Supporting Information for

Expeditious access to *cis*-β-aryl, γ-alkyl disubstituted (±)-γ-butyrolactones *via* Nickel-hydride catalysis

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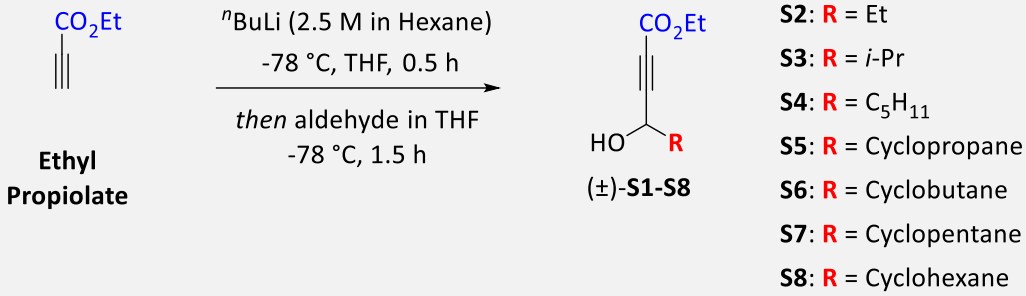
General Methods

All reagents and starting materials were obtained from commercial sources and used as received. All solvents were analytical grade and used as purchased. Reactions were performed under nitrogen atmosphere, using a balloon filled with nitrogen. All reactions performed at elevated temperatures were heated using laboratory heating blocks. Brine refers to a saturated aqueous solution of sodium chloride. Flash column chromatography was performed using silica gel 60Å (35−70 μm). Merck Kieselgel aluminium-backed plates precoated with silica gel 60Å F254 were used for thin-layer chromatography (TLC) and were visualised by ultraviolet light (254 nm) and/or heating the plate after staining with vanillin or potassium permanganate ($KMnO_4$). The ^1H NMR spectra were recorded on Bruker Avance III HD 500 (500 MHz) or Avance III HD 400 (400 MHz) ultrashield™ spectrometers and data are reported as follows: chemical shift (δ_H) in ppm relative to tetramethylsilane as the internal standard ($CDCl_3$, δ 7.26 ppm; DMSO-d_6 δ 2.50 ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or overlap of non-equivalent resonances), and integration. The ^{13}C NMR spectra were recorded on a NMR spectrometer at either 101 or 126 MHz and data are reported as follows: chemical shift (δ_C) in ppm relative to tetramethylsilane or the solvent as an internal standard ($CDCl_3$, δ 77.16 ppm; DMSO-d_6 δ 39.5 ppm). The multiplicity of each carbon with respect to hydrogen was deduced from DEPT (Distortionless Enhancement by Polarization Transfer) 135 experiments, which determined whether they are C, CH, CH_2, or CH_3. All the ^{13}C and ^{19}F NMR were ran and reported as hydrogen decoupled. The infrared spectra were recorded on a Perkin Elmer FT-IR spectrometer using neat samples; Wavelengths of maximum absorbance (ν_{max}) are quoted in wavenumbers (cm^{-1}). Only selected, characteristic IR absorption data are provided for each compound. The mass spectra were recorded using electron impact or electrospray techniques. The High-resolution mass spectra (HRMS) were recorded by the University of Glasgow school of chemistry mass spectrometry service on Agilent 6546 LC/Q-TOF using electrospray ionisation (ESI). The parent ion $[M]^+$, $[M+H]^+$ or $[M+Na]^+$ is calculated to 4 decimal places from the molecular formula, and all values are within a tolerance of 5 ppm. Melting points were measured on Büchi melting point B-545 apparatus and are uncorrected.

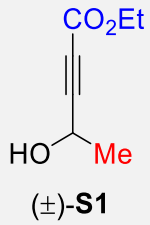
Synthesis of Alkynoates (±)-S1-S9



General experimental procedure for the synthesis of alkynoates (±)-S1-S8:

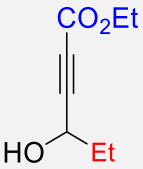
^{n}BuLi (2.5 M in Hexanes, 36.69 mmol, 14.68 mL, 1.2 eq.) was added to a cooled stirring solution of ethyl propiolate (3.0 g, 30.58 mmol, 1.0 eq.) in THF (200 mL) at -78˚C in a pre-flame dried 500 mL round bottom flask under the inert atmosphere of nitrogen. The resulting light-yellow solution was stirred at -78 ˚C for 30 minutes. Afterwards, a solution of the corresponding aldehyde (14.68 mmol, 1.2 eq.) in THF (30 mL) was added, slowly, at -78 ˚C. The resulting mixture was stirred at -78 ˚C for 1.5 h, the cooling bath was removed, and the mixture was poured into a separating funnel which contains saturated aqueous solution of ammonium chloride (100 mL). The aqueous layer was extracted with EtOAc (60 mL). The combined organic layers were washed with brine (70 mL), dried with $MgSO_4$, filtered, and solvent removed *in vacuo*. Purification by silica gel chromatography using 5-20% EtOAc in hexanes furnished the alkynoates.

