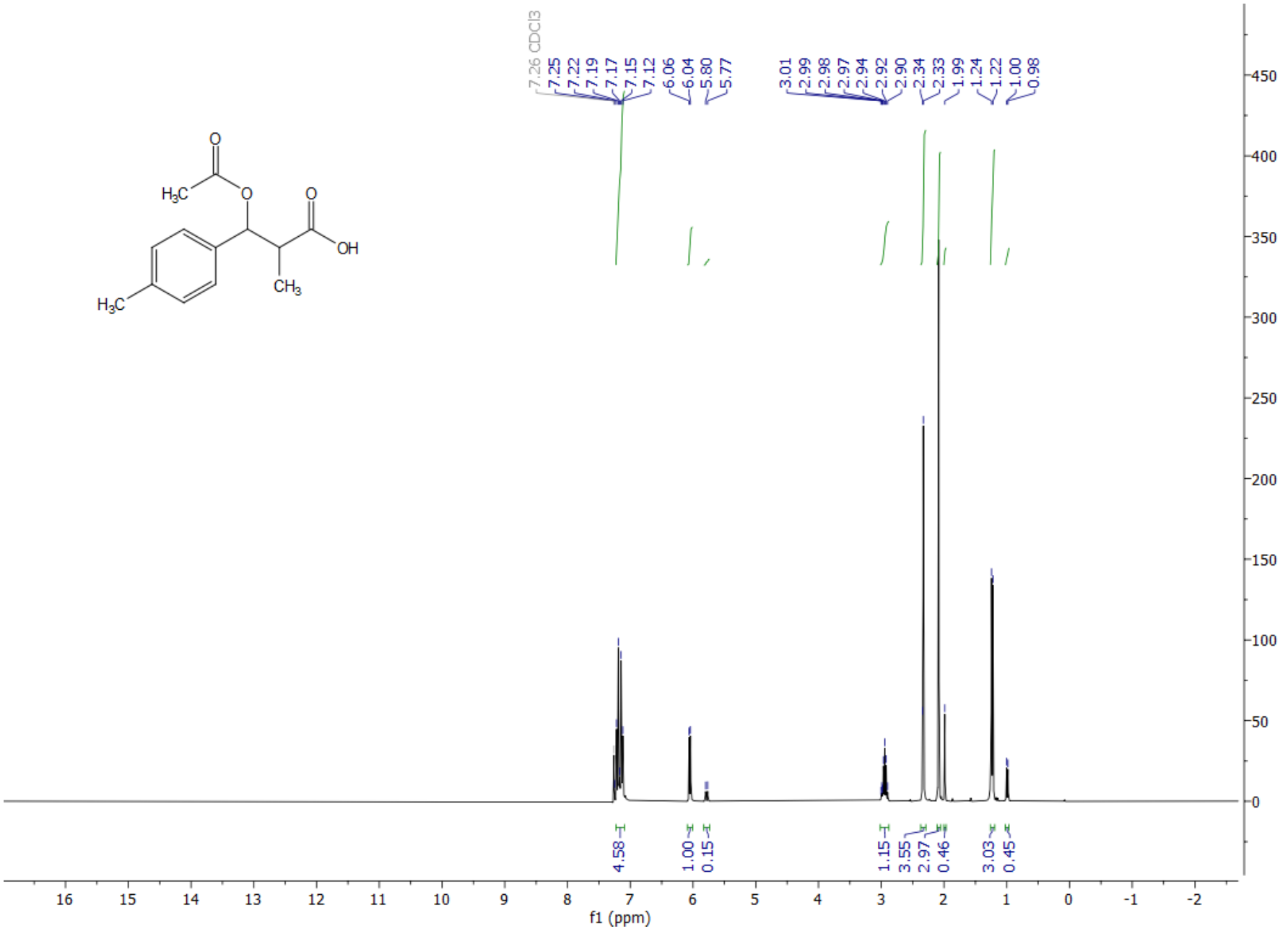
Correlation ^{1}H NMR (300.13 MHz, $CDCl_3$), **2-(Acetoxy(phenyl)methyl)pentanoic acid, 2n**



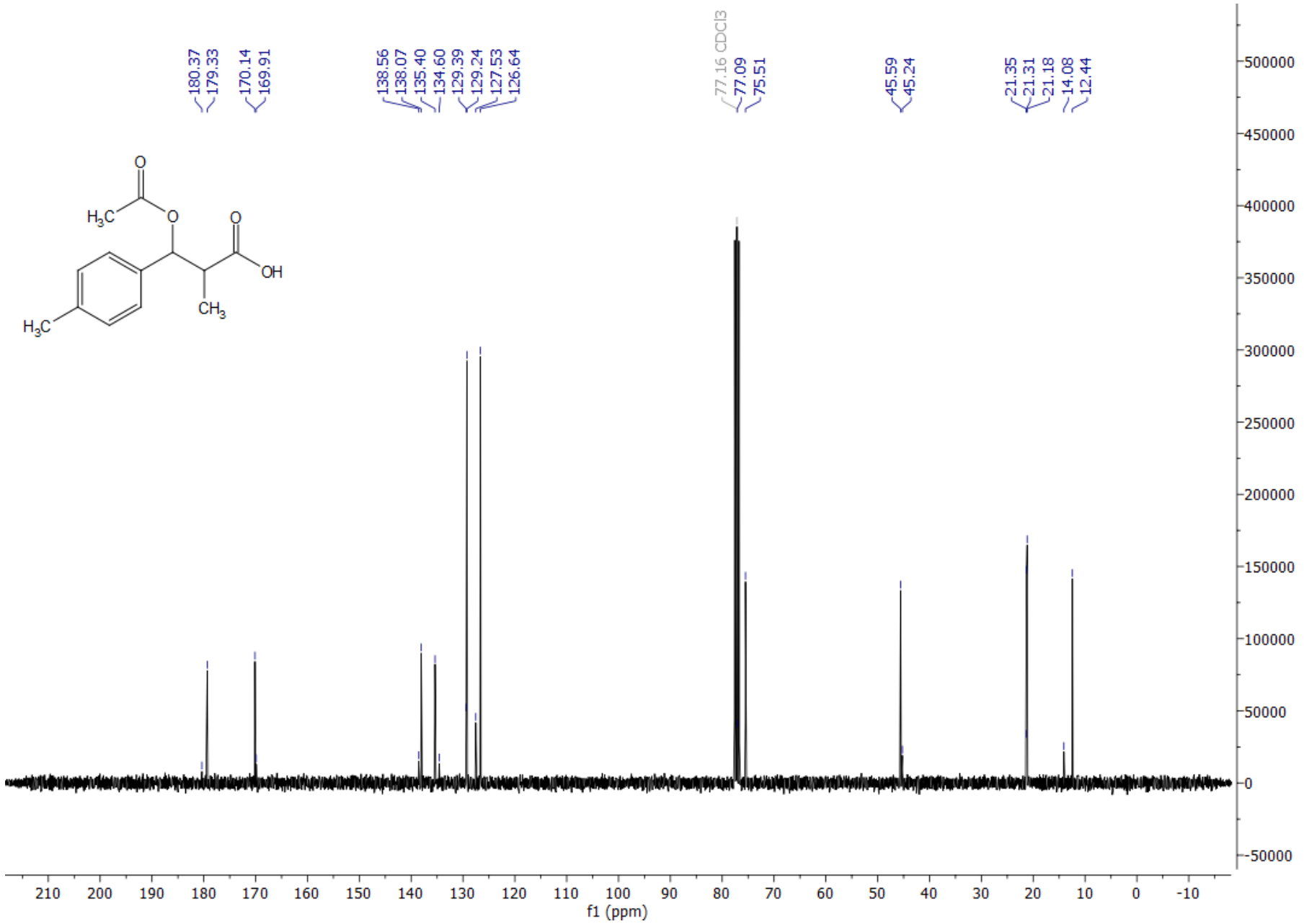
Correlation $^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **2-(Acetoxy(phenyl)methyl)pentanoic acid, 2n**



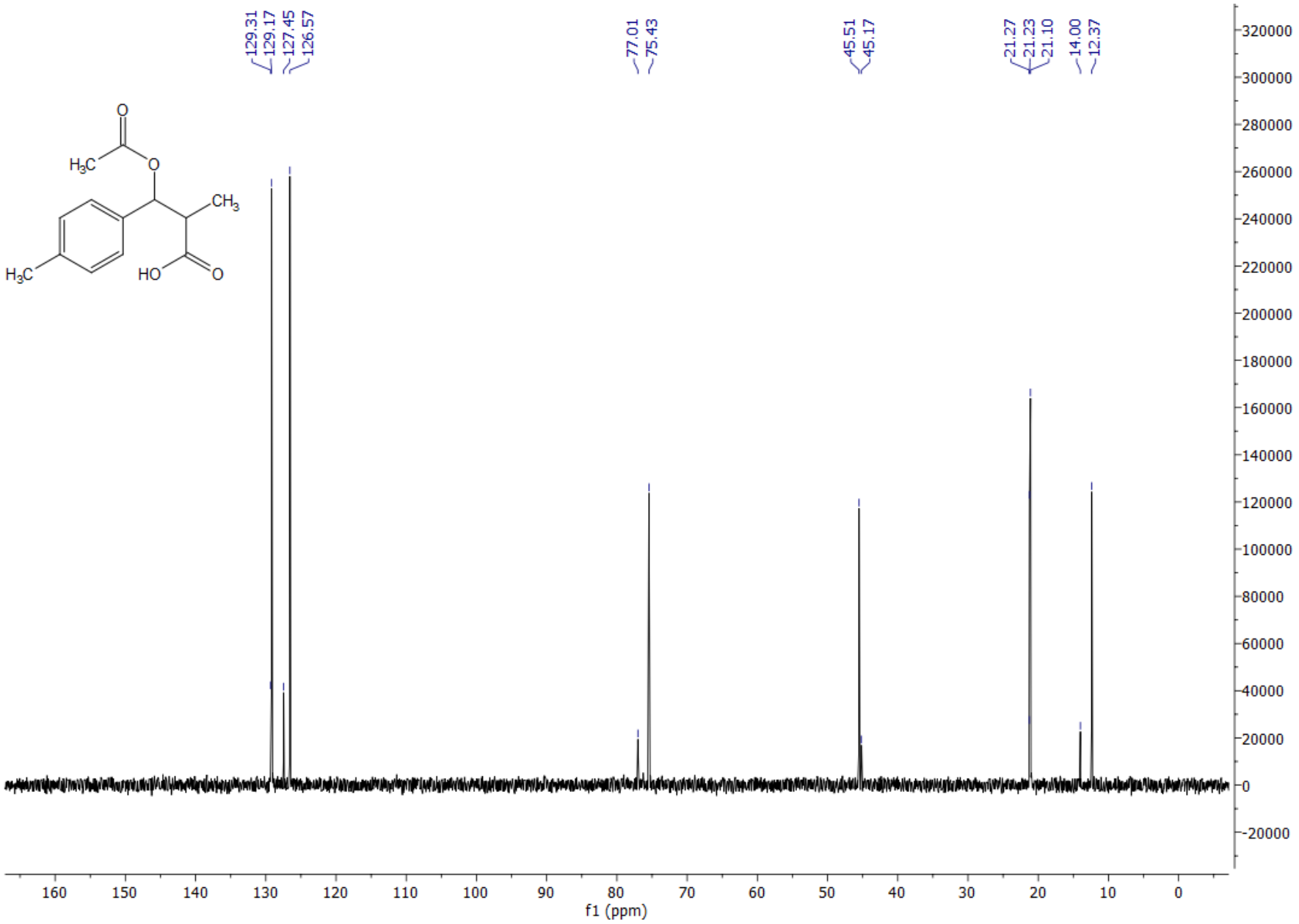
^{1}H NMR (300.13 MHz, $CDCl_3$), **3-Acetoxy-2-methyl-3-(*p*-tolyl)propanoic acid, 2o**



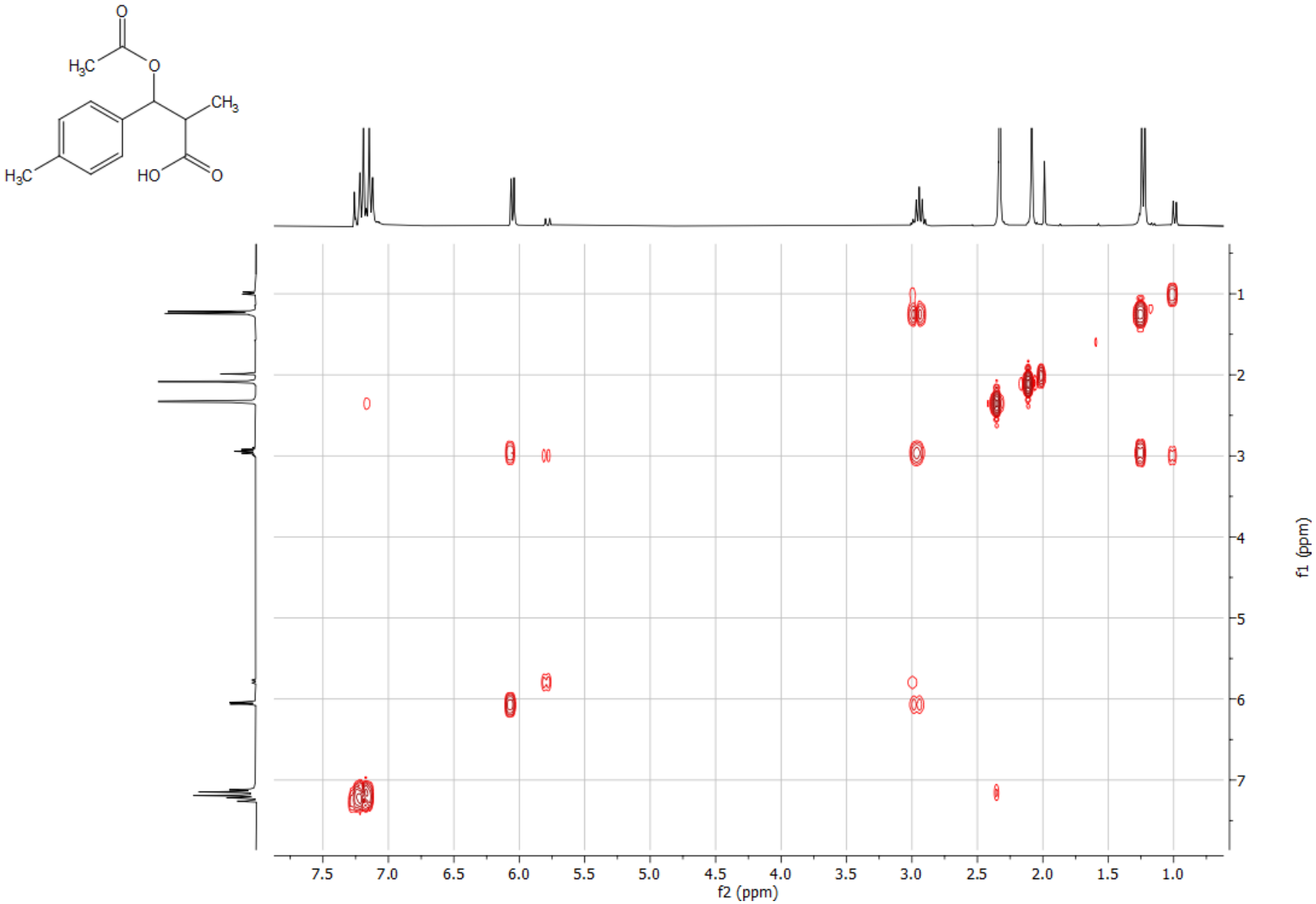
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **3-Acetoxy-2-methyl-3-(*p*-tolyl)propanoic acid, 2o**



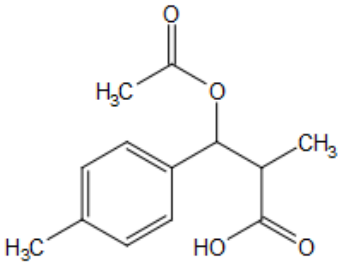
^{13}C DEPT135, **3-Acetoxy-2-methyl-3-(*p*-tolyl)propanoic acid, 2o**