Ethyl 4-hydroxy-2-pentynoate (±)-S1



The general procedure for the synthesis of alkynoates was followed as described using ^{n}BuLi (2.5 M in Hexanes, 36.69 mmol, 14.68 mL, 1.2 eq.), ethyl propiolate (3.0 g, 30.58 mmol, 1.0 eq.) in THF (200 mL) and acetaldehyde (0.65 g, 14.68 mmol, 1.2 eq.) in THF (30 mL). Compound (±)-**S1** was obtained as colourless oil (2.96 g, 20.79 mmol, 68% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.63 (qd, *J* = 6.7, 5.6 Hz, 1H), 4.23 (q, *J* = 7.1 Hz, 2H), 2.48 (d, *J* = 5.6 Hz, 1H, OH), 1.50 (d, *J* = 6.7 Hz, 3H), 1.30 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 153.5, 88.4, 75.9, 62.3, 58.1, 23.4, 14.1. The data obtained agrees with literature data.[1a]

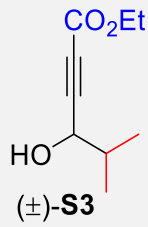
Ethyl 4-hydroxy-2-hexynoate (±)-**S2**



(±)-**S2**

The general procedure for the synthesis of alkynoates was followed as described using ^{n}BuLi (2.5 M in Hexanes, 36.69 mmol, 14.68 mL, 1.2 eq.), ethyl propiolate (3.0 g, 30.58 mmol, 1.0 eq.) in THF (200 mL) and propionaldehyde (0.85 g, 14.68 mmol, 1.2 eq.) in THF (30 mL). Compound (±)-**S2** was obtained as light-yellow oil (3.39 g, 21.71 mmol, 71% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.44 (q, *J* = 6.3 Hz, 1H), 4.24 (q, *J* = 7.1 Hz, 2H), 1.97 (d, *J* = 5.9 Hz, 1H, OH), 1.80 (qd, *J* = 7.4, 6.4 Hz, 2H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.04 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 153.5, 87.6, 76.7, 63.4, 62.3, 30.1, 14.1, 9.4. The data obtained agrees with literature data.[1b]

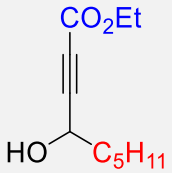
Ethyl 4-hydroxy-5-methyl-2-hexynoate (±)-**S3**



(±)-**S3**

The general procedure for the synthesis of alkynoates was followed as described using ^{n}BuLi (2.5 M in Hexanes, 36.69 mmol, 14.68 mL, 1.2 eq.), ethyl propiolate (3.0 g, 30.58 mmol, 1.0 eq.) in THF (200 mL) and isobutyraldehyde (1.06 g, 14.68 mmol, 1.2 eq.) in THF (30 mL). Compound (±)-**S3** was obtained as light-red oil (3.75 g, 22.02 mmol, 72% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.28 (t, *J* = 5.9 Hz, 1H), 4.24 (q, *J* = 7.2 Hz, 2H), 2.05 (br s, 1H, OH), 1.96 (pd, *J* = 6.8, 5.7 Hz, 1H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.03 (t, *J* = 6.9 Hz, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 153.5, 86.8, 77.5, 67.7, 62.2, 34.3, 18.0, 17.6, 14.1. The data obtained agrees with literature data.[2a]

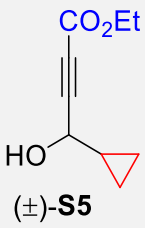
Ethyl 4-hydroxy-2-nonynoate (±)-**S4**



(±)-**S4**

The general procedure for the synthesis of alkynoates was followed as described using ^{n}BuLi (2.5 M in Hexanes, 36.69 mmol, 14.68 mL, 1.2 eq.), ethyl propiolate (3.0 g, 30.58 mmol, 1.0 eq.) in THF (200 mL) and hexanal (1.47 g, 14.68 mmol, 1.2 eq.) in THF (30 mL). Compound (±)-**S4** was obtained as colourless oil (4.60 g, 23.24 mmol, 76% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.48 (td, *J* = 6.6, 5.8 Hz, 1H), 4.24 (q, *J* = 7.1 Hz, 2H), 2.01 (d, *J* = 5.9 Hz, 1H, OH), 1.72-1.80 (m, 2H), 1.42-1.52 (m, 2H), 1.28-1.35 (m, 7H), 0.85-0.93 (m, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 153.5, 87.8, 76.7, 62.3, 62.2, 37.0, 31.4, 24.7, 22.6, 14.14, 14.10. The data obtained agrees with literature data.[2b]

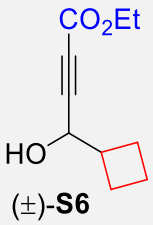
Ethyl 4-cyclopropyl-4-hydroxy-2-butynoate (±)-**S5**



(±)-**S5**

The general procedure for the synthesis of alkynoates was followed as described using ^{n}BuLi (2.5 M in Hexanes, 36.69 mmol, 14.68 mL, 1.2 eq.), ethyl propiolate (3.0 g, 30.58 mmol, 1.0 eq.) in THF (200 mL) and cyclopropanecarboxyaldehyde (1.02 g, 14.68 mmol, 1.2 eq.) in THF (30 mL). Compound (±)-**S5** was obtained as light-red oil (3.08 g, 18.35 mmol, 60% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.18-4.28 (m, 3H), 2.16 (d, *J* = 6.3 Hz, 1H, OH), 1.26-1.35 (m, 4H), 0.55-0.68 (m, 2H), 0.43-0.52 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 153.4, 85.9, 76.6, 65.6, 62.3, 16.8, 14.1, 3.5, 2.0. **IR** (neat) V_{max} (cm^{-1}): 3399, 2234, 1708, 1242. **HRMS** (ESI): calc for $C_9H_{12}O_3$ m/z: 168.0786 found $[M+H]^+$ 169.0860.

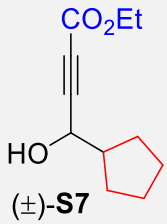
Ethyl 4-cyclobutyl-4-hydroxy-2-butynoate (±)-**S6**



(±)-**S6**

The general procedure for the synthesis of alkynoates was followed as described using ^{n}BuLi (2.5 M in Hexanes, 36.69 mmol, 14.68 mL, 1.2 eq.), ethyl propiolate (3.0 g, 30.58 mmol, 1.0 eq.) in THF (200 mL) and cyclobutanecarboxyaldehyde (1.23 g, 14.68 mmol, 1.2 eq.) in THF (30 mL). Compound (±)-**S6** was obtained as light-brown oil (3.06 g, 16.82 mmol, 55% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.42 (dd, *J* = 7.1, 6.1 Hz, 1H), 4.24 (q, *J* = 7.1 Hz, 2H), 2.58-2.70 (m, 1H), 2.01-2.14 (m, 2H), 1.99 (d, *J* = 6.2 Hz, 1H, OH), 1.90-1.97 (m, 3H), 1.83-1.89 (m, 1H), 1.31 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 153.5, 86.7, 77.0, 65.7, 62.3, 40.2, 24.1, 23.5, 17.8, 14.1. **IR** (neat) V_{max} (cm^{-1}): 3398, 2235, 1709, 1245. **HRMS** (ESI): calc for $C_{10}H_{14}O_3$ m/z: 182.0943 found $[M+H]^+$ 183.1017.

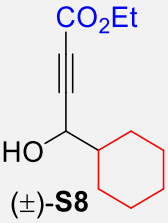
Ethyl 4-cyclopentyl-4-hydroxy-2-butynoate (±)-**S7**



(±)-**S7**

The general procedure for the synthesis of alkynoates was followed as described using ^{n}BuLi (2.5 M in Hexanes, 36.69 mmol, 14.68 mL, 1.2 eq.), ethyl propiolate (3.0 g, 30.58 mmol, 1.0 eq.) in THF (200 mL) and cyclopentanecarboxyaldehyde (1.44 g, 14.68 mmol, 1.2 eq.) in THF (30 mL). Compound (±)-**S7** was obtained as colourless oil (3.60 g, 18.35 mmol, 60% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.34 (dd, *J* = 7.2, 6.0 Hz, 1H), 4.23 (q, *J* = 7.1 Hz, 2H), 2.24 (h, *J* = 7.9 Hz, 1H), 2.01 (d, *J* = 6.1 Hz, 1H, OH), 1.75-1.87 (m, 2H), 1.60-1.69 (m, 2H), 1.53-1.59 (m, 2H), 1.39-1.51 (m, 2H), 1.31 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 153.5, 87.5, 77.3, 66.1, 62.2, 45.7, 28.8, 28.5, 25.75, 25.70, 14.1. **IR** (neat) V_{max} (cm^{-1}): 3397, 2954, 2236, 1710, 1242. **HRMS** (ESI): calc for $C_{11}H_{16}O_3$ m/z: 196.1099 found $[M+H]^+$ 197.1173.