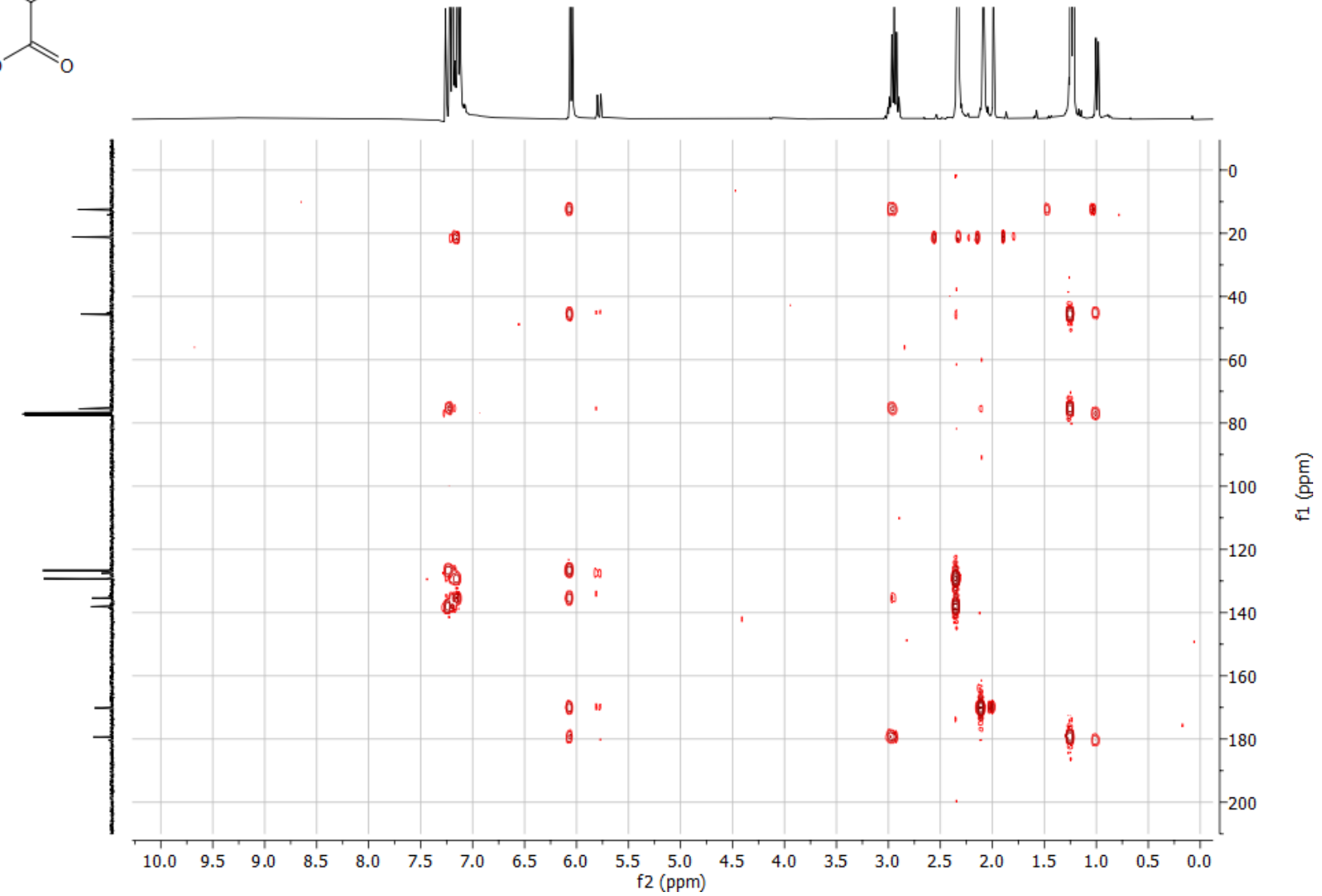


{^{1}H - ^{1}H} COSY of **3-Acetoxy-2-methyl-3-(*p*-tolyl)propanoic acid, 2o**

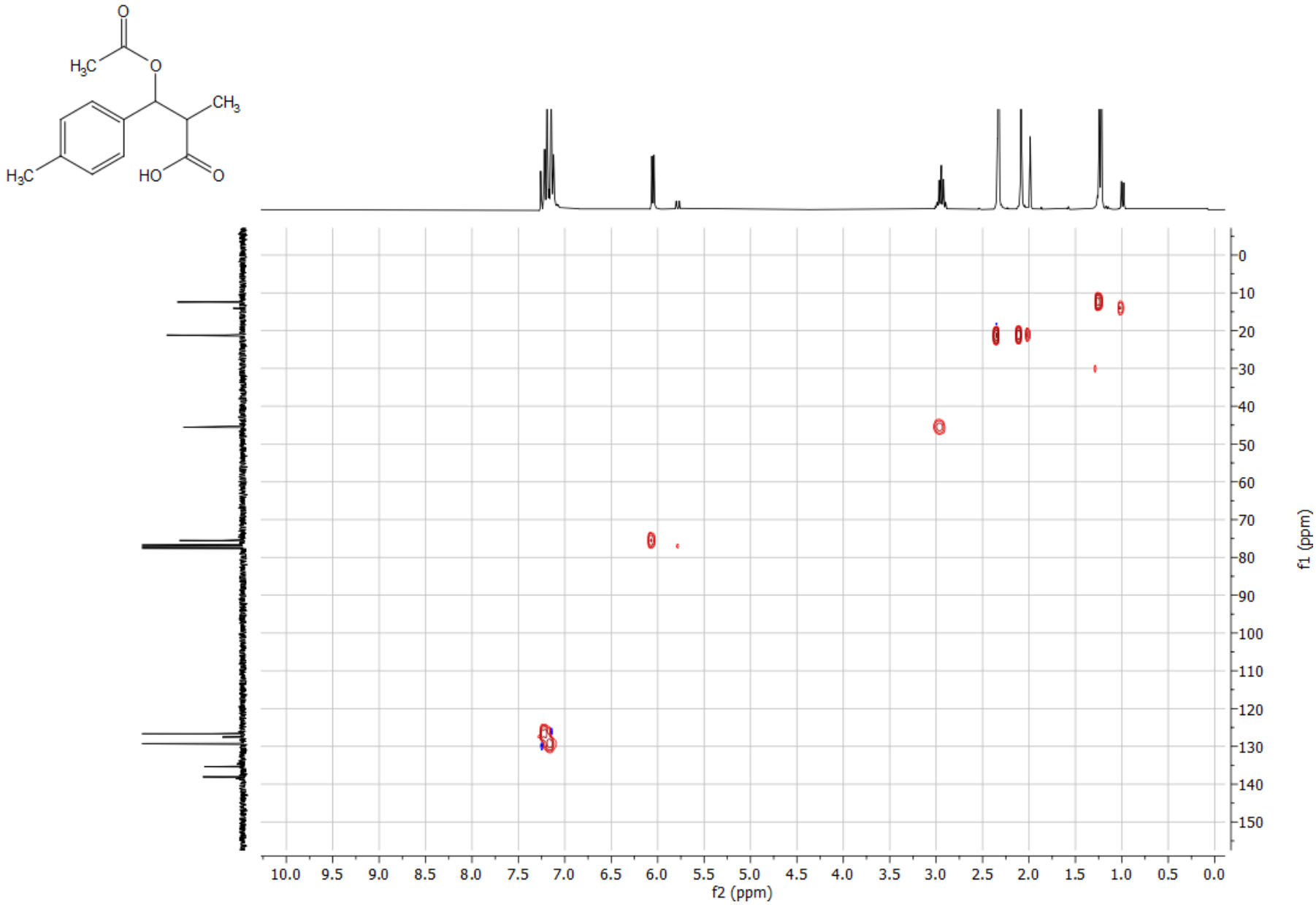


{^{1}H - ^{13}C} HMBC of **3-Acetoxy-2-methyl-3-(*p*-tolyl)propanoic acid, 2o**

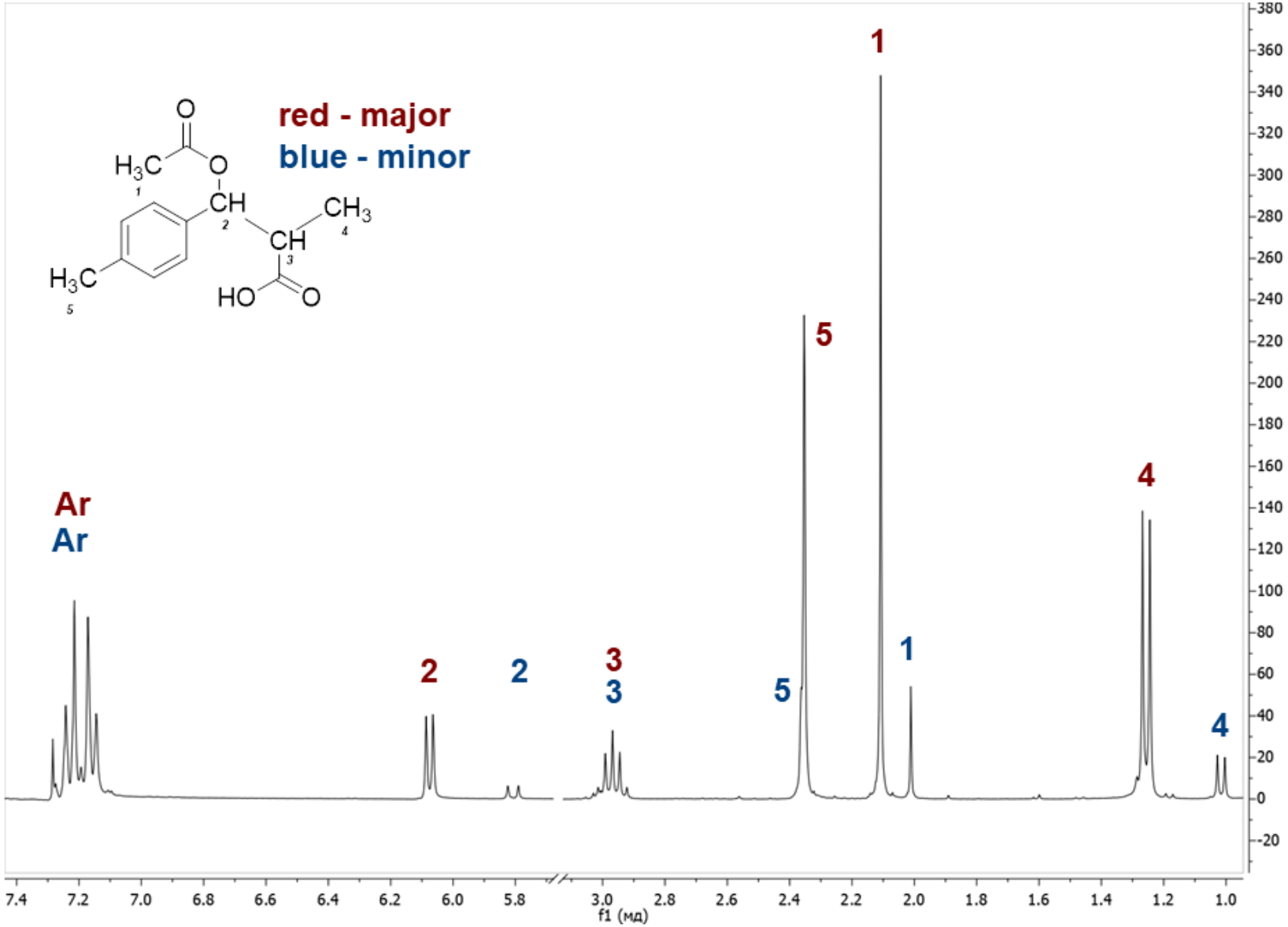




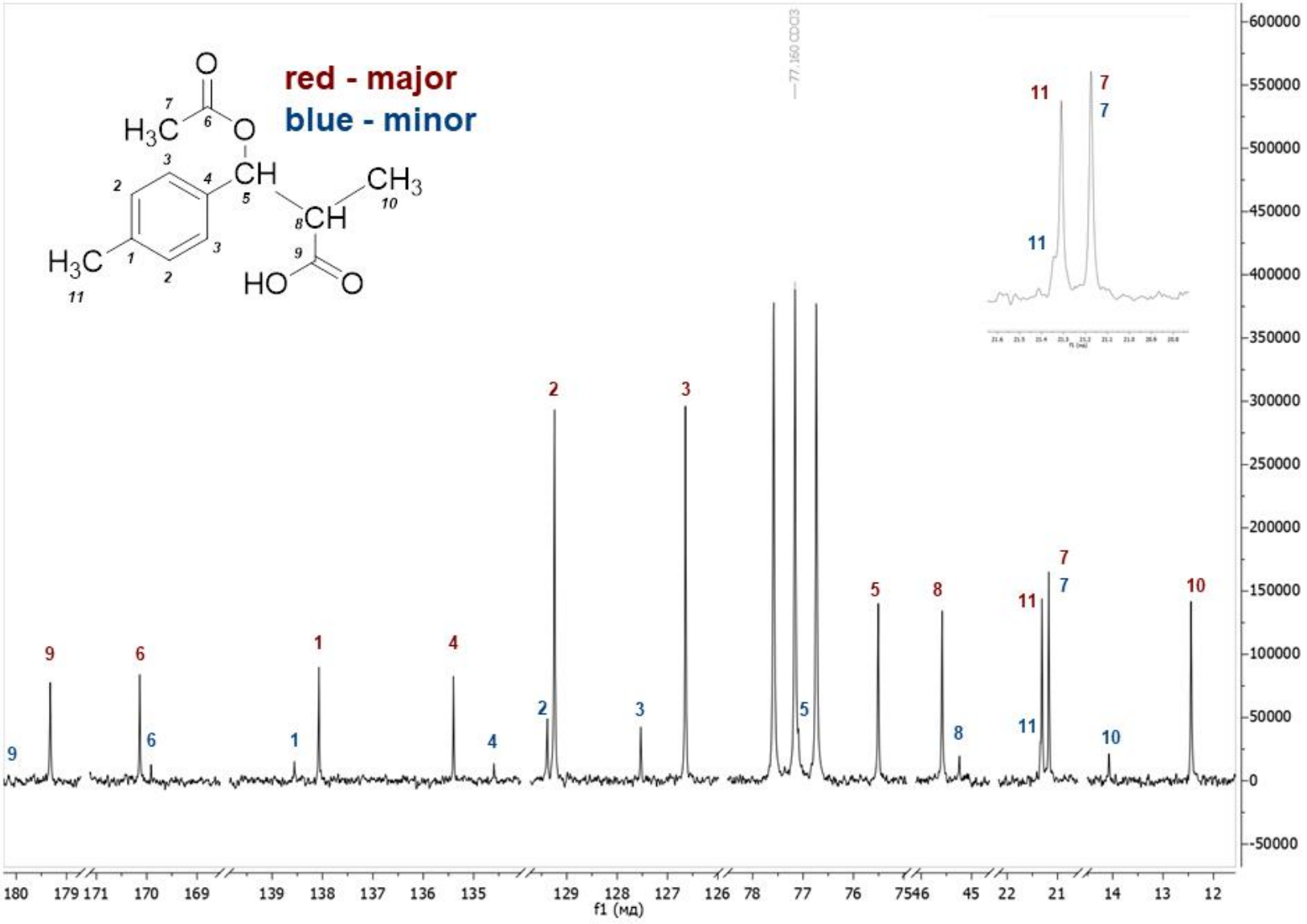
{^{1}H - ^{13}C} HSQC of **3-Acetoxy-2-methyl-3-(*p*-tolyl)propanoic acid, 2o**



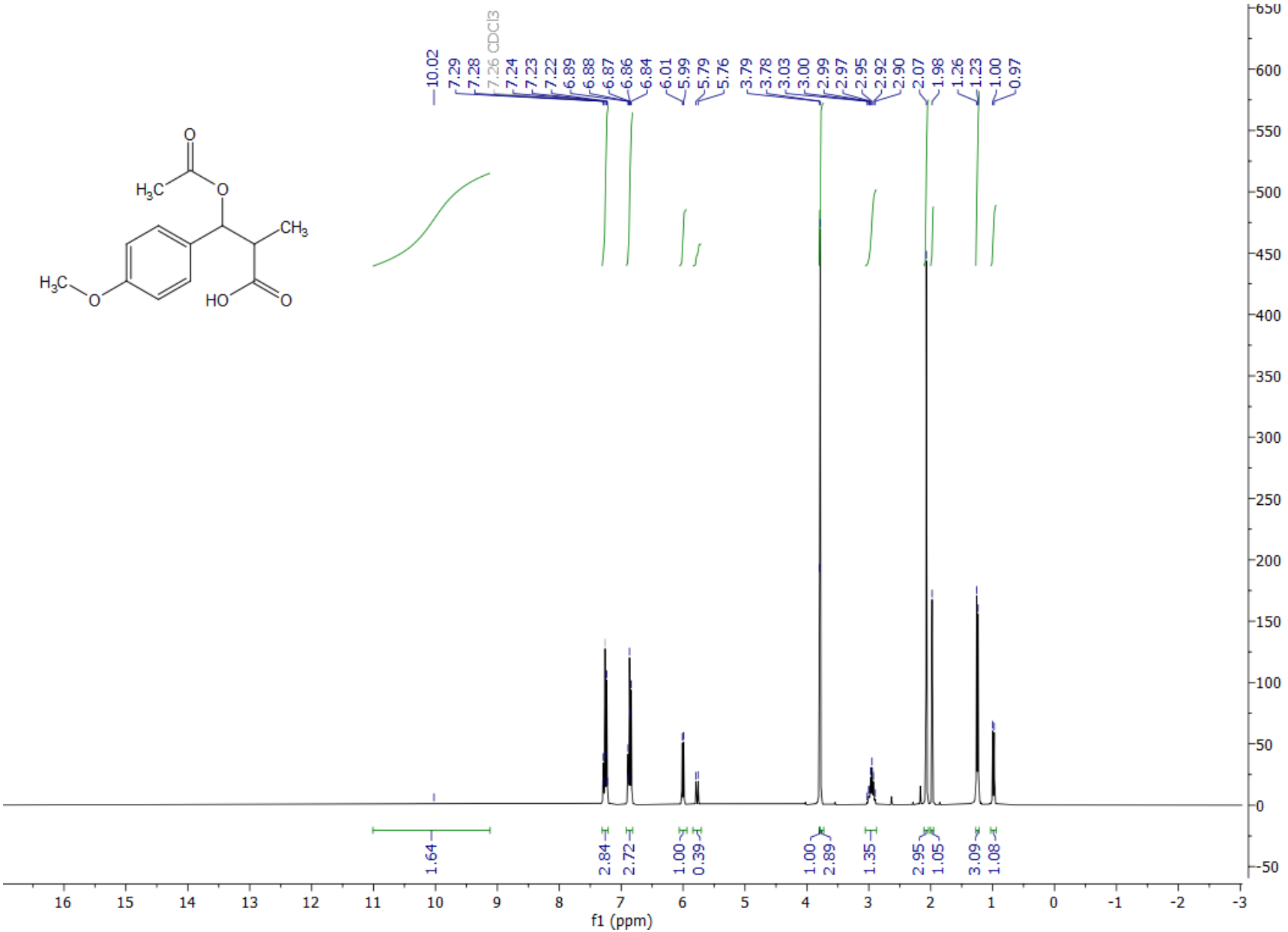
Correlation ^{1}H NMR (300.13 MHz, $CDCl_3$), **3-Acetoxy-2-methyl-3-(*p*-tolyl)propanoic acid, 2o**



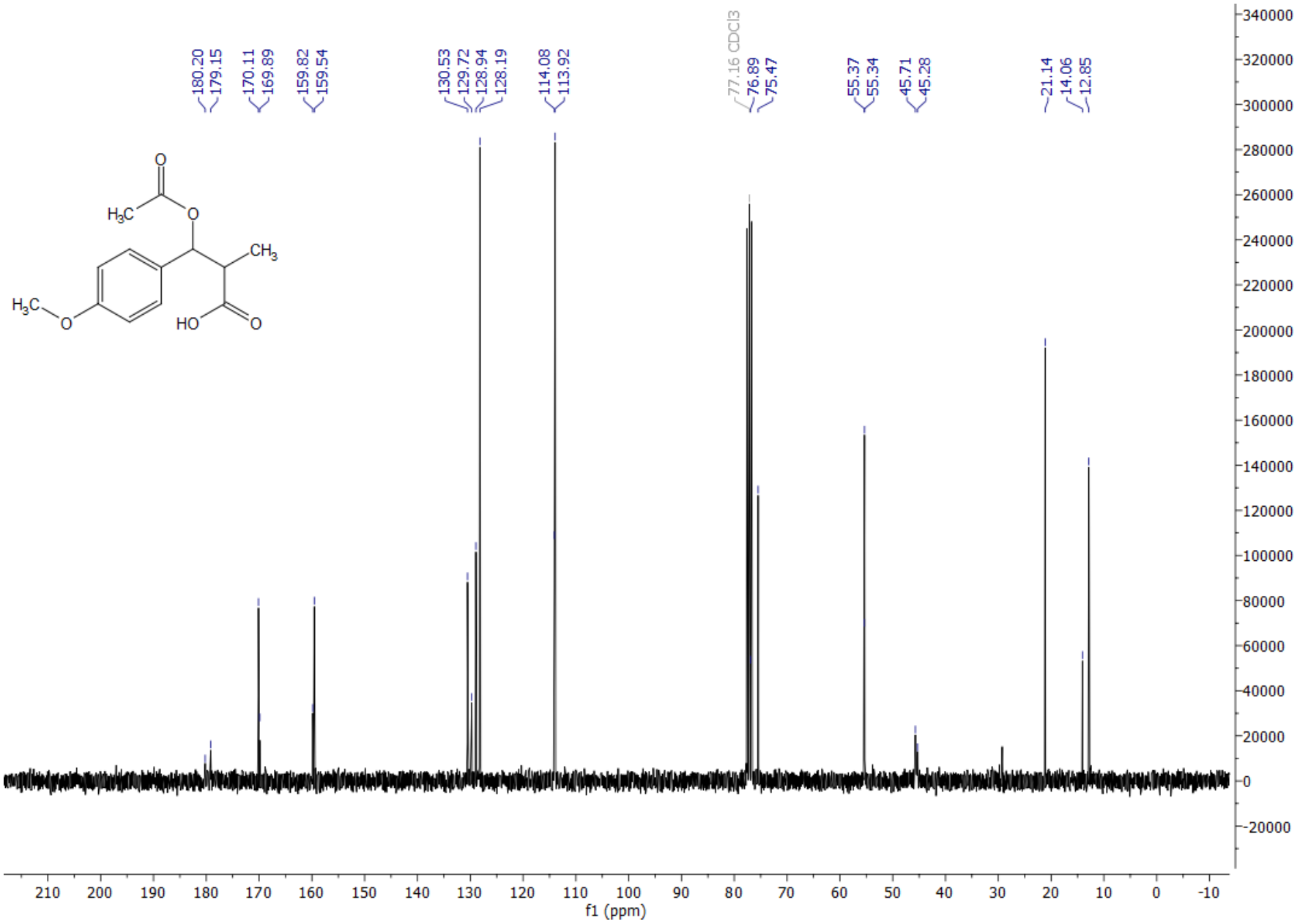
Correlation $^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **3-Acetoxy-2-methyl-3-(*p*-tolyl)propanoic acid, 2o**



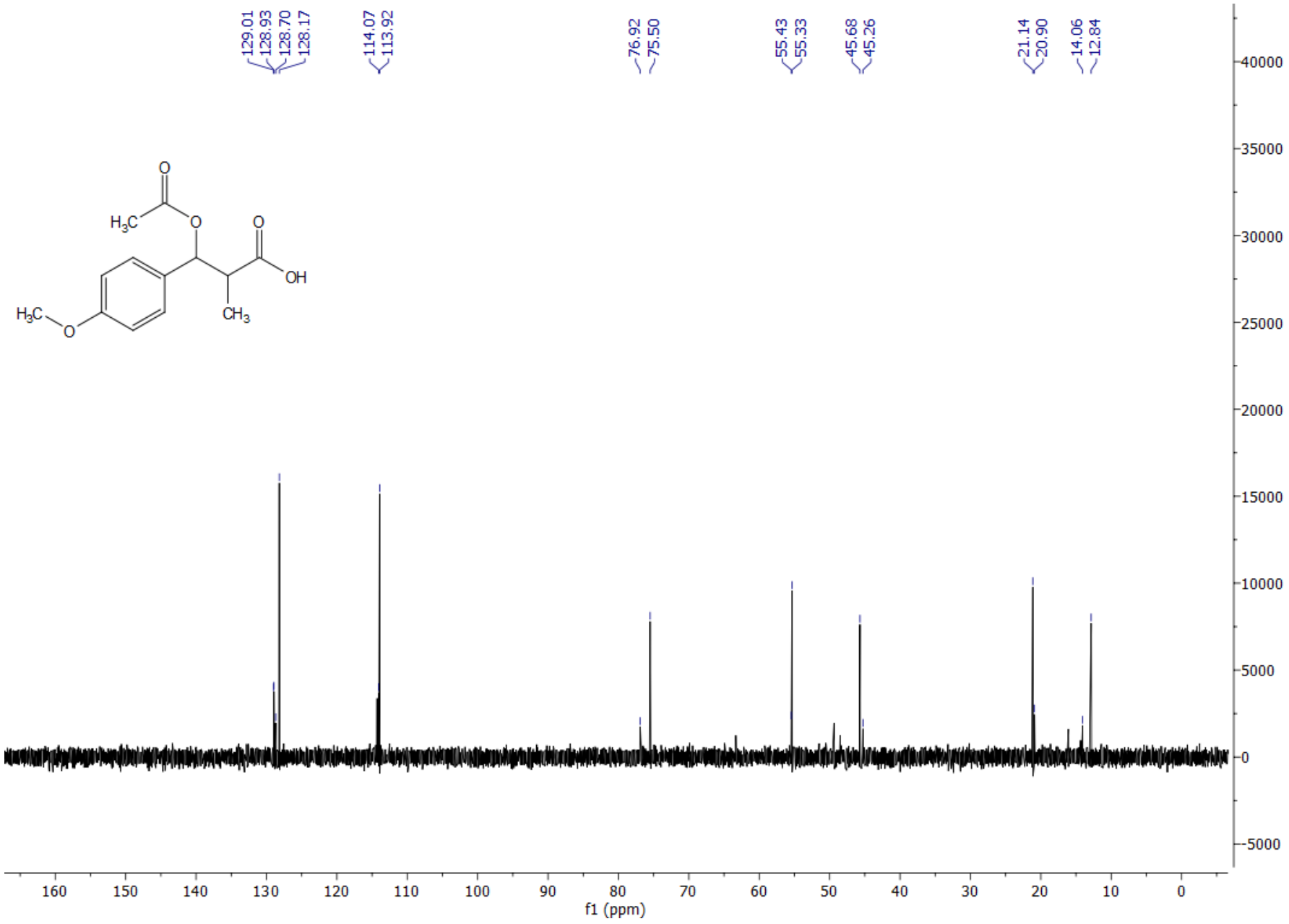
^{1}H NMR (300.13 MHz, $CDCl_3$), **3-Acetoxy-3-(4-methoxyphenyl)-2-methylpropanoic acid, 2p**



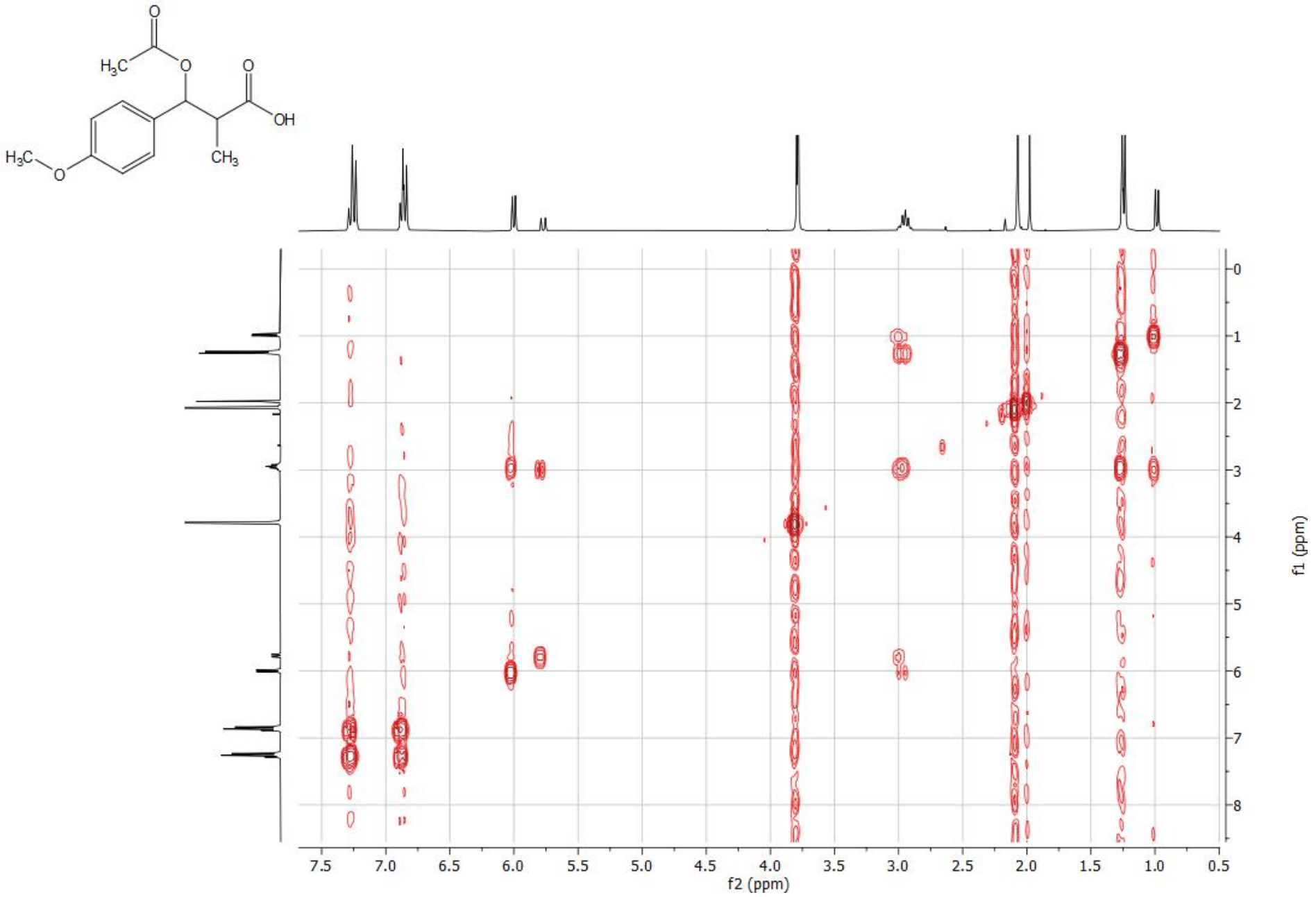
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **3-Acetoxy-3-(4-methoxyphenyl)-2-methylpropanoic acid, 2p**



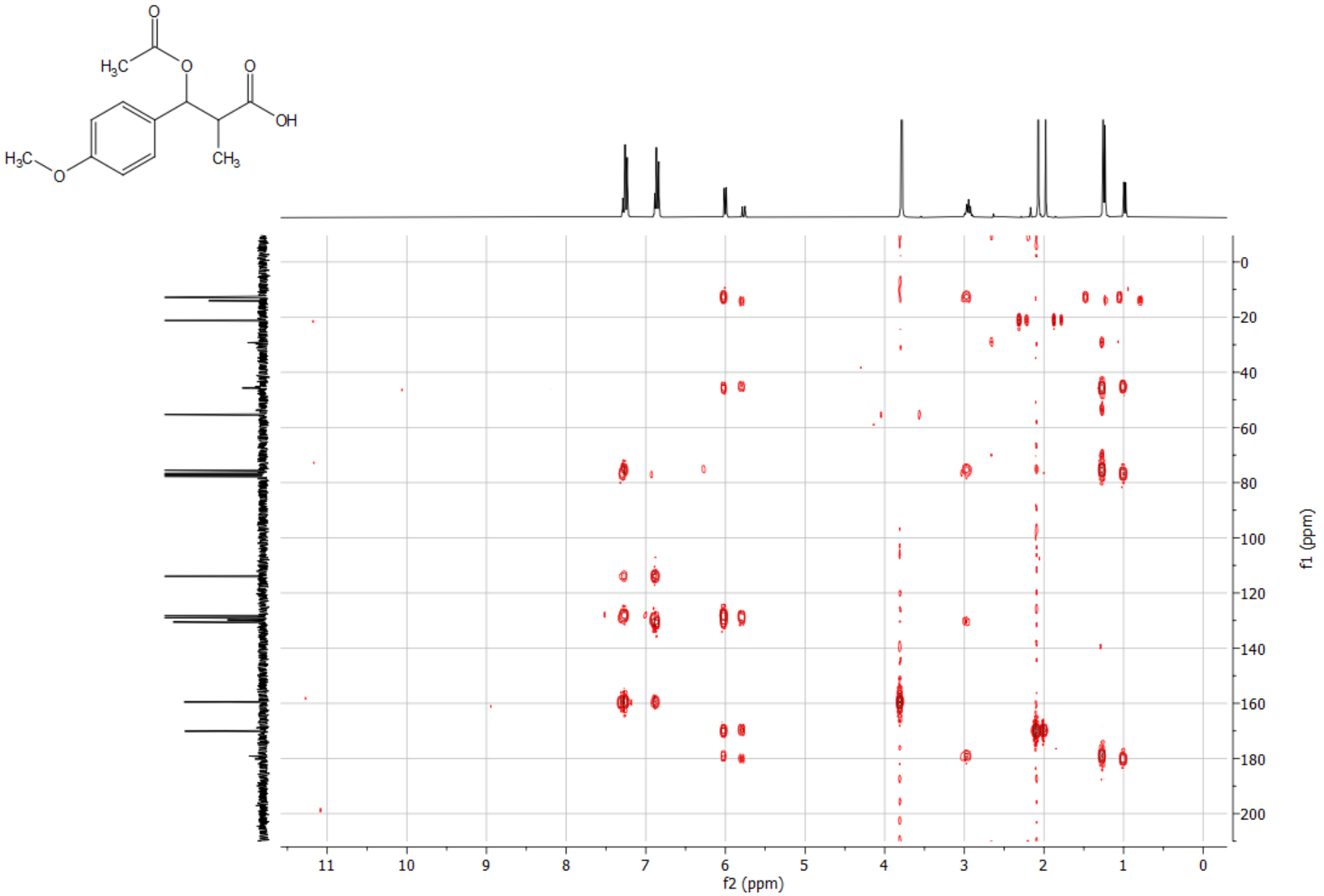
^{13}C DEPT135, **3-Acetoxy-3-(4-methoxyphenyl)-2-methylpropanoic acid, 2p**



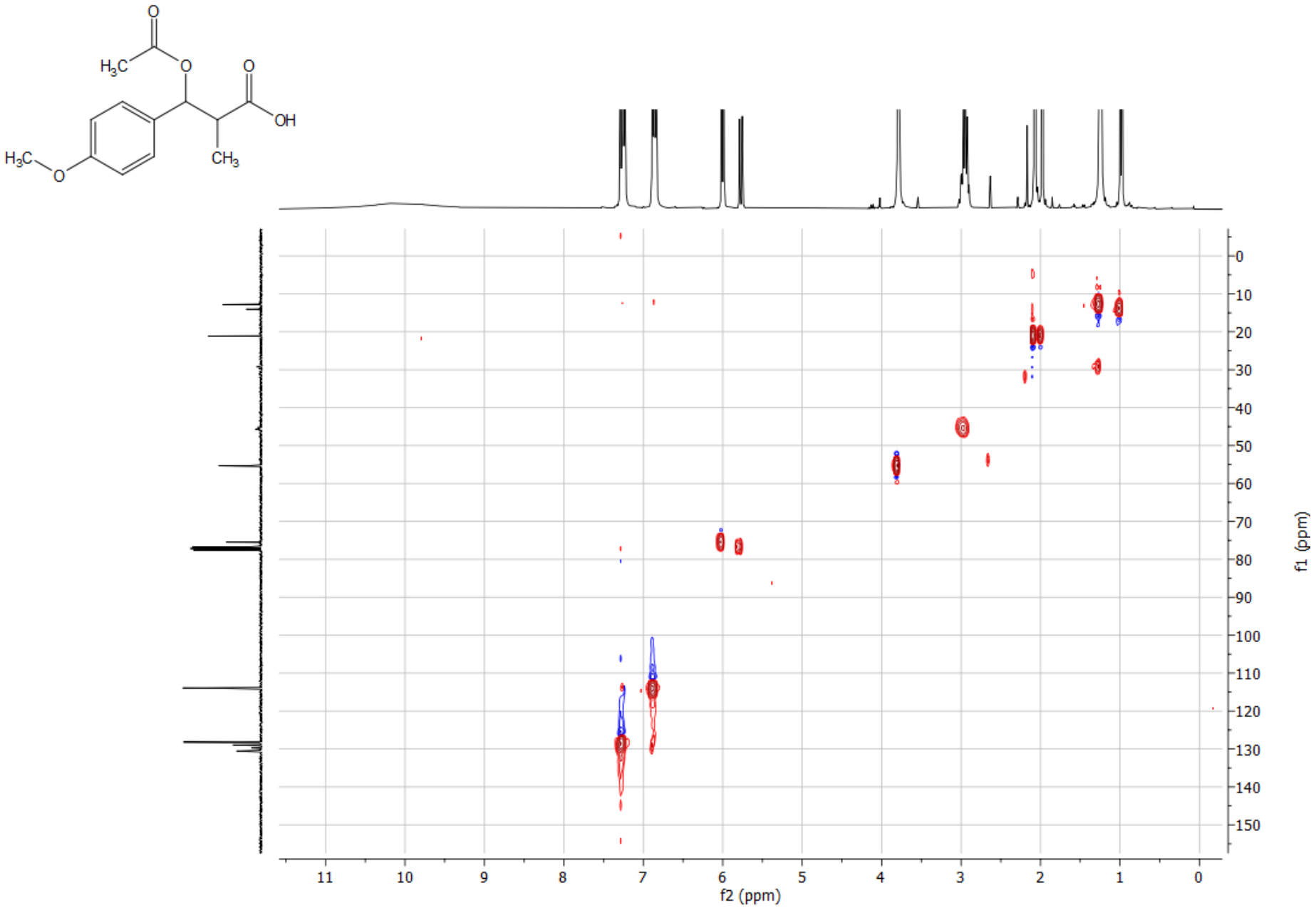
{^{1}H - ^{1}H} COSY of **3-Acetoxy-3-(4-methoxyphenyl)-2-methylpropanoic acid, 2p**



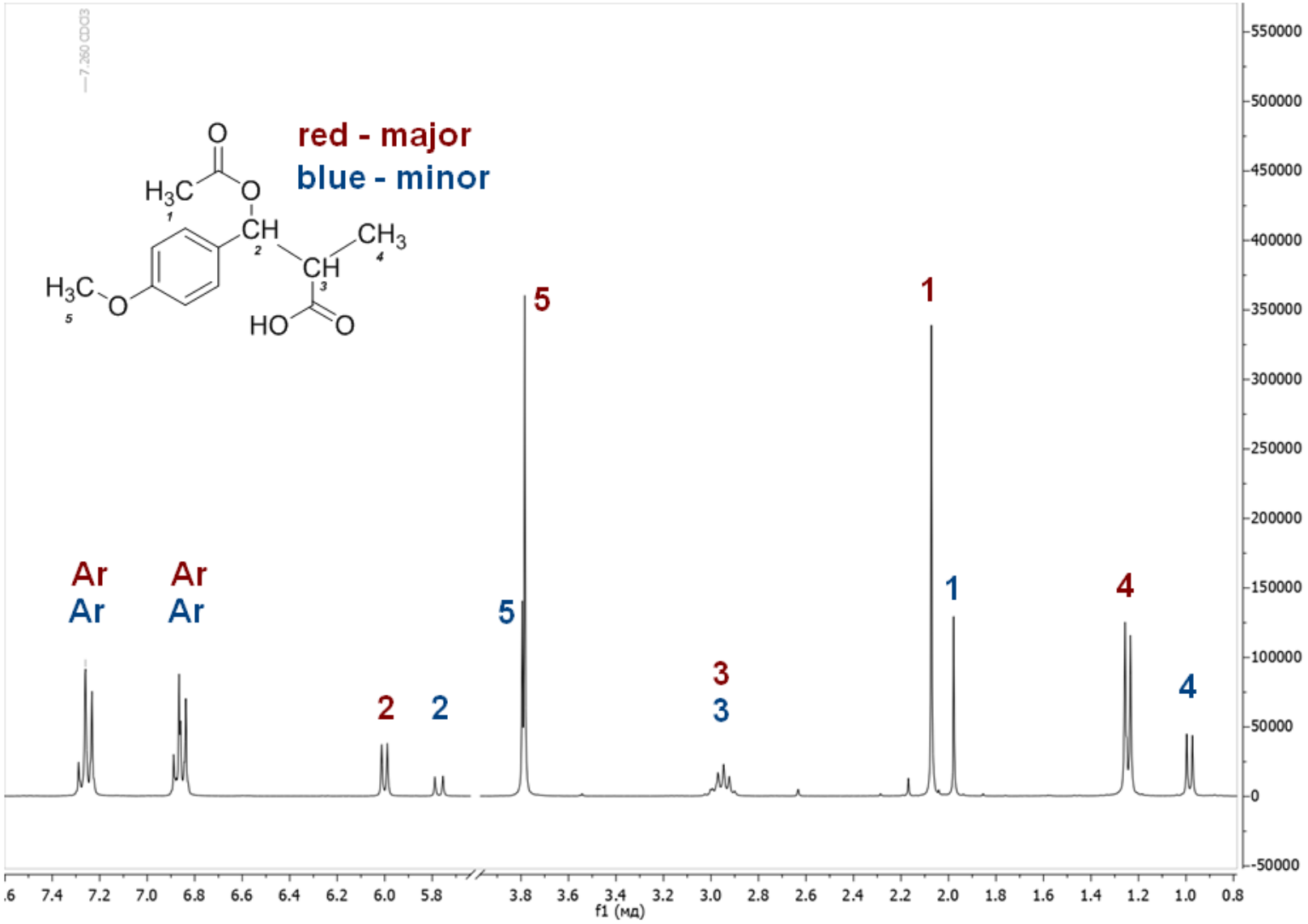
{^{1}H - ^{13}C} HMBC of **3-Acetoxy-3-(4-methoxyphenyl)-2-methylpropanoic acid, 2p**



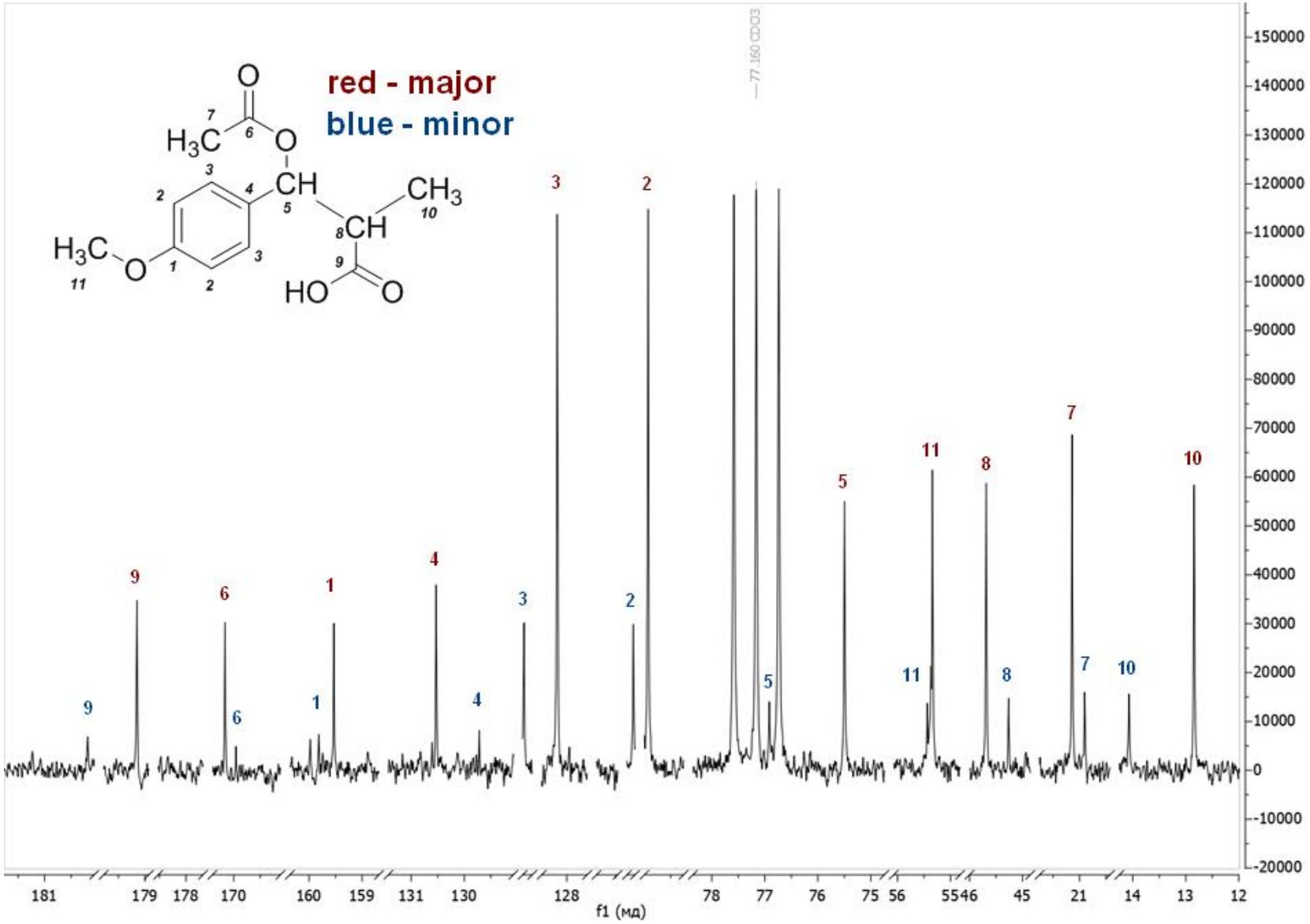
{^{1}H - ^{13}C} HSQC of **3-Acetoxy-3-(4-methoxyphenyl)-2-methylpropanoic acid, 2p**