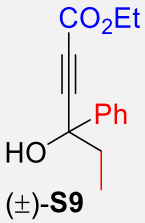
Ethyl 4-cyclohexyl-4-hydroxy-2-butynoate (±)-**S8**



(±)-**S8**

The general procedure for the synthesis of alkynoates was followed as described using ^{n}BuLi (2.5 M in Hexanes, 36.69 mmol, 14.68 mL, 1.2 eq.), ethyl propiolate (3.0 g, 30.58 mmol, 1.0 eq.) in THF (200 mL) and cyclohexanecarboxyaldehyde (0.65 g, 14.68 mmol, 1.2 eq.) in THF (30 mL). Compound (±)-**S8** was obtained as light-brown oil (2.96 g, 20.79 mmol, 68% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.22-4.29 (m, 3H), 1.84-1.89 (m, 3H), 1.74-1.82 (m, 2H), 1.58-1.72 (m, 2H), 1.32 (t, *J* = 7.1 Hz, 3H), 1.18-1.29 (m, 3H), 1.06-1.18 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 153.5, 87.2, 77.5, 67.0, 62.2, 43.7, 28.4, 28.2, 26.2, 25.87, 25.86, 14.1. The data obtained agrees with literature data.[3a]

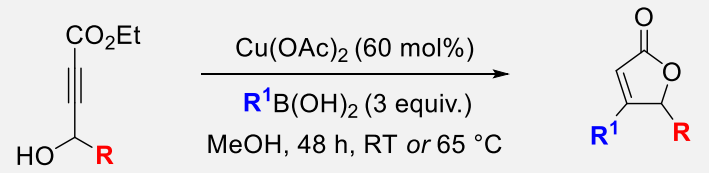
Ethyl 4-hydroxy-4-phenyl-2-hexynoate (±)-**S9**



(±)-**S9**

The general procedure for the synthesis of alkynoates was followed as described using ^{n}BuLi (2.5 M in Hexanes, 36.69 mmol, 14.68 mL, 1.2 eq.), ethyl propiolate (3.0 g, 30.58 mmol, 1.0 eq.) in THF (200 mL) and propiophenone (1.97 g, 14.68 mmol, 1.2 eq.) in THF (30 mL). Compound (±)-**S9** was obtained as colourless oil (3.76 g, 16.21 mmol, 53% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.48-7.54 (m, 2H), 7.28-7.34 (m, 2H), 7.21-7.27 (m, 1H), 4.19 (q, *J* = 7.1 Hz, 2H), 2.54 (s, 1H, OH), 1.85-2.04 (m, 2H), 1.26 (t, *J* = 7.1 Hz, 3H), 0.90 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 153.5, 142.7, 128.5, 128.3, 125.4, 88.7, 77.7, 73.9, 62.3, 37.9, 14.1, 8.9. The data obtained agrees with literature data.[3b]

Synthesis of β- and γ- disubstituted, α,β-unsaturated lactones (±)-11a-11w



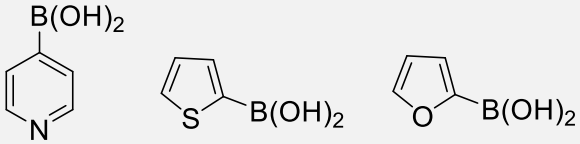
(±)-**S1-S8** (±)-**11a-11x**

General experimental procedure for the synthesis of butenolides (±)-11a-11w:

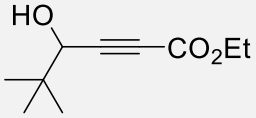
A solution of alkynoate (5.50 mmol, 1.0 eq.) in MeOH (10 mL) was added to the stirring blue pre-mixed solution of $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and aryl boronic acid (16.5 mmol, 3.0 eq.) in MeOH (30 mL). The resulting mixture was stirred at room temperature* (unless stated otherwise e.g. at 65 ˚C) for 48 h. Afterwards, the mixture was diluted with EtOAc (50 mL), and then added into a separating funnel which contains saturated aqueous solution of ammonium chloride (30 mL). The aqueous layer was further extracted with EtOAc (20 mL). The combined organic layers were washed with de-ionised water (20 mL), brine (45 mL), dried with $MgSO_4$, filtered, and concentrated *in vacuo*. Purification by silica gel chromatography using 5-10% EtOAc in hexanes furnished β- and γ- disubstituted, α,β-unsaturated lactones.