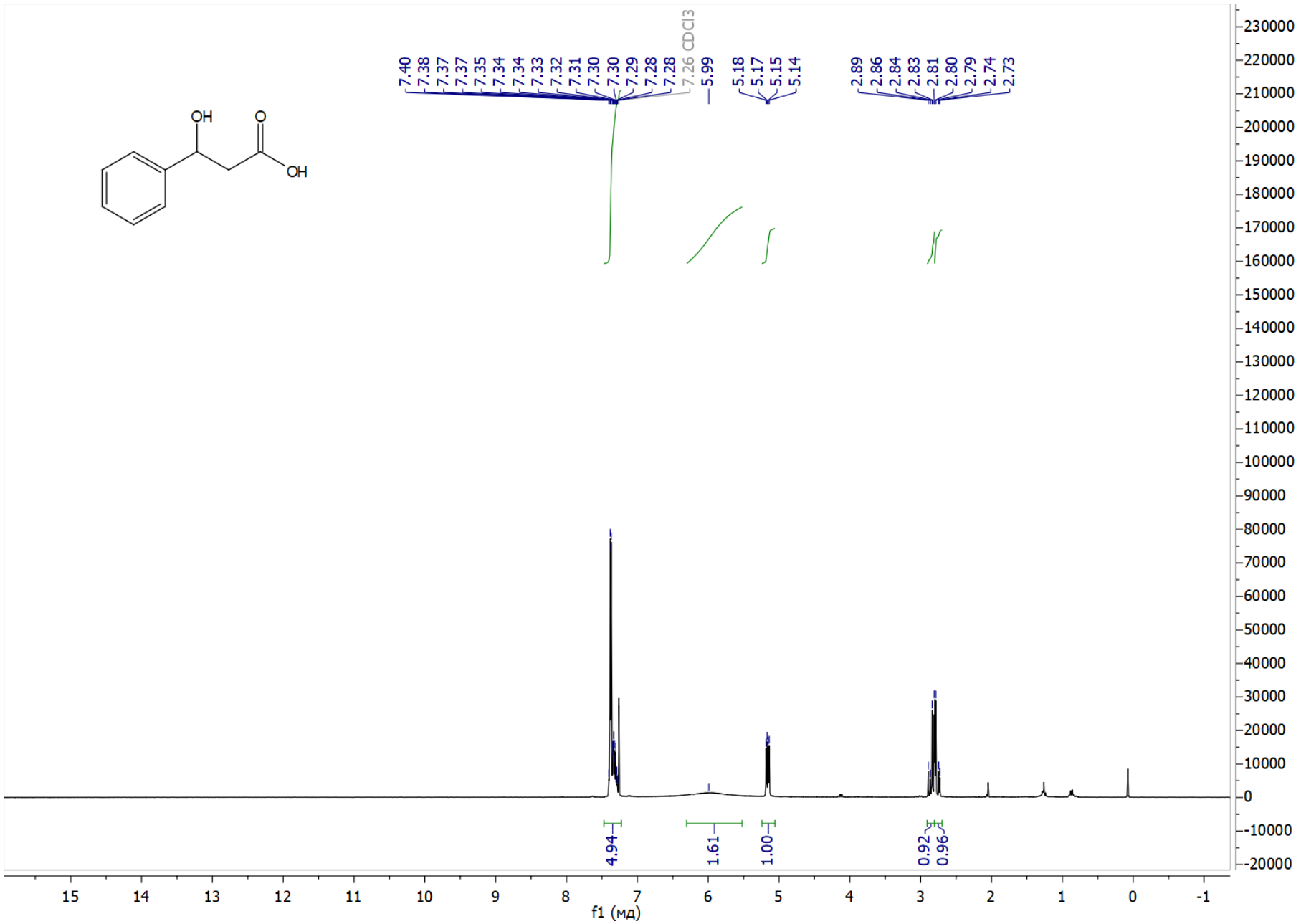
Correlation ^{1}H NMR (300.13 MHz, $CDCl_3$), **3-Acetoxy-3-(4-methoxyphenyl)-2-methylpropanoic acid, 2p**



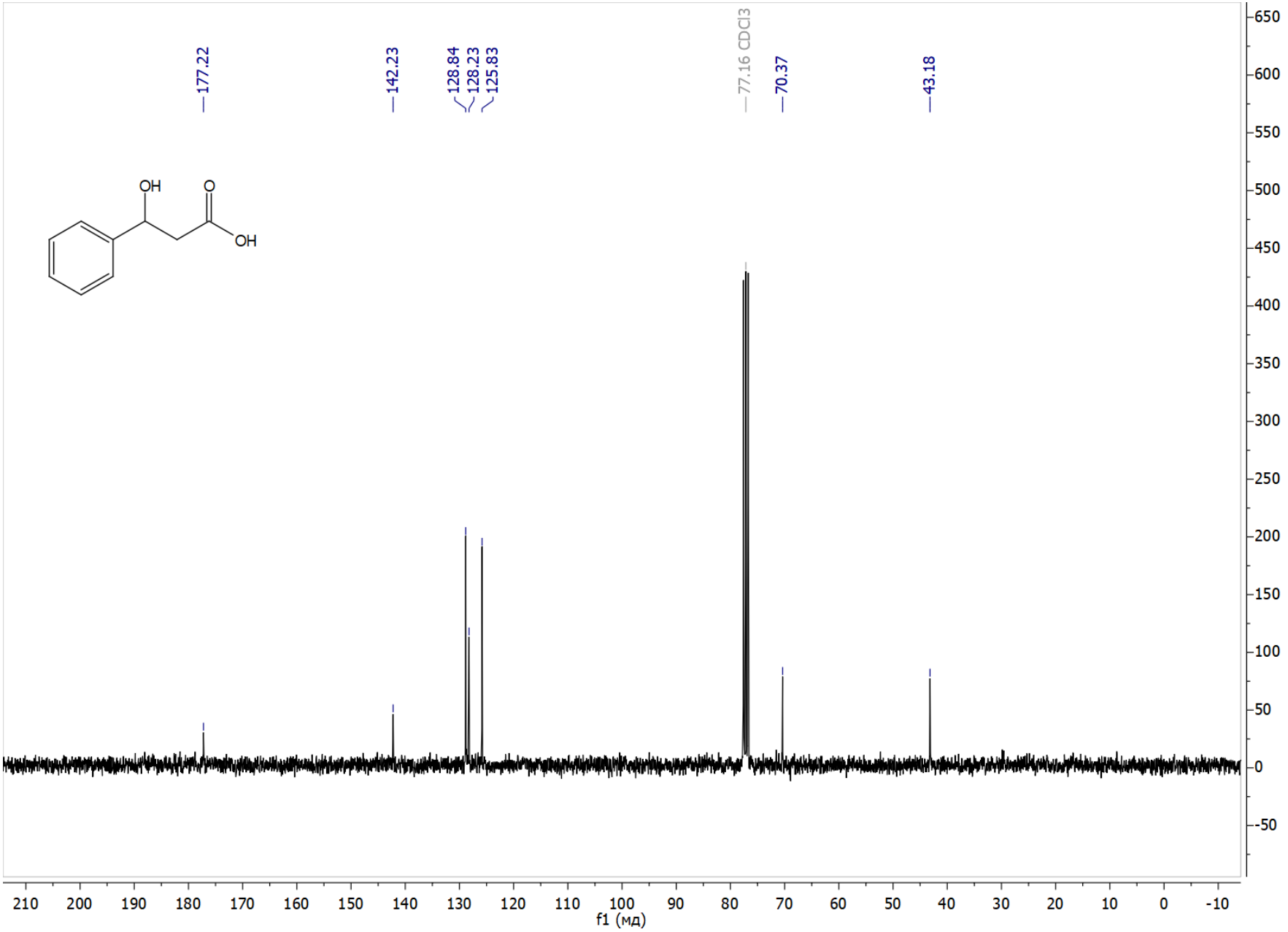
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **3-Acetoxy-3-(4-methoxyphenyl)-2-methylpropanoic acid, 2p**



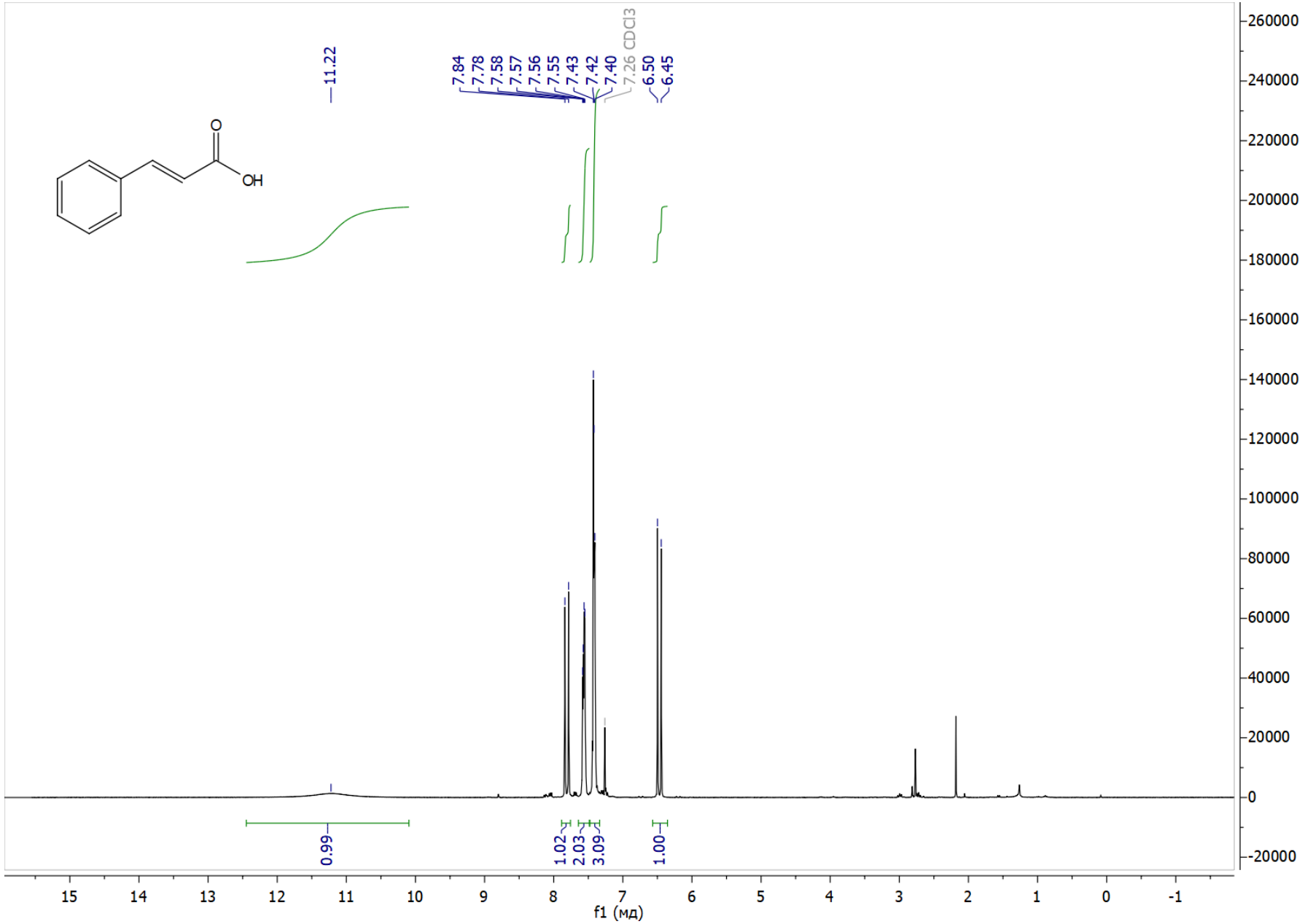
^{1}H NMR (300.13 MHz, $CDCl_3$), **3-Hydroxy-3-phenylpropanoic acid, 3a**



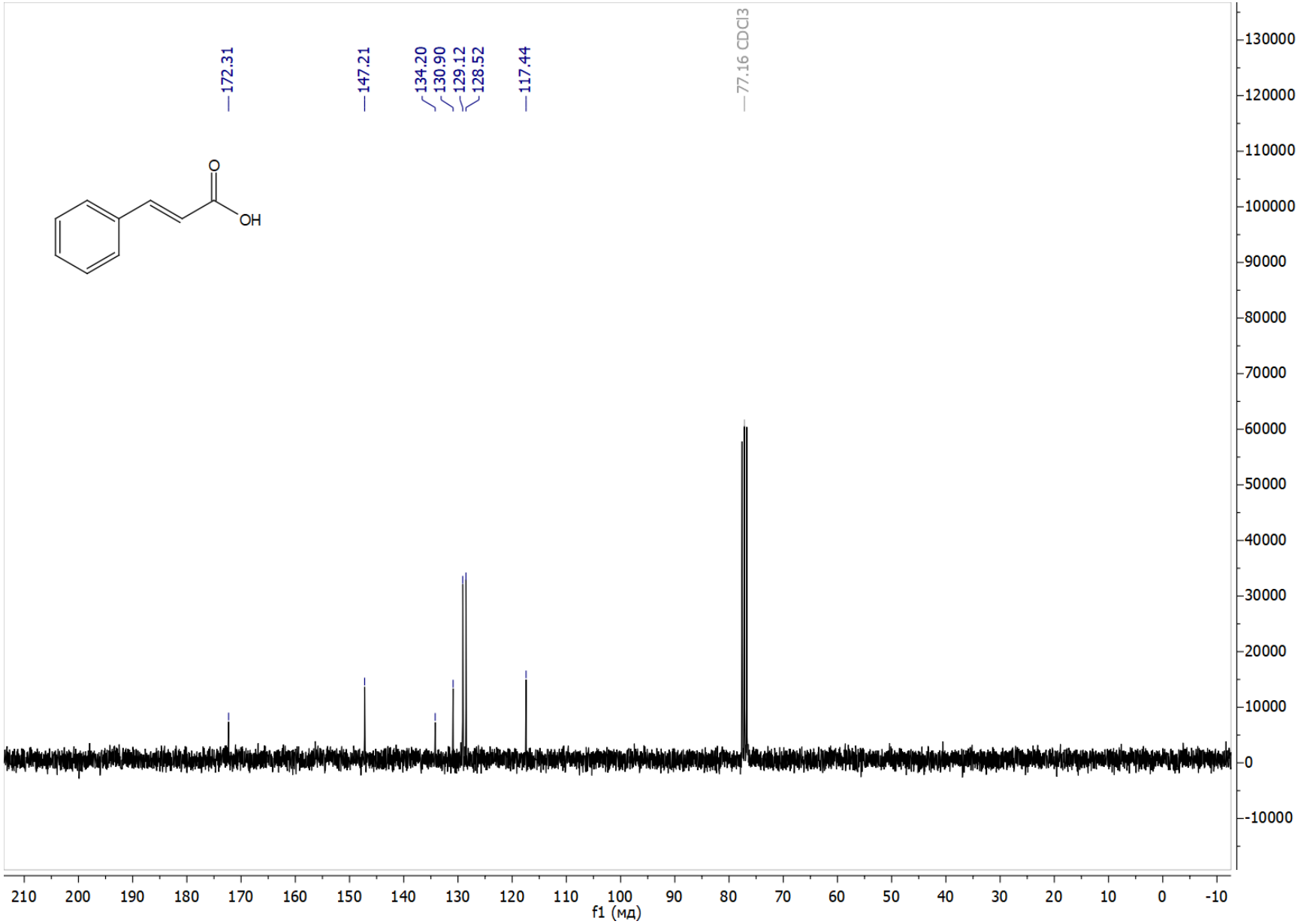
$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **3-Hydroxy-3-phenylpropanoic acid, 3a**



^{1}H NMR (300.13 MHz, $CDCl_3$), **3-Phenylacrylic acid, 4a**

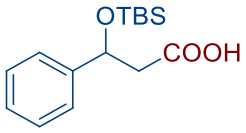


$^{13}C\{^{1}H\}$ NMR (75.48 MHz, $CDCl_3$), **3-Phenylacrylic acid, 4a**



HRMS spectra of the obtained products.

HRMS spectrum of **3-((*Tert*-butyldimethylsilyl)oxy)-3-phenylpropanoic acid, P5**



Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2024\Ustuzhanin\0319039.d		
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV MG451	Instrument / Ser#	micrOTOF 10248
Comment	C15H24O3Si mH281.1567 calibrant added CH3CN	Acquisition Date	19.03.2024 17:57:12

Acquisition Parameter

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Focus	Not active			Set Dry Heater	180 °C
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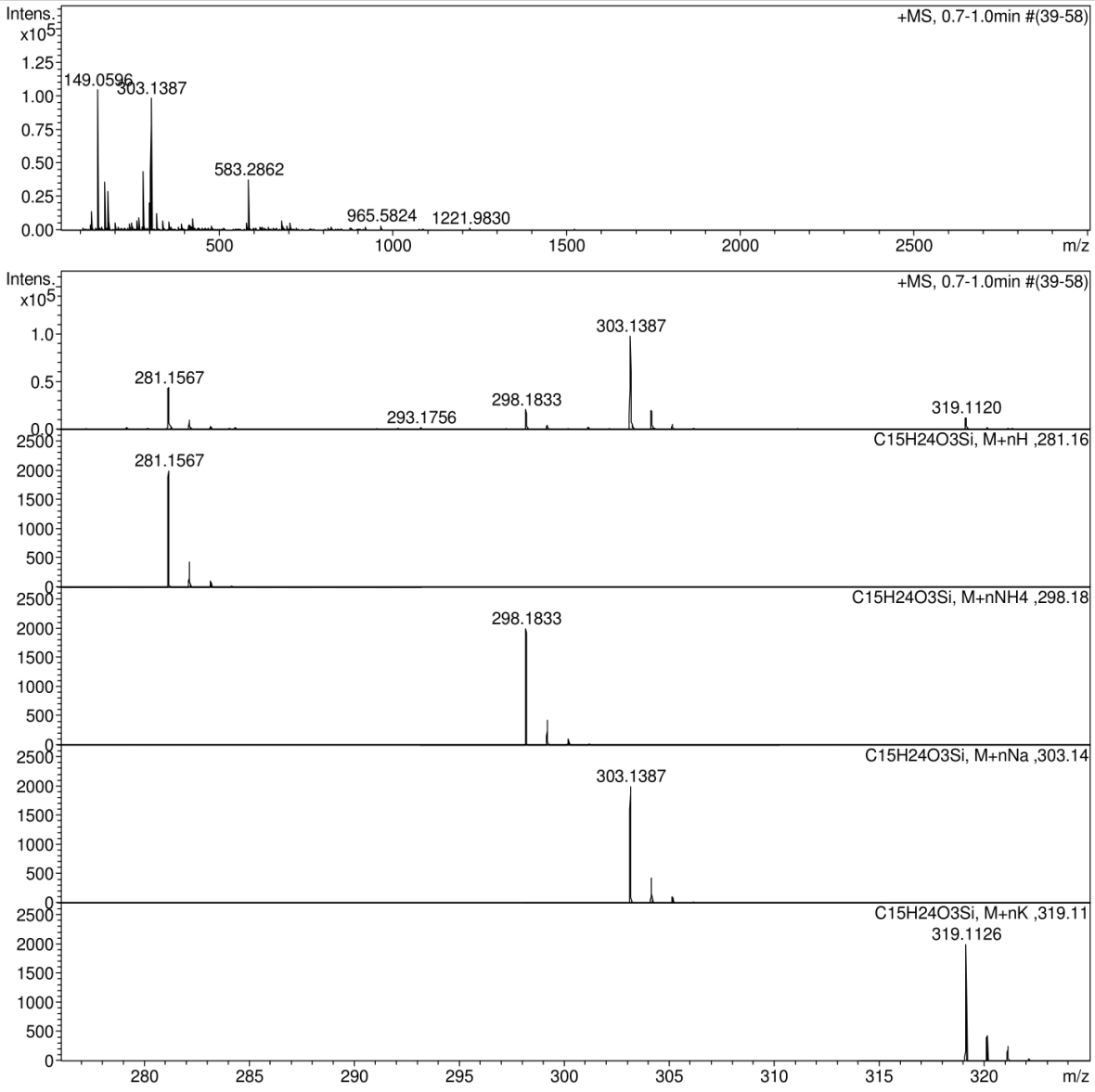
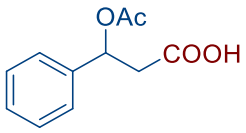
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of **1-Phenylvinyl acetate, 2a**



Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2023\Ustuzhanin\0124040.d	Acquisition Date	24.01.2023 17:34:24
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Sample Name	/TERN MK133	Instrument / Ser#	micrOTOF 10248
Comment	C11H12O4 mH209.0808 calibrant added CH3CN		

Acquisition Parameter

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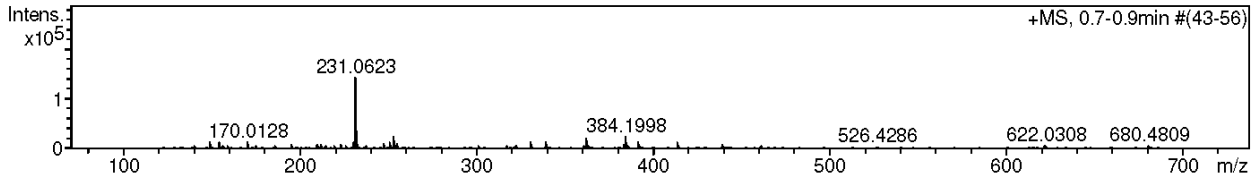
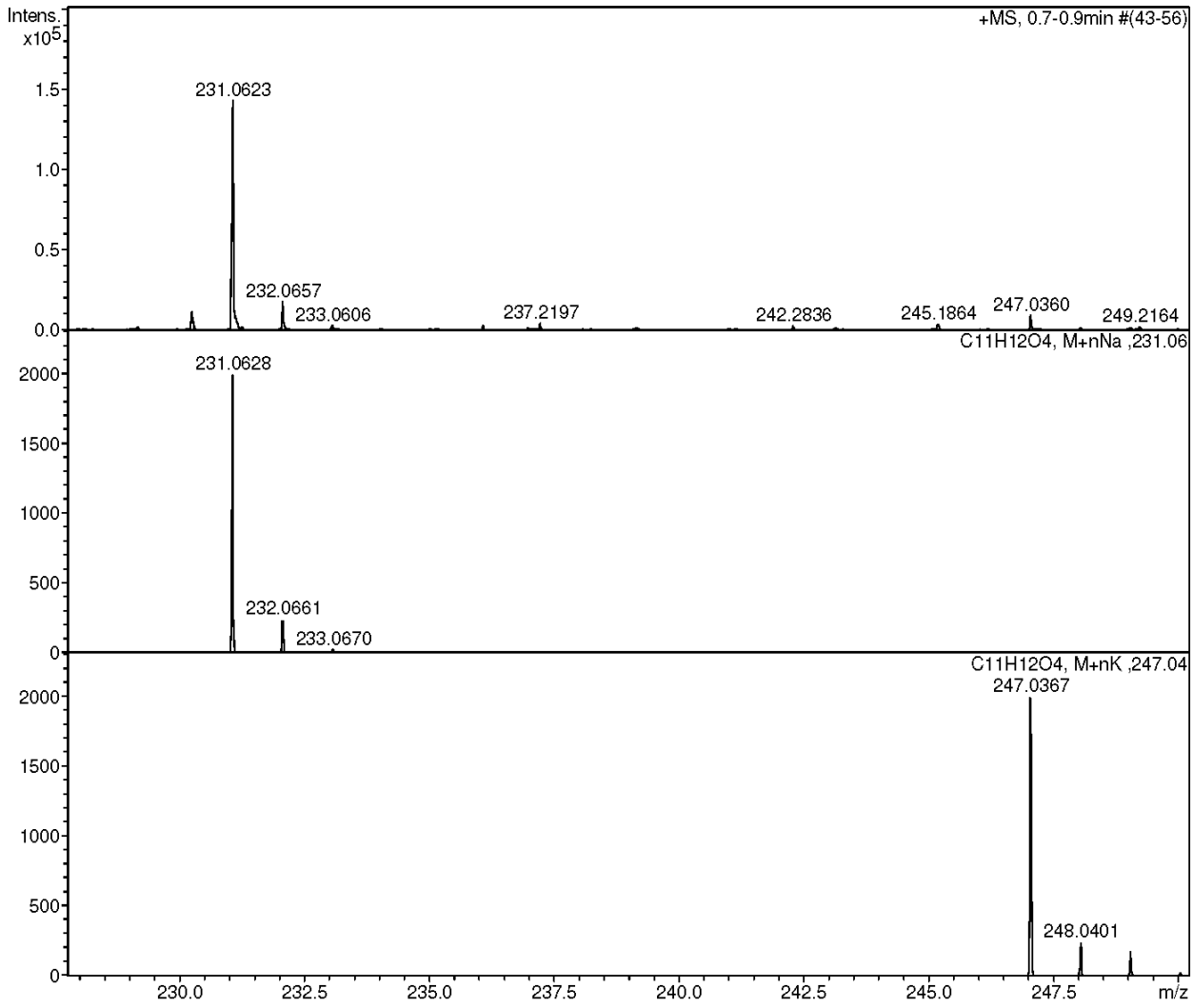
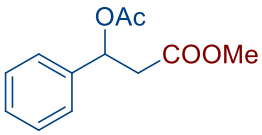
Scan End	2500 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste





HRMS spectrum of **methyl 3-acetoxy-3-phenylpropanoate, 2a'**



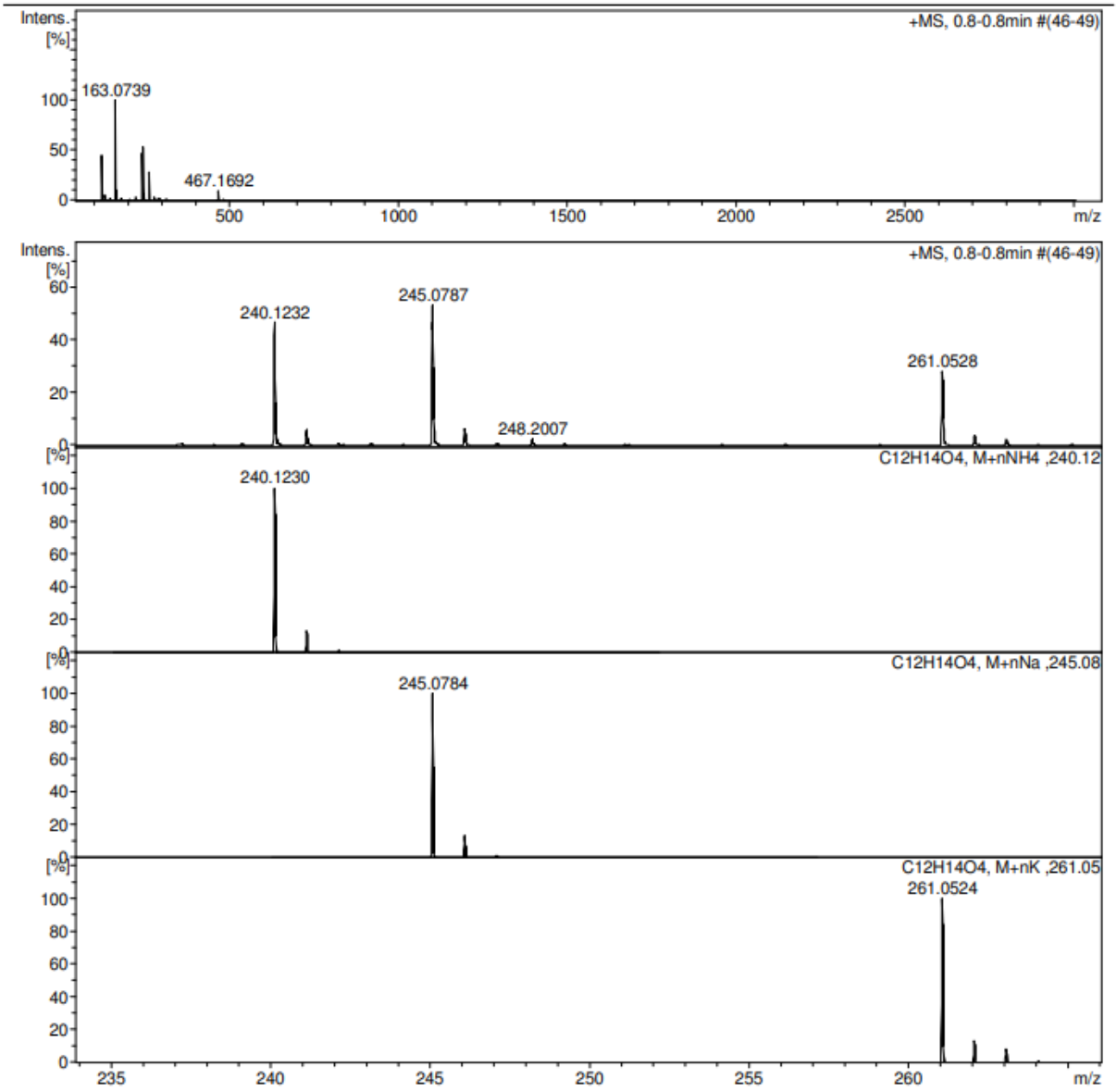
Display Report

Analysis Info

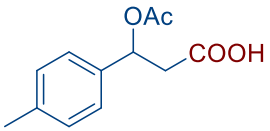
Analysis Name	D:\Data\Kolotyrkina\2024\Ustuzhanin\0627002.d	Acquisition Date	27.06.2024 9:30:25
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV MG313	Instrument / Ser#	micrOTOF 10248
Comment	C12H14O4 mH223.0964 calibrant added CH3CN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of **3-Acetoxy-3-(p-tolyl)propanoic acid, 2b**



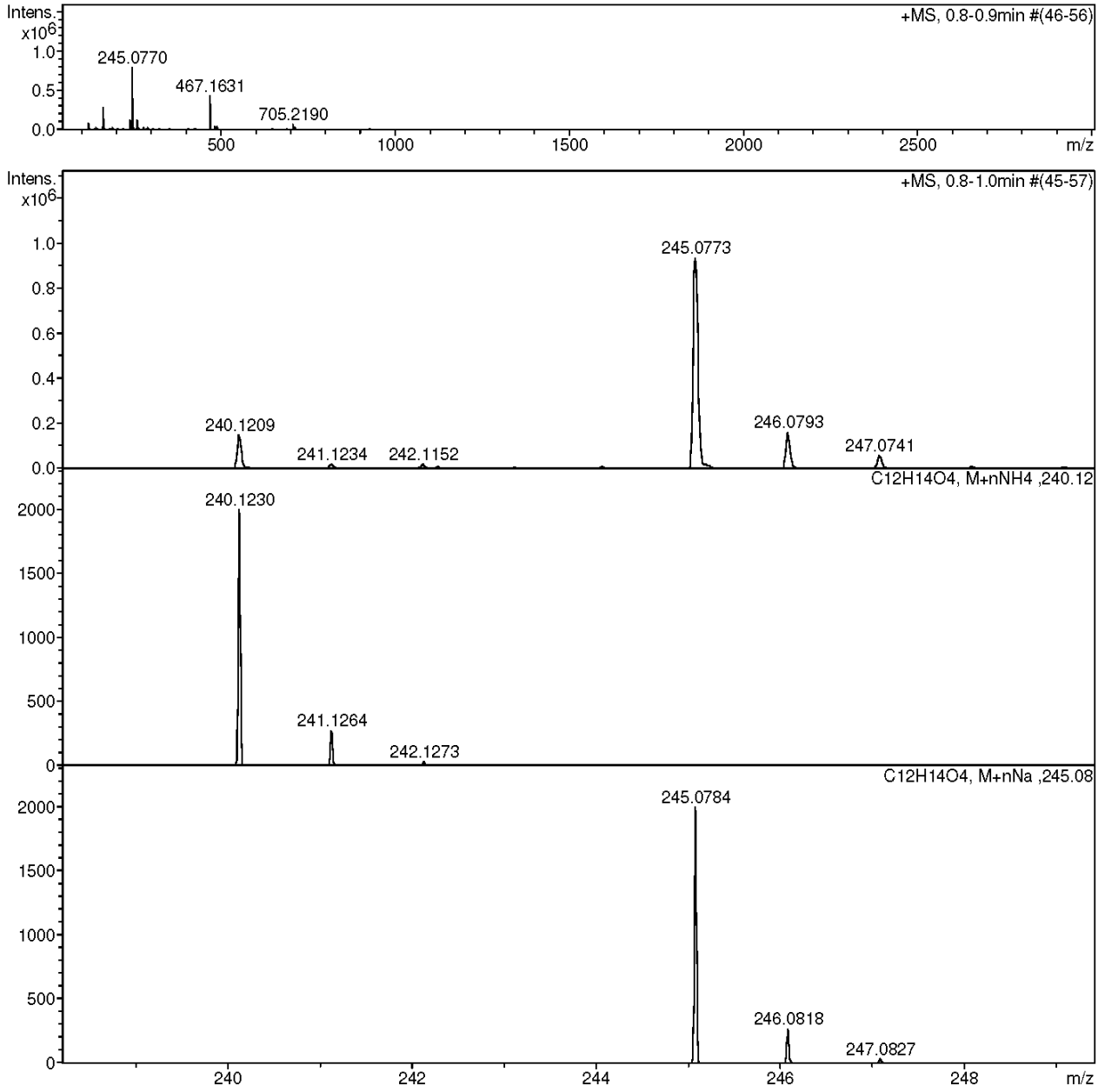
Display Report

Analysis Info

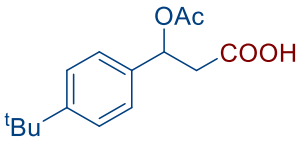
Analysis Name	D:\Data\Kolotyrkina\2023\Ustuzhanin\1129011.d	Acquisition Date	29.11.2023 11:59:56
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV MG322	Instrument / Ser#	micrOTOF 10248
Comment	C12H14O4 mH223.0964 calibrant added CH3OH		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of **3-Acetoxy-3-(4-(tert-butyl)phenyl)propanoic acid, 2c**



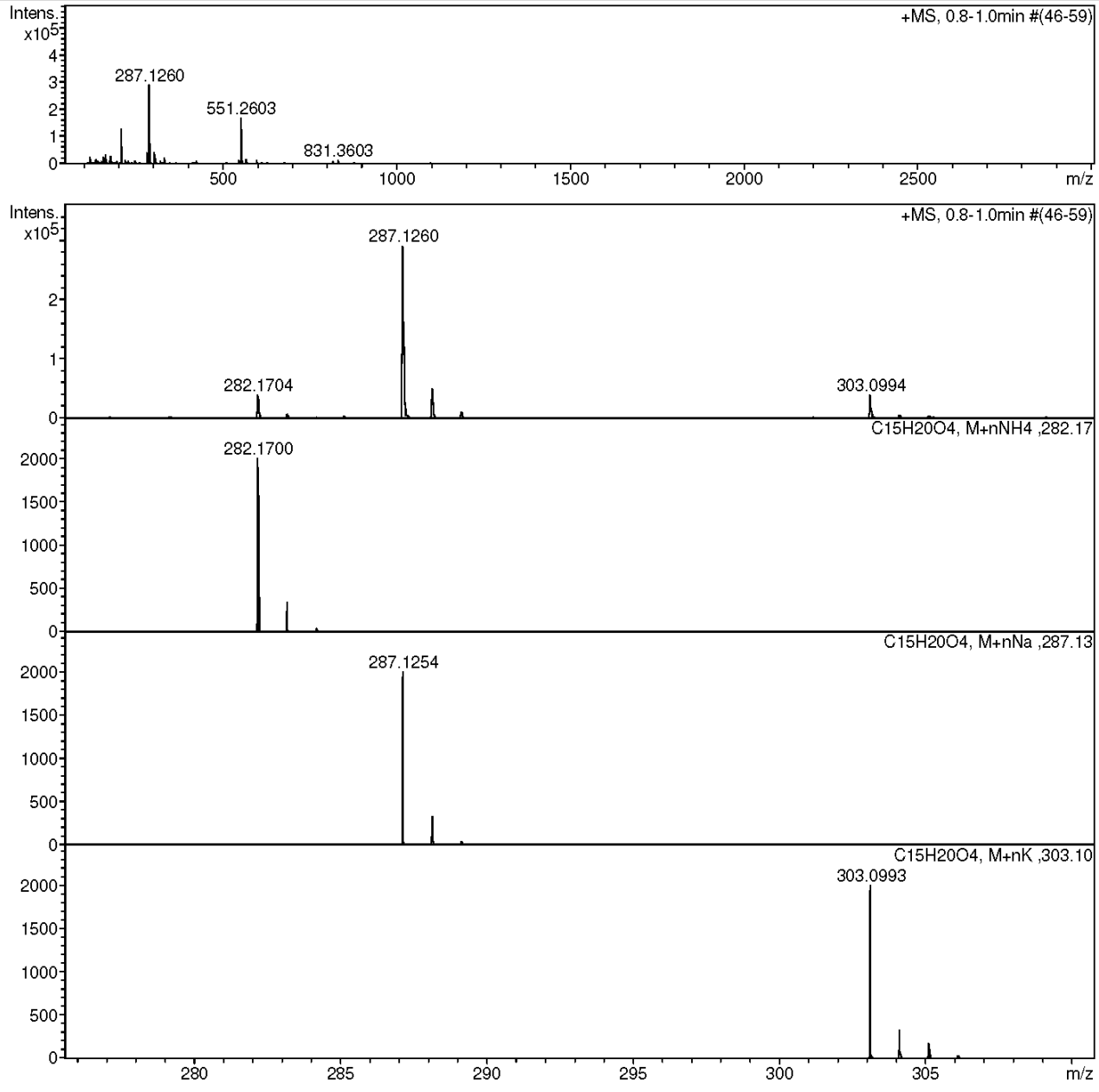
Display Report

Analysis Info

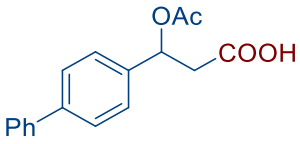
Analysis Name	D:\Data\Kolotyrkina\2023\Ustuzhanin\1208028.d	Acquisition Date	08.12.2023 16:56:10
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV MG390	Instrument / Ser#	micrOTOF 10248
Comment	C15H20O4 mH265.1434 calibrant added CH3OH		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of **3-([1,1'-Biphenyl]-4-yl)-3-acetoxypropanoic acid, 2d**



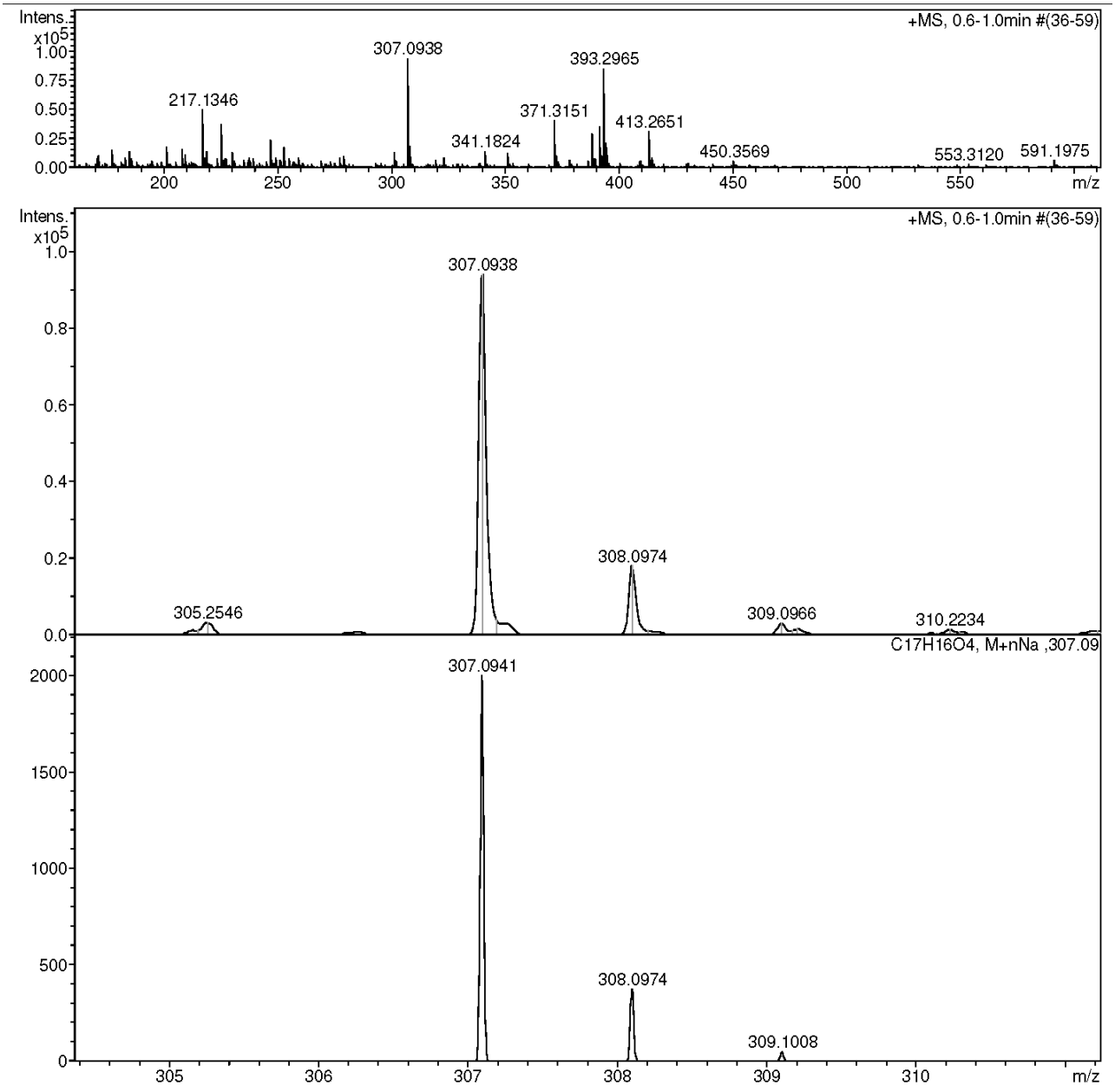
Display Report

Analysis Info

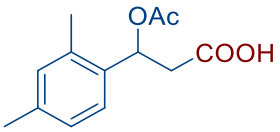
Analysis Name	D:\Data\Kolotyrkina\2023\Ustuzhanin\1207018.d	Acquisition Date	07.12.2023 14:12:14
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV MG387	Instrument / Ser#	micrOTOF 10248
Comment	C17H16O4 mH 285.1121 calibrant added CH3OH		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of **3-Acetoxy-3-(2,4-dimethylphenyl)propanoic acid, 2e**



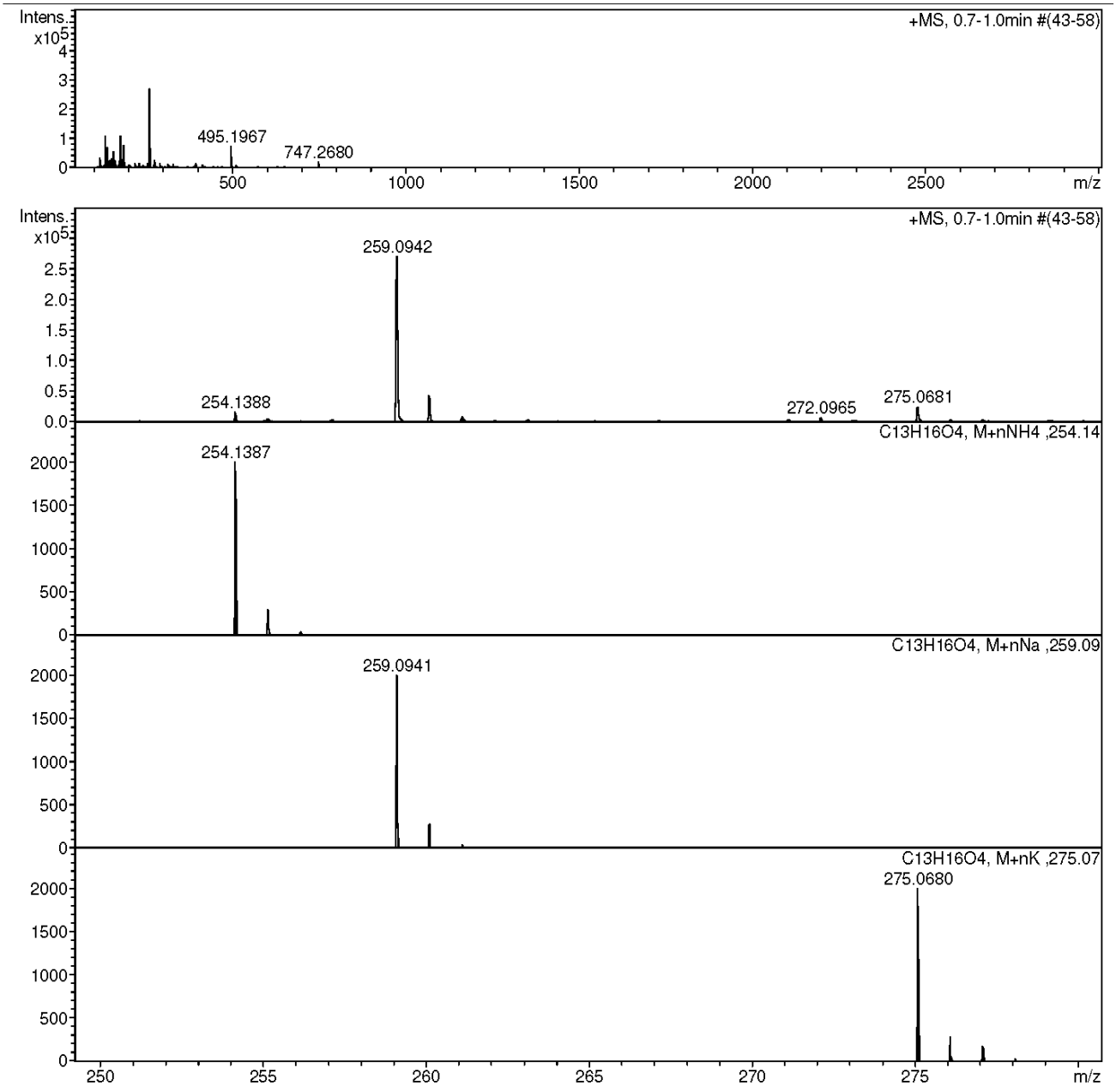
Display Report

Analysis Info

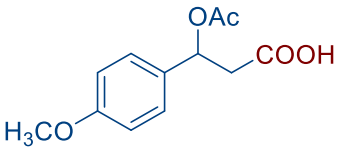
Analysis Name	D:\Data\Kolotyrkina\2023\Ustuzhanin\1208005.d	Acquisition Date	08.12.2023 8:42:29
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV MG388	Instrument / Ser#	micrOTOF 10248
Comment	C13H16O4 mH 237.1121 calibrant added CH3OH		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of **3-Acetoxy-3-(4-methoxyphenyl)propanoic acid, 2f**



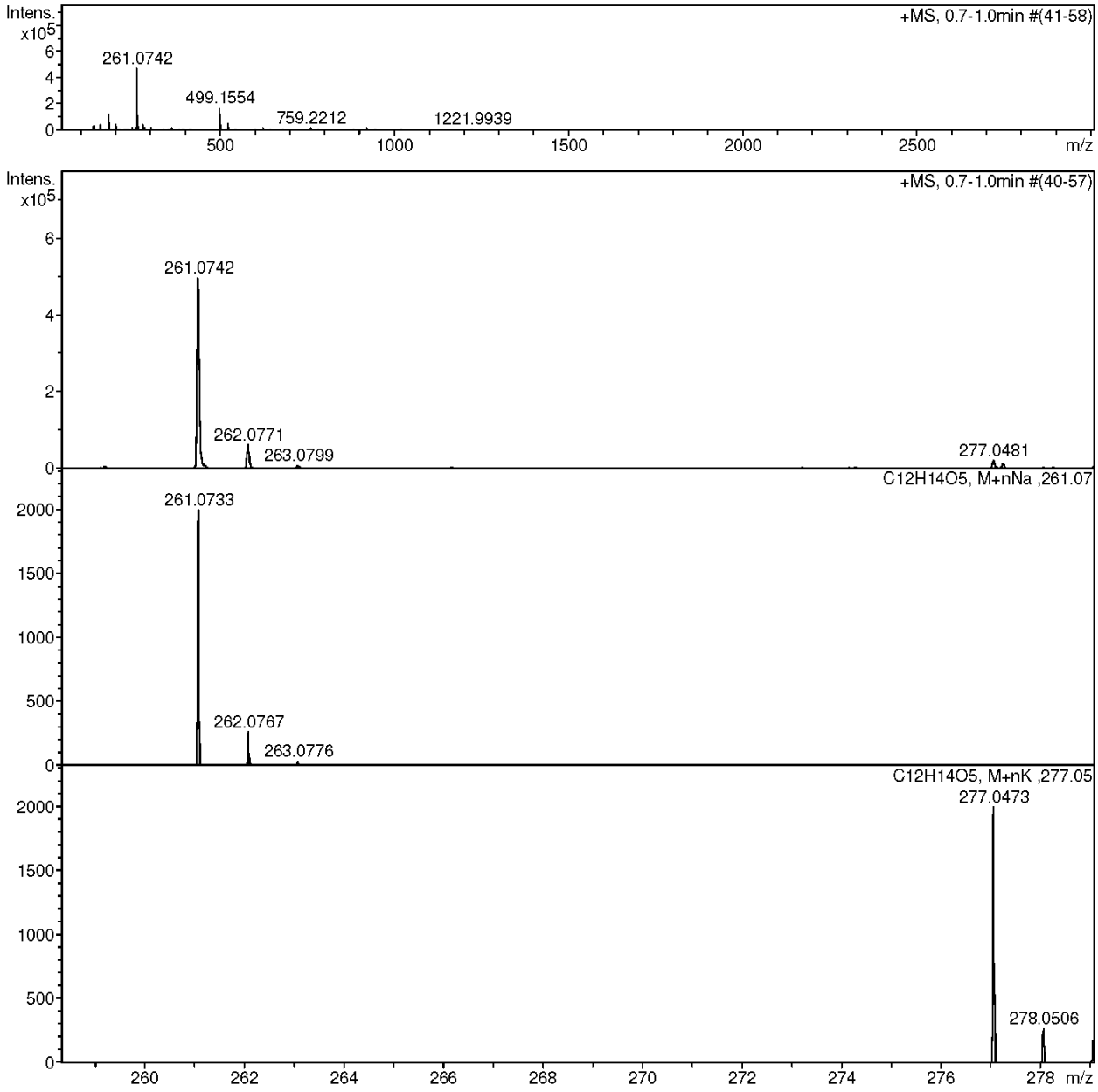
Display Report

Analysis Info

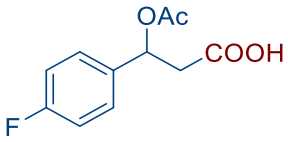
Analysis Name	D:\Data\Kolotyrkina\2023\Ustuzhanin\1129008.d	Acquisition Date	29.11.2023 11:27:23
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV MG323	Instrument / Ser#	micrOTOF 10248
Comment	C12H14O5 mH239.0914 calibrant added CH3OH		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of **3-Acetoxy-3-(4-fluorophenyl)propanoic acid, 2g**



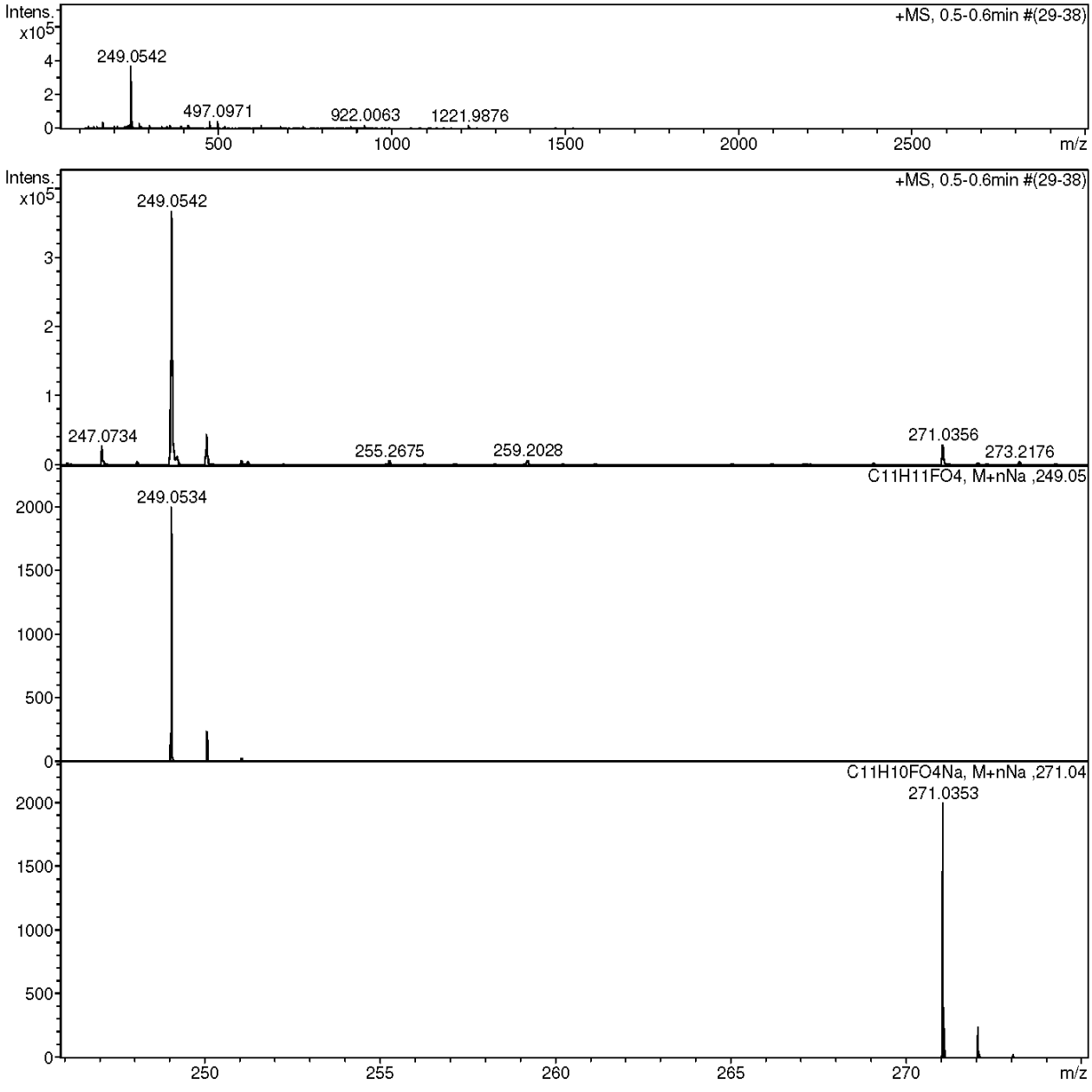
Display Report

Analysis Info

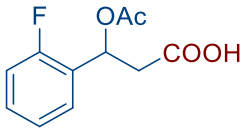
Analysis Name	D:\Data\Kolotyrkina\2023\Ustuzhanin\1129012.d	Acquisition Date	29.11.2023 12:08:53
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV MG291	Instrument / Ser#	micrOTOF 10248
Comment	C11H11FO4 mH227.0714 calibrant added CH3OH		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of **3-Acetoxy-3-(2-fluorophenyl)propanoic acid, 2h**



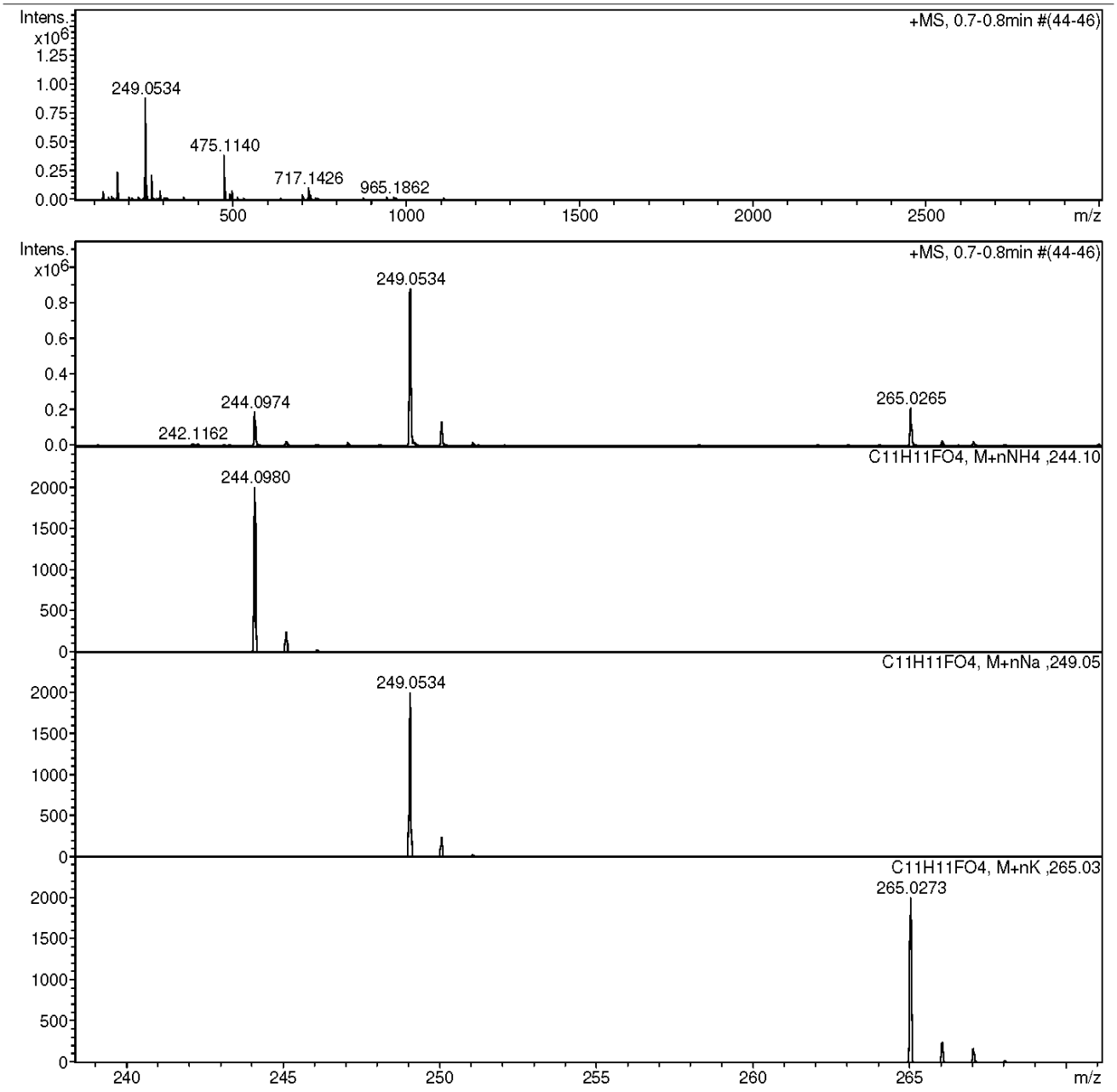
Display Report

Analysis Info

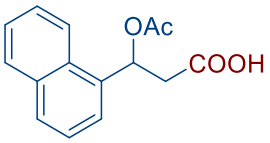
Analysis Name	D:\Data\Kolotyrkina\2023\Ustuzhanin\1031034.d	Acquisition Date	31.10.2023 16:17:10
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/TERN MG-394	Instrument / Ser#	micrOTOF 10248
Comment	C11H11FO4 mH227.0714 calibrant added CH3OH		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of **3-Acetoxy-3-(naphthalen-1-yl)propanoic acid, 2i**