Note: The reaction works well either under the atmosphere of nitrogen or air. The reaction doesn't work if crude alkynoate was employed. In most cases, the butenolides have the same R_f as the alkynoates and very difficult to separate by silica gel chromatography. However, it was noted that all the alkynoates were fully consumed within 48 h, although some might not need 48 h reaction time. Hetero aryl boronic acids failed to perform in this reaction (either at RT or 65˚C), alkynoates were recovered in all cases. *Room temperature refers to the laboratory/fumehood temperature which was between 15 ˚C–18 ˚C during Scottish winter.

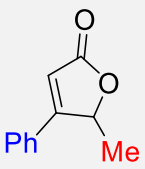
Failed boronic acids



Failed Alkynoate



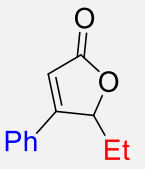
5-Methyl-4-phenyl-2(5*H*)-furanone (±)-**11a**



(±)-**11a**

The general procedure for the synthesis of butenolides was followed as described using (±)-**S1** (0.78 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and phenyl boronic acid (2.01 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11a** as a colourless oil (0.77 g, 4.42 mmol, 80% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.45–7.51 (m, 5H), 6.27 (d, *J* = 1.4 Hz, 1H), 5.56 (qd, *J* = 6.8, 1.5 Hz, 1H), 1.53 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.7, 169.0, 131.4, 130.0, 129.3, 127.3, 113.8, 78.7, 19.9. The data obtained agrees with literature data.[4a]

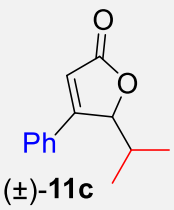
5-Ethyl-4-phenyl-2(5*H*)-furanone (±)-**11b**



(±)-**11b**

The general procedure for the synthesis of butenolides was followed as described using (±)-**S2** (0.86 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and phenyl boronic acid (2.01 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11b** as a colourless oil (0.81 g, 4.29 mmol, 78% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.44–7.50 (m, 5H), 6.28 (d, *J* = 1.5 Hz, 1H), 5.50 (ddd, *J* = 6.9, 3.4, 1.5 Hz, 1H), 2.10 (dqd, *J* = 14.8, 7.4, 3.4 Hz, 1H), 1.66 (dq, *J* = 14.8, 7.4 Hz, 1H), 0.92 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.0, 167.5, 131.3, 130.3, 129.3, 127.2, 114.8, 83.1, 26.4, 8.3. The data obtained agrees with literature data.[4b]

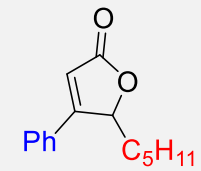
5-(1-Methylethyl)-4-phenyl-2(5*H*)-furanone (±)-**11c**



(±)-**11c**

The general procedure for the synthesis of butenolides was followed as described using (±)-**S3** (0.94 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and phenyl boronic acid (2.01 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11c** as a colourless oil (0.91 g, 4.51 mmol, 82% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.41–7.54 (m, 5H), 6.27 (d, *J* = 1.5 Hz, 1H), 5.44 (dd, *J* = 2.5, 1.6 Hz, 1H), 2.17 (pd, *J* = 6.9, 2.5 Hz, 1H), 1.23 (d, *J* = 6.9 Hz, 3H), 0.63 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.3, 167.4, 131.3, 130.6, 129.3, 127.2, 115.1, 86.4, 30.8, 20.3, 13.5. The data obtained agrees with literature data.[4c]

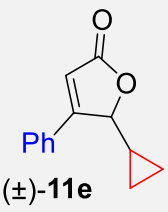
5-Pentyl-4-phenyl-2(5*H*)-furanone (±)-**11d**



(±)-**11d**

The general procedure for the synthesis of butenolides was followed as described using (±)-**S4** (1.09 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and phenyl boronic acid (2.01 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11d** as a colourless oil (0.86 g, 3.74 mmol, 68% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.43–7.51 (m, 5H), 6.27 (d, *J* = 1.5 Hz, 1H), 5.50 (ddd, *J* = 7.9, 3.1, 1.5 Hz, 1H), 1.93-2.04 (m, 1H), 1.51-1.64 (m, 1H), 1.35-1.50 (m, 2H), 1.20-1.33 (m, 4H), 0.78-0.87 (m, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.0, 167.9, 131.3, 130.3, 129.3, 127.2, 114.5, 82.4, 33.6, 31.4, 24.3, 22.5, 14.0. The data obtained agrees with literature data.[4d]

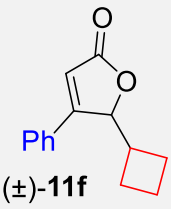
5-Cyclopropyl-4-phenyl-2(5*H*)-furanone (±)-**11e**