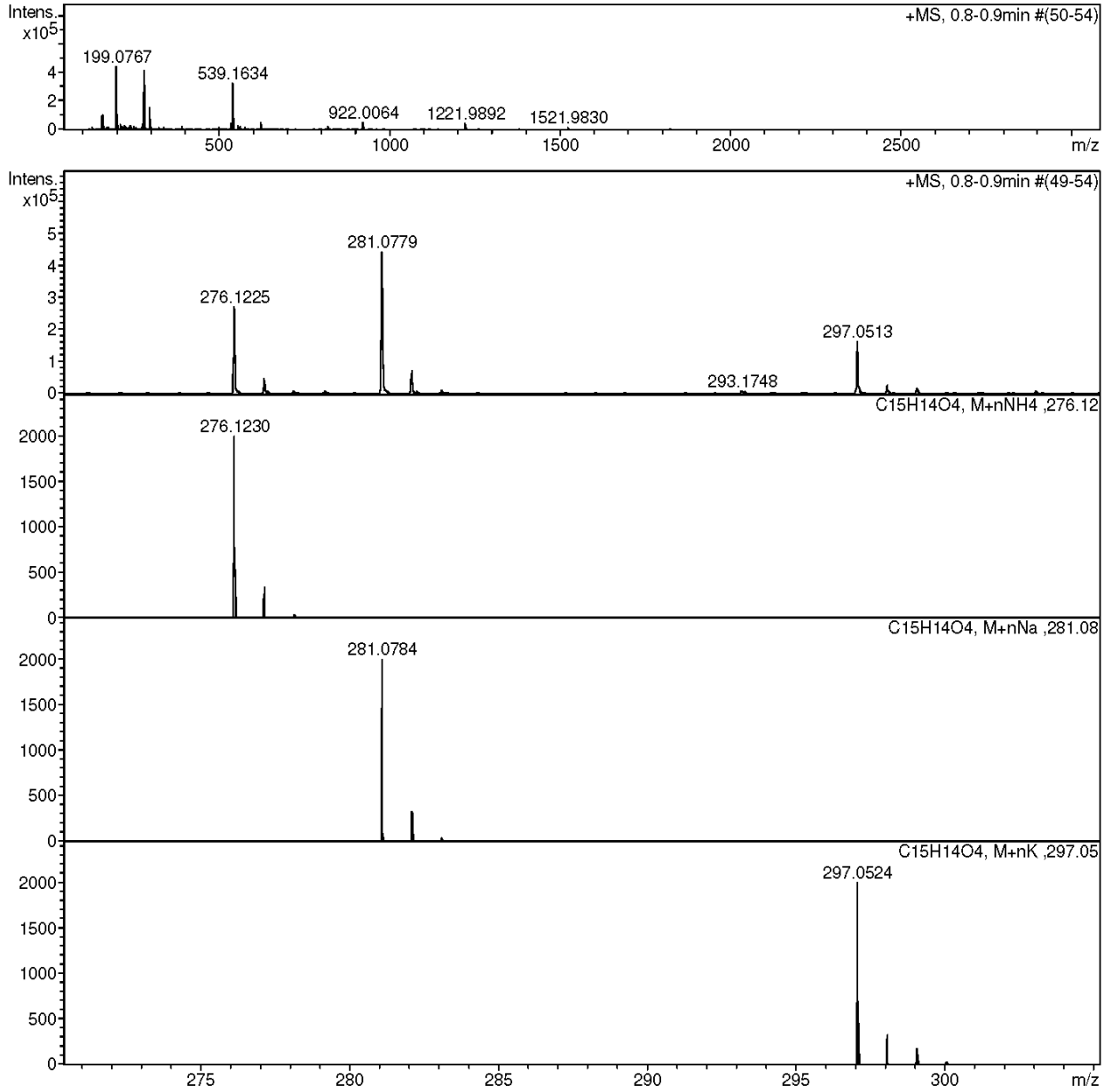
Display Report

Analysis Info

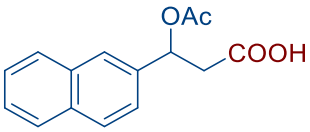
Analysis Name	D:\Data\Kolotyrkina\2023\Ustuzhanin\1128023.d	Acquisition Date	28.11.2023 15:30:37
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV MG359	Instrument / Ser#	micrOTOF 10248
Comment	C15H14O4 mH259.0964 calibrant added CH3OH		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of **3-Acetoxy-3-(naphthalen-2-yl)propanoic acid, 2j**



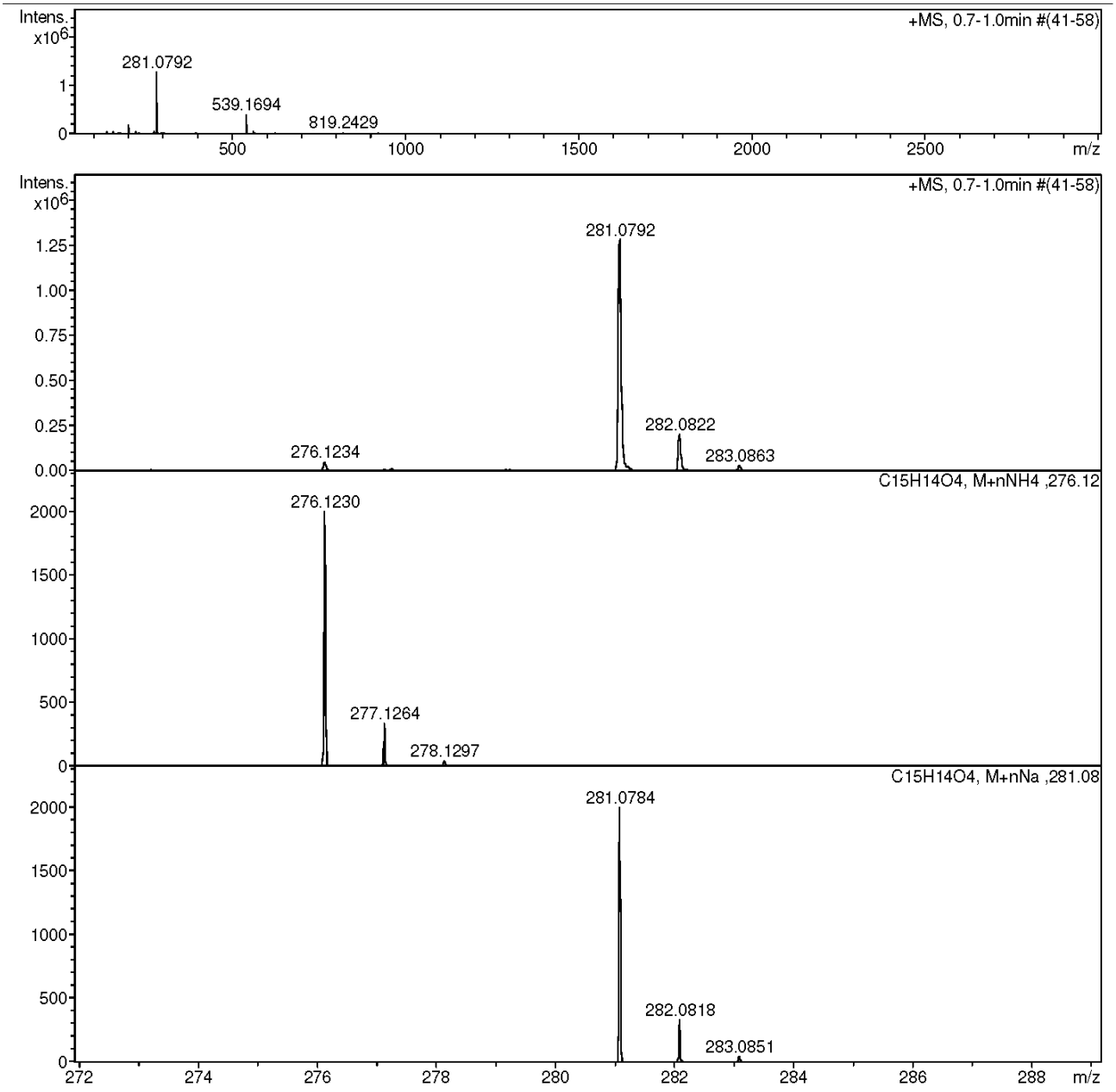
Display Report

Analysis Info

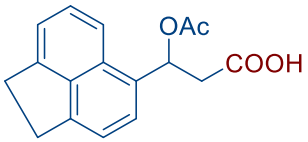
Analysis Name	D:\Data\Kolotyrkina\2023\Ustuzhanin\1130006.d	Acquisition Date	30.11.2023 10:30:30
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV MG278	Instrument / Ser#	micrOTOF 10248
Comment	C15H14O4 mH 259.0964 calibrant added CH3OH		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of **3-Acetoxy-3-(1,2-dihydroacenaphthylen-5-yl)propanoic acid, 2k**



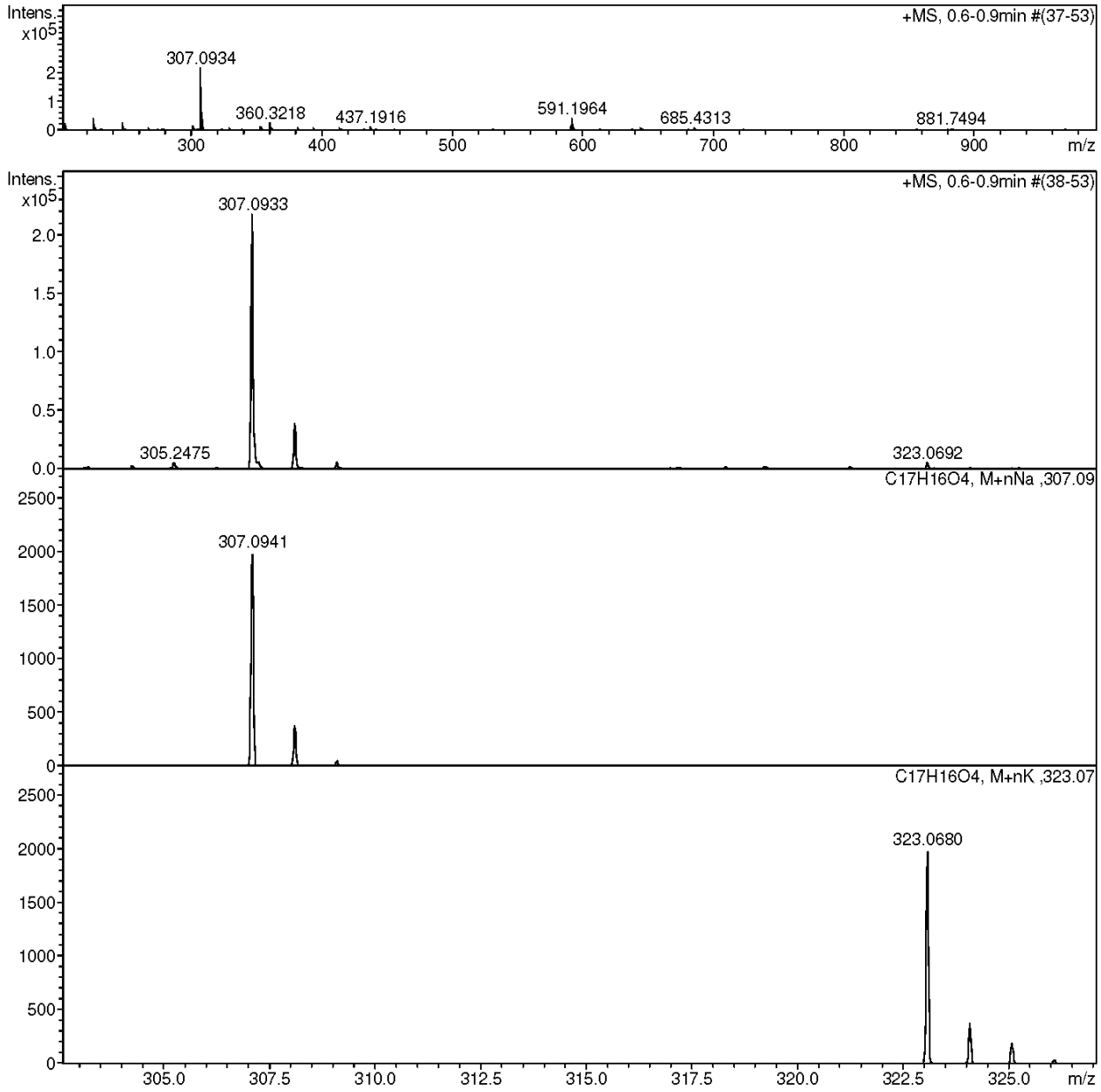
Display Report

Analysis Info

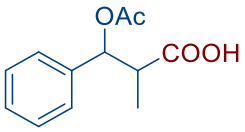
Analysis Name	D:\Data\Kolotyrkina\2023\Ustuzhanin\1122011.d	Acquisition Date	22.11.2023 12:45:35
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV MG-368	Instrument / Ser#	micrOTOF 10248
Comment	C17H16O4 mH285.1121 clb added CH3OH		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of **3-Acetoxy-2-methyl-3-phenylpropanoic acid, 2l**



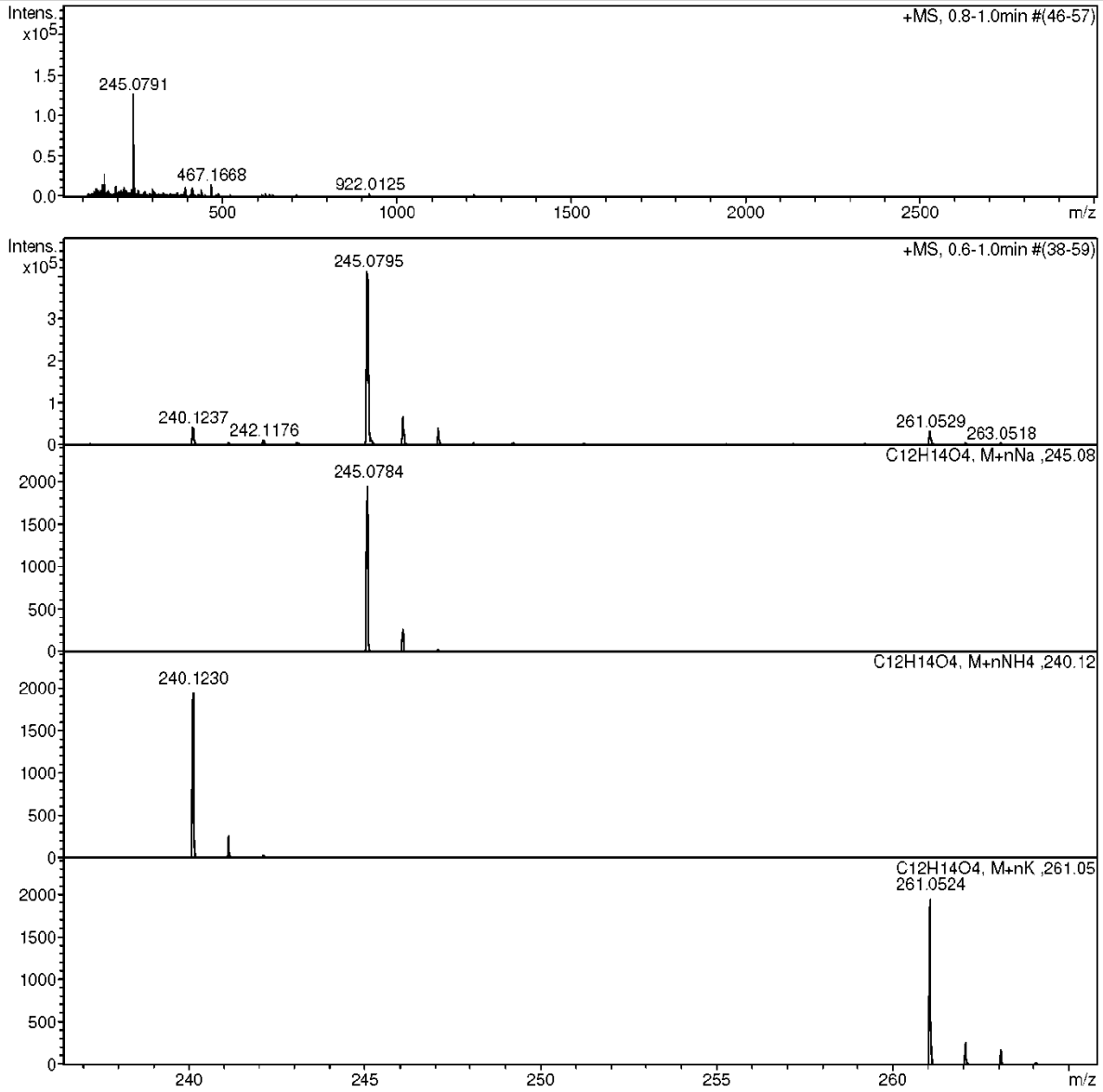
Display Report

Analysis Info

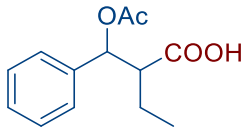
Analysis Name	D:\Data\Kolotyrkina\2023\Ustuzhanin\1214010.d	Acquisition Date	14.12.2023 13:05:59
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV MG295	Instrument / Ser#	micrOTOF 10248
Comment	C12H14O4 mH223.0964 clb added CH3OH		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of **2-(Acetoxy(phenyl)methyl)butanoic acid, 2m**



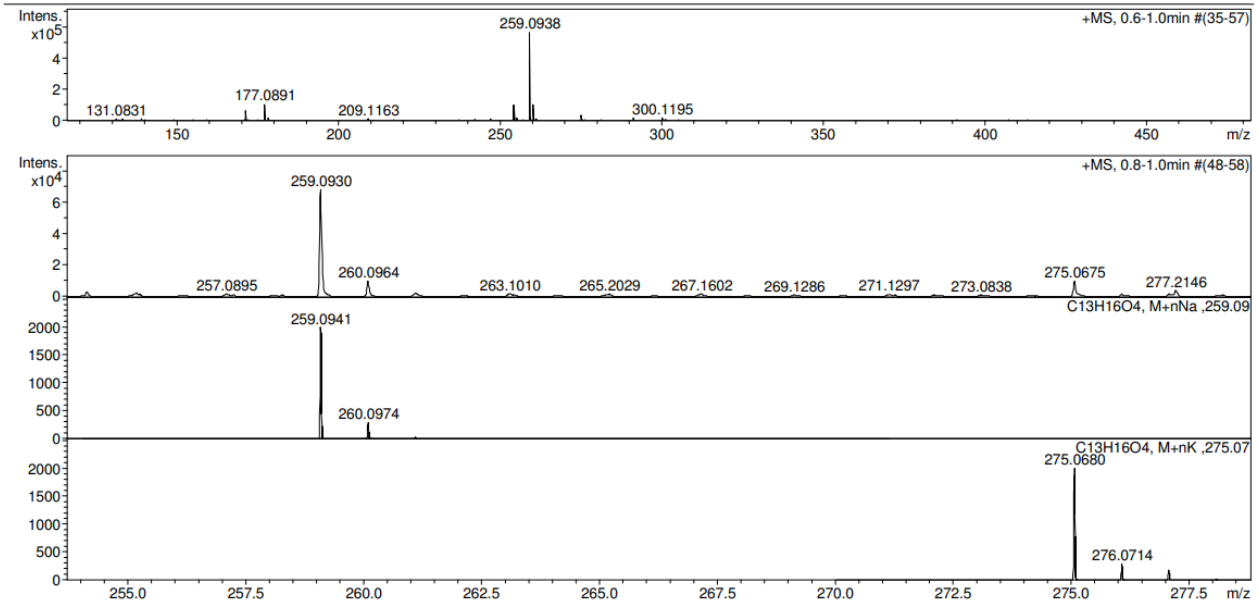
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2023\Ustuzhanin\1130026.d	Acquisition Date	30.11.2023 15:02:03
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV MG366	Instrument / Ser#	micrOTOF 10248
Comment	C13H16O4 mH 237.1121 calibrant added CH3OH		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



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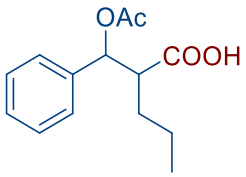
HRMS spectrum of **2-(Acetoxy(phenyl)methyl)pentanoic acid, 2n**