(±)-**11e**

The general procedure for the synthesis of butenolides was followed as described using (±)-**S5** (0.92 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and phenyl boronic acid (2.01 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11e** as a white solid (0.66 g, 3.30 mmol, 60% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.53–7.57 (m, 2H), 7.47–7.50 (m, 3H), 6.25 (d, *J* = 1.5 Hz, 1H), 5.19 (dd, *J* = 6.5, 1.5 Hz, 1H), 1.02-1.13 (m, 1H), 0.60-0.72 (m, 2H), 0.47-0.57 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.7, 168.2, 131.3, 130.5, 129.2, 127.6, 114.3, 84.0, 83.9, 14.0, 4.2, 1.8. **IR** (neat) V_{max} (cm^{-1}): 1718, 1316, 1264, 1031. **HRMS** (ESI): calc for $C_{13}H_{12}O_2$ m/z: 200.0837 found $[M+H]^+$ 201.0911. **MP**: 50-52˚C.

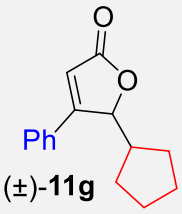
5-Cyclobutyl-4-phenyl-2(5*H*)-furanone (±)-**11f**



(±)-**11f**

The general procedure for the synthesis of butenolides was followed as described using (±)-**S6** (1.00 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and phenyl boronic acid (2.01 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11f** as a white solid (0.71 g, 3.30 mmol, 60% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40–7.52 (m, 5H), 6.24 (d, *J* = 1.4 Hz, 1H), 5.44 (dd, *J* = 3.5, 1.5 Hz, 1H), 2.77 (pd, *J* = 8.4, 3.4 Hz, 1H), 2.22-2.37 (m, 1H), 1.99-2.07 (m, 1H), 1.66-1.87 (m, 3H), 1.47-1.59 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.3, 166.6, 131.3, 130.5, 129.2, 127.2, 114.7, 83.4, 37.5, 24.6, 20.5, 17.8. **IR** (neat) V_{max} (cm^{-1}): 1725, 1307, 1269, 1169, 1033. **HRMS** (ESI): calc for $C_{14}H_{14}O_2$ m/z: 214.0994 found $[M+H]^+$ 215.1068. **MP**: 55-57˚C.

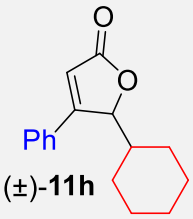
5-Cyclopentyl-4-phenyl-2(5*H*)-furanone (±)-**11g**



(±)-**11g**

The general procedure for the synthesis of butenolides was followed as described using (±)-**S7** (1.07 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and phenyl boronic acid (2.01 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11g** as a white solid (0.94 g, 4.13 mmol, 75% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.42–7.51 (m, 5H), 6.25 (d, *J* = 1.5 Hz, 1H), 5.60 (dd, *J* = 3.0, 1.5 Hz, 1H), 2.32 (pd, *J* = 8.5, 3.0 Hz, 1H), 1.75-1.91 (m, 2H), 1.62-1.72 (m, 1H), 1.53-1.62 (m, 1H), 1.46-1.52 (m, 1H), 1.34-1.42 (m, 1H), 1.13-1.23 (m, 1H), 1.00-1.11 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.4, 168.0, 131.2, 130.6, 129.3, 127.2, 115.0, 83.9, 41.6, 29.7, 25.8, 25.5, 23.8. **IR** (neat) V_{max} (cm^{-1}): 1728, 1314, 1268, 1170, 1034. **HRMS** (ESI): calc for $C_{15}H_{16}O_2$ m/z: 228.1150 found $[M+H]^+$ 229.1224. **MP**: 60-62 °C.

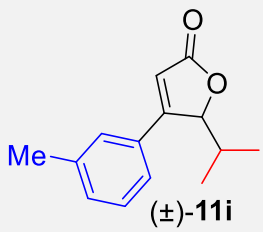
5-Cyclohexyl-4-phenyl-2(5*H*)-furanone (±)-**11h**



(±)-**11h**

The general procedure for the synthesis of butenolides was followed as described using (±)-**S8** (1.16 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and phenyl boronic acid (2.01 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11h** as a white solid (1.11 g, 4.57 mmol, 83% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.41-7.56 (m, 5H), 6.25 (d, *J* = 1.5 Hz, 1H), 5.39 (t, *J* = 1.9 Hz, 1H), 1.74-1.89 (m, 3H), 1.56-1.67 (m, 3H), 1.07-1.33 (m, 3H), 0.95-1.07 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.2, 167.1, 131.3, 130.7, 129.3, 127.2, 115.1, 86.3, 40.3, 30.6, 26.6, 25.9, 25.7, 23.8. The data obtained agrees with literature data.[4c]

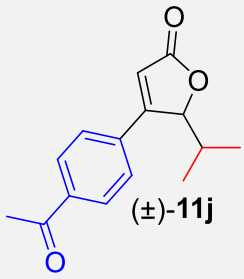
5-isopropyl-4-(*m*-tolyl)furan-2(5*H*)-one (±)-**11i**



(±)-**11i**

The general procedure for the synthesis of butenolides was followed as described using (±)-**S3** (0.94 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and *m*-tolyl boronic acid (2.24 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11i** as a colourless oil (0.83 g, 3.85 mmol, 70% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33-7.37 (m, 1H), 7.28-7.31 (m, 1H), 7.22-7.25 (m, 2H), 6.25 (d, *J* = 1.5 Hz, 1H), 5.42 (dd, *J* = 2.5, 1.5 Hz, 1H), 2.41 (s, 3H), 2.16 (pd, *J* = 6.9, 2.5 Hz, 1H), 1.23 (d, *J* = 6.9 Hz, 3H), 0.63 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.3, 167.6, 139.2, 132.1, 130.5, 129.2, 127.8, 124.3, 114.9, 86.4, 30.8, 21.5, 20.3, 13.5. **IR** (neat) V_{max} (cm^{-1}): 2926, 1727, 1449, 1174. **HRMS** (ESI): calc for $C_{14}H_{16}O_2$ m/z: 216.1150 found $[M+H]^+$ 217.1224.

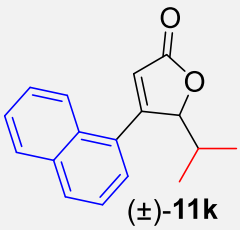
4-(4-acetylphenyl)-5-isopropylfuran-2(5*H*)-one (±)-**11j**



(±)-**11j**

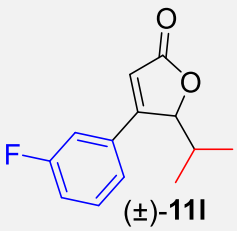
The general procedure for the synthesis of butenolides was followed as described using (±)-**S3** (0.94 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and 4-acetylphenylboronic acid (2.71 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11j** as a white solid (1.03 g, 4.24 mmol, 77% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.6 Hz, 2H), 7.55 (d, *J* = 8.6 Hz, 2H), 6.37 (d, *J* = 1.6 Hz, 1H), 5.46 (dd, *J* = 2.5, 1.6 Hz, 1H), 2.64 (s, 3H), 2.14 (pd, *J* = 6.9, 2.5 Hz, 1H), 1.24 (d, *J* = 6.9 Hz, 3H), 0.63 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.1, 172.6, 165.9, 138.9, 134.7, 129.2, 127.5, 117.2, 86.4, 30.7, 26.8, 20.2, 13.6. **IR** (neat) V_{max} (cm^{-1}): 2964, 1727, 1685, 1263, 1174. **HRMS** (ESI): calc for $C_{15}H_{16}O_3$ m/z: 244.1099 found $[M+H]^+$ 245.1173. **MP**: 85-87°C.

5-isopropyl-4-(naphthalen-1-yl)furan-2(5*H*)-one (±)-**11k**


(±)-**11k**

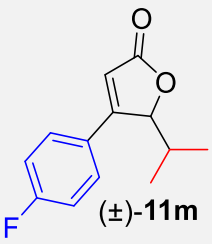
The general procedure for the synthesis of butenolides was followed as described using (±)-**S3** (0.94 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and Naphthalene-1-boronic acid (2.84 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11k** as a white solid (1.19 g, 4.73 mmol, 86% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.87-7.94 (m, 4H), 7.52-7.61 (m, 3H), 6.39 (d, *J* = 1.5 Hz, 1H), 5.57 (dd, *J* = 2.5, 1.5 Hz, 1H), 2.26 (pd, *J* = 6.9, 2.5 Hz, 1H), 1.28 (d, *J* = 6.9 Hz, 3H), 0.65 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.3, 167.2, 134.5, 133.0, 129.3, 128.8, 128.08, 128.04, 127.9, 127.35, 127.30, 124.1, 115.4, 86.5, 31.0, 20.4, 13.6. **IR** (neat) V_{max} (cm^{-1}): 2966, 1727, 1464, 1180. **HRMS** (ESI): calc for $C_{17}H_{16}O_2$ m/z: 252.1150 found $[M+H]^+$ 253.1224. **MP**: 95-97˚C.