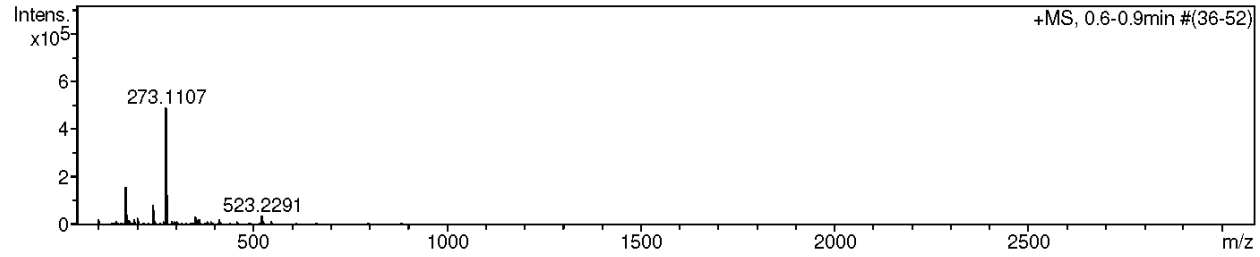
Display Report

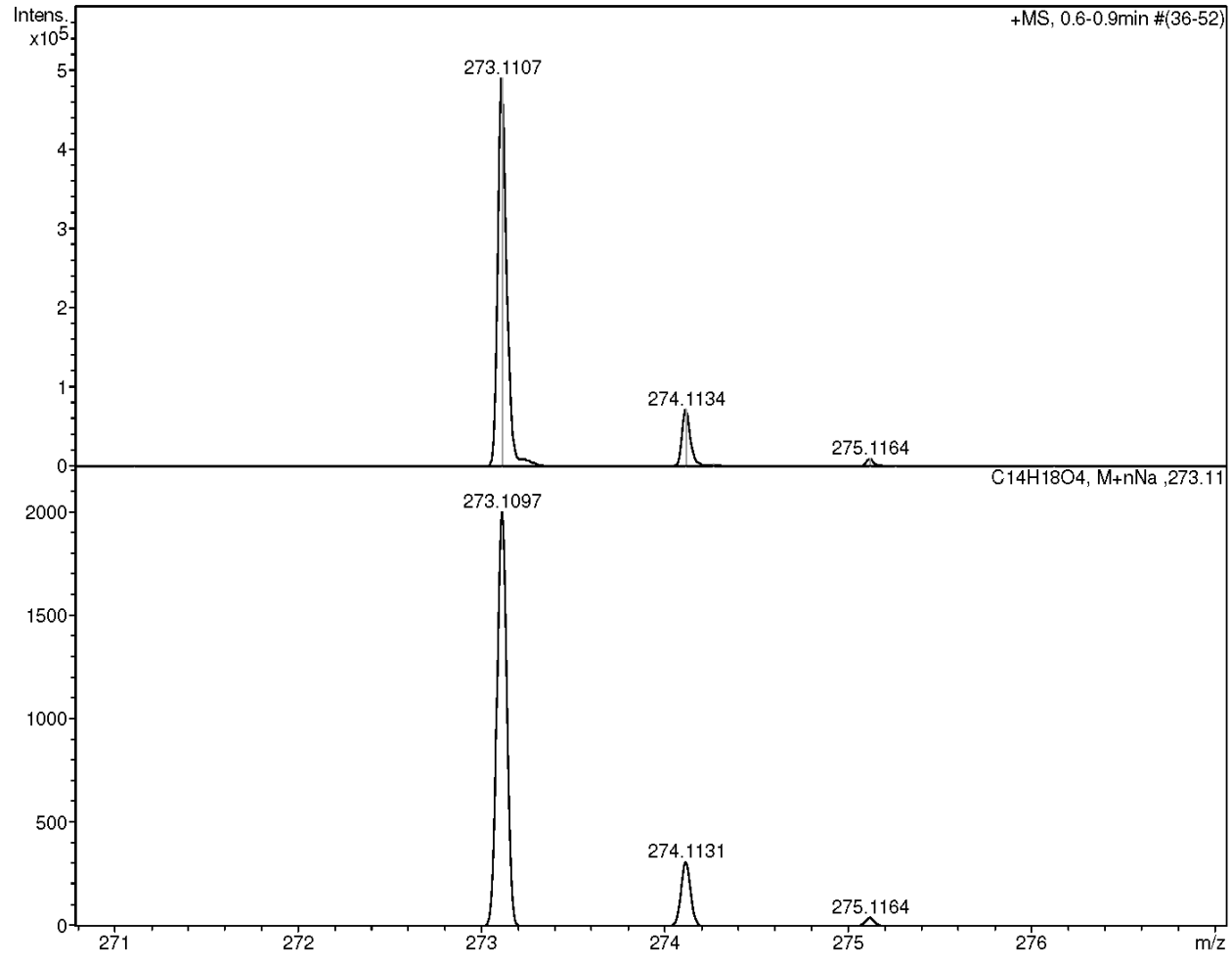
Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2023\Ustuzhanin\1222002.d	Acquisition Date	22.12.2023 10:20:10
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV MG398	Instrument / Ser#	micrOTOF 10248
Comment	C14H18O4 mH 251.1277 clb added CH3OH		

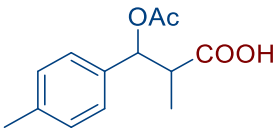
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste





HRMS spectrum of **3-Acetoxy-2-methyl-3-(p-tolyl)propanoic acid, 2o**



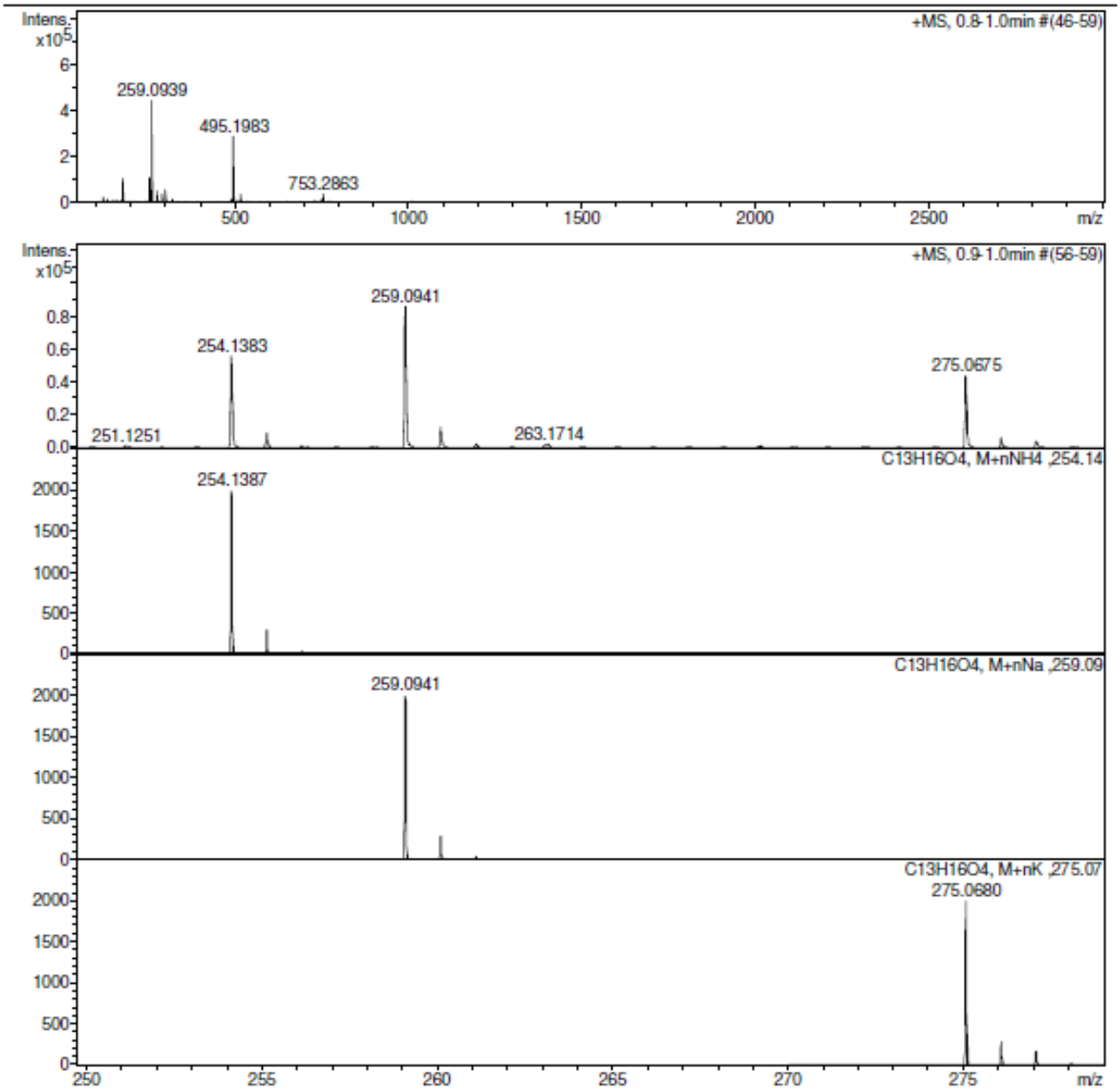
Display Report

Analysis Info

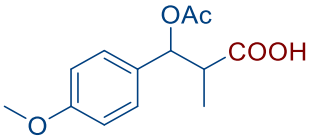
Analysis Name	D:\Data\Kolotyrkina\2024\Ustuzhanin\0306002.d	Acquisition Date	06.03.2024 11:00:38
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV MG445	Instrument / Ser#	micrOTOF 10248
Comment	C13H16O4 mH237.1121 calibrant added CH3OH		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of **3-Acetoxy-3-(4-methoxyphenyl)-2-methylpropanoic acid, 2p**



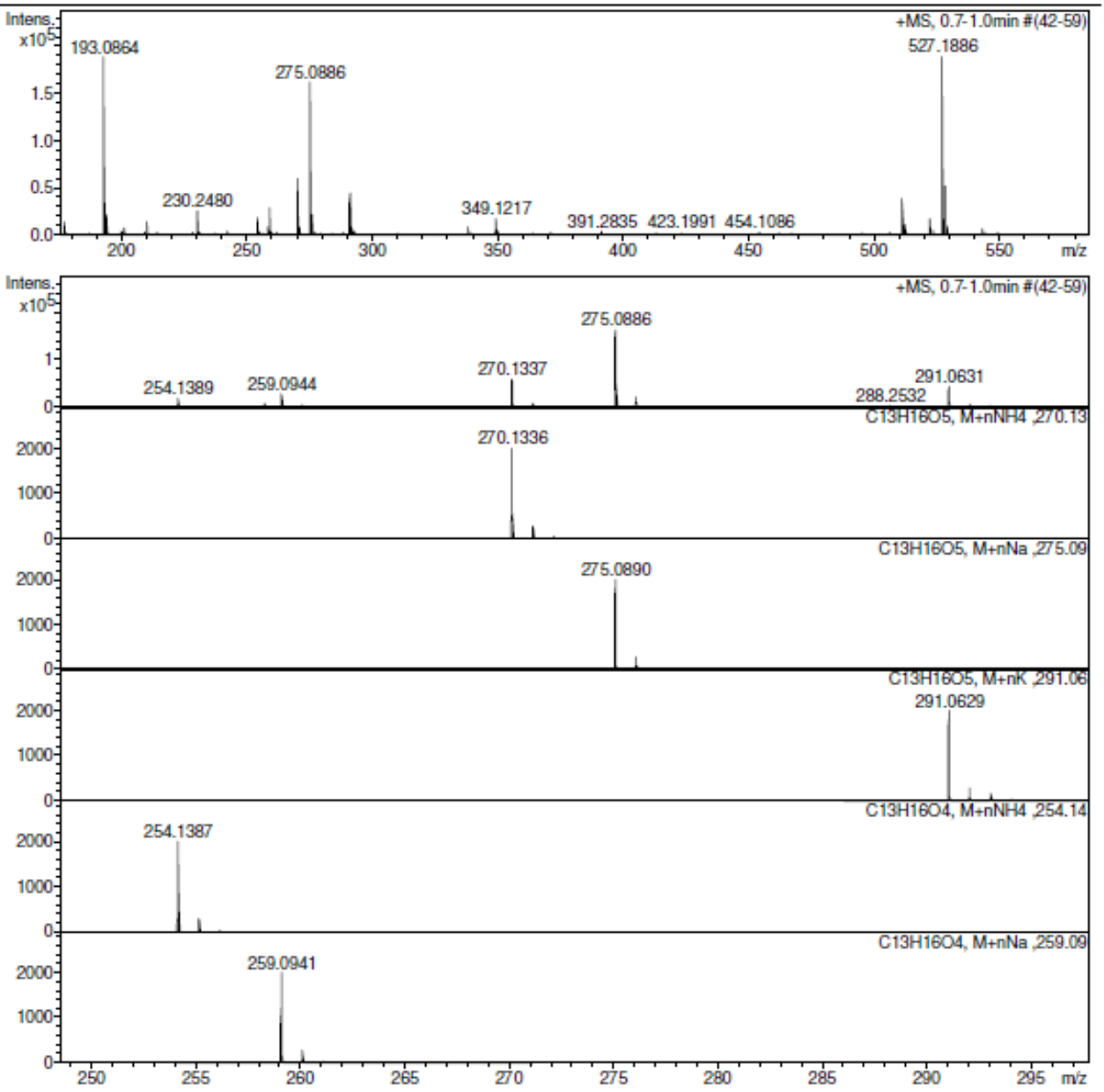
Display Report

Analysis Info

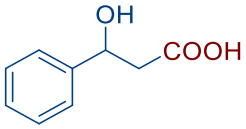
Analysis Name	D:\Data\Kolotyrkina\2024\Ustuzhanin\0320004.d	Acquisition Date	20.03.2024 9:52:46
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV MG464	Instrument / Ser#	micrOTOF 10248
Comment	C13H16O5 mH253.1070 calibrant added CH3OH		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of **3-Hydroxy-3-phenylpropanoic acid, 3a**



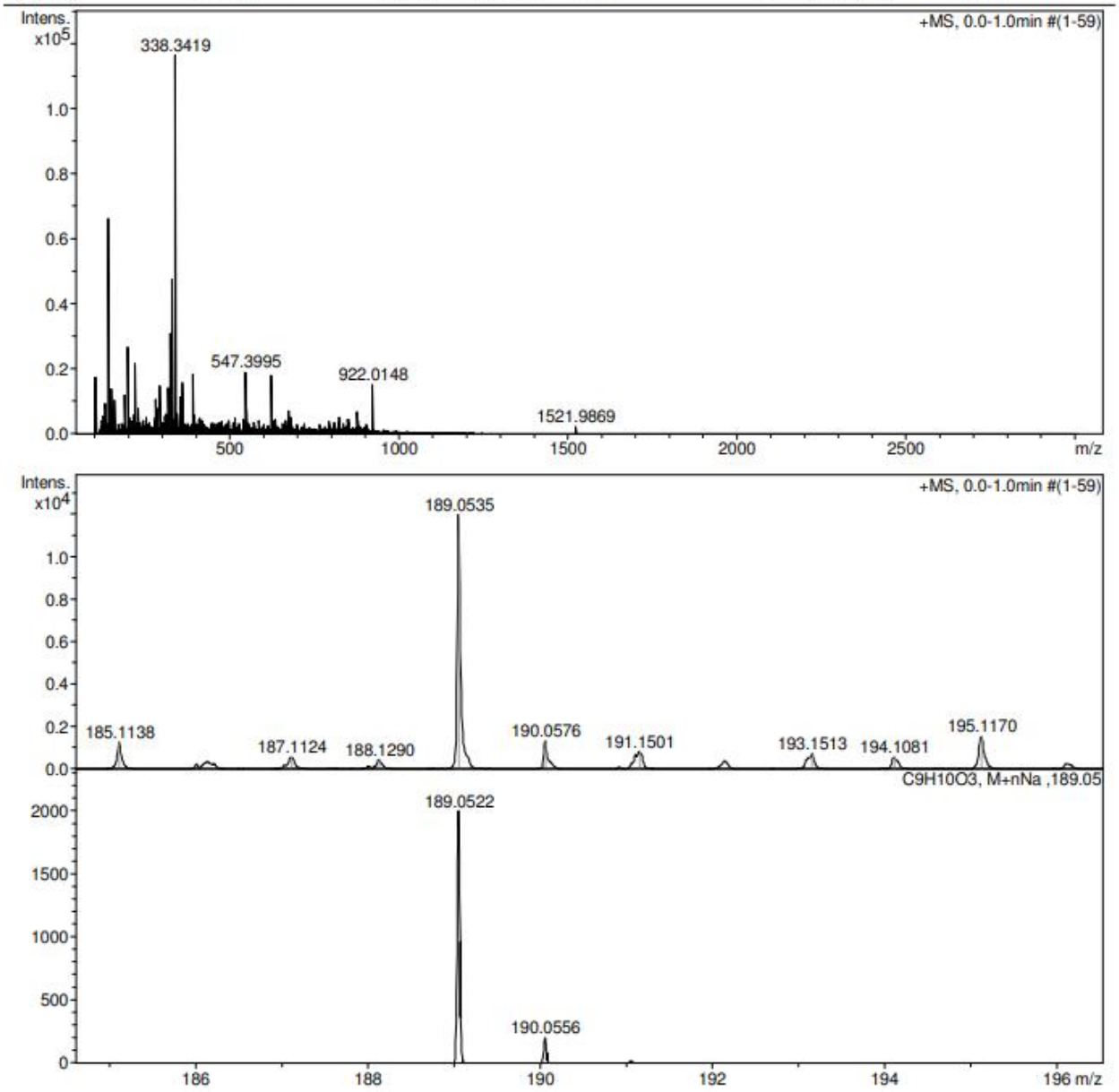
Display Report

Analysis Info

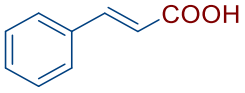
Analysis Name	D:\Data\Chizhov\Terentiev\Ustyuzhanin\mg296_&clblow.d	Acquisition Date	16.02.2024 16:44:52
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV MG296	Instrument / Ser#	micrOTOF 10248
Comment	CH3CN 100 %, dil. 20, calibrant added		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



HRMS spectrum of **3-Phenylacrylic acid, 4a**



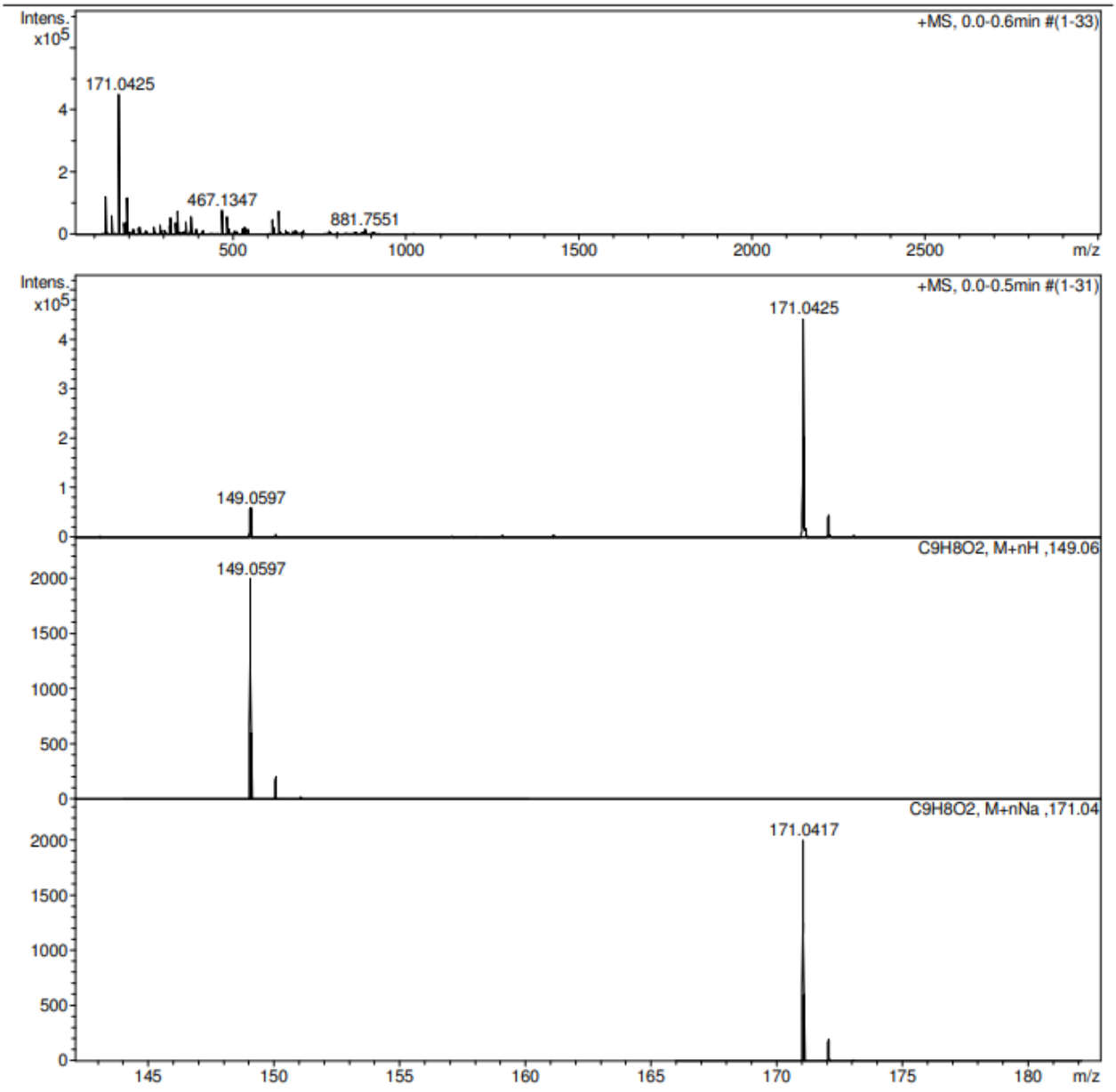
Display Report

Analysis Info

Analysis Name	D:\Data\Kolotyrkina\2024\Ustuzhanin\0423046.d	Acquisition Date	23.04.2024 17:05:54
Method	tune_low.m	Operator	BDAL@DE
Sample Name	/VILV MG486-3	Instrument / Ser#	micrOTOF 10248
Comment	C9H8O2 mH149.0597 calibrant added CH3CN		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



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