4-(3-fluorophenyl)-5-isopropylfuran-2(5*H*)-one (±)-**11l**


(±)-**11l**

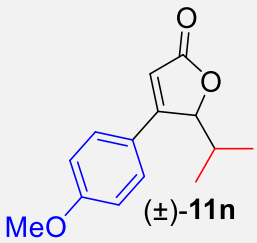
The general procedure for the synthesis of butenolides was followed as described using (±)-**S3** (0.94 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and 3-fluorophenyl boronic acid (2.31 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL) at 65 ˚C. Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11l** as a white solid (0.73 g, 3.30 mmol, 60% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.43-7.48 (m, 1H), 7.23-7.25 (m, 1H), 7.13-7.22 (m, 2H), 6.29 (d, *J* = 1.5 Hz, 1H), 5.40 (dd, *J* = 2.5, 1.5 Hz, 1H), 2.15 (pd, *J* = 6.9, 2.5 Hz, 1H), 1.24 (d, *J* = 6.9 Hz, 3H), 0.64 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.7, 165.9 (d, J_{C-F} = 2.5 Hz, 1C), 163.1 (d, J_{C-F} = 248.5 Hz, 1C), 132.6 (d, J_{C-F} = 7.8 Hz, 1C), 131.1 (d, J_{C-F} = 8.3 Hz, 1C), 122.9 (d, J_{C-F} = 3.1 Hz, 1C), 118.2 (d, J_{C-F} = 21.2 Hz, 1C), 116.4, 114.2 (d, J_{C-F} = 22.5 Hz, 1C), 86.3, 30.7, 20.2, 13.6. **^{19}F NMR** (377 MHz, $CDCl_3$) δ -110.94. **IR** (neat) V_{max} (cm^{-1}): 2968, 1727, 1449, 1176. **HRMS** (ESI): calc for $C_{13}H_{13}FO_2$ m/z: 200.0900 found $[M+H]^+$ 201.0974. **MP**: 110-112˚C.

4-(4-fluorophenyl)-5-isopropylfuran-2(5*H*)-one (±)-**11m**



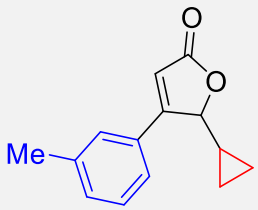
The general procedure for the synthesis of butenolides was followed as described using (±)-**S3** (0.94 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and 4-fluorophenyl boronic acid (2.31 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL) at 65 °C. Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11m** as a white solid (0.79 g, 3.63 mmol, 66% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.44-7.49 (m, 2H), 7.14-7.20 (m, 2H), 6.24 (d, *J* = 1.5 Hz, 1H), 5.40 (dd, *J* = 2.5, 1.5 Hz, 1H), 2.13 (pd, *J* = 6.9, 2.5 Hz, 1H), 1.24 (d, *J* = 6.9 Hz, 3H), 0.62 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.0, 166.1, 164.4 (d, $J_{C\text{-}F}$ = 253.2 Hz, 1C), 129.3 (d, $J_{C\text{-}F}$ = 8.7 Hz, 1C), 126.8 (d, $J_{C\text{-}F}$ = 3.5 Hz, 1C), 116.6 (d, $J_{C\text{-}F}$ = 22.0 Hz, 1C), 115.0 (d, $J_{C\text{-}F}$ = 1.6 Hz, 1C), 86.3, 30.8, 20.3, 13.5. **^{19}F NMR** (377 MHz, $CDCl_3$) δ -107.64. **IR** (neat) V_{max} (cm^{-1}): 2967, 1727, 1510, 1186. **HRMS** (ESI): calc for $C_{13}H_{13}FO_2$ m/z: 200.0900 found $[M+H]^+$ 201.0974. **MP**: 110-112°C.

5-isopropyl-4-(4-methoxyphenyl)furan-2(5*H*)-one (±)-**11n**



The general procedure for the synthesis of butenolides was followed as described using (±)-**S3** (0.94 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and 4-methoxylphenyl boronic acid (2.51 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11n** as a yellow oil (1.02 g, 4.40 mmol, 80% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.9 Hz, 2H), 6.99 (d, *J* = 8.9 Hz, 2H), 6.19 (d, *J* = 1.4 Hz, 1H), 5.41 (dd, *J* = 2.5, 1.4 Hz, 1H), 3.88 (s, 3H), 2.19 (pd, *J* = 6.9, 2.5 Hz, 1H), 1.26 (d, *J* = 6.9 Hz, 3H), 0.64 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.6, 166.9, 162.1, 128.9, 123.0, 114.7, 112.8, 86.2, 55.6, 31.0, 20.3, 13.4. **IR** (neat) V_{max} (cm^{-1}): 2933, 1727, 1512, 1259. **HRMS** (ESI): calc for $C_{14}H_{16}O_3$ m/z: 232.1099 found $[M+H]^+$ 233.1173.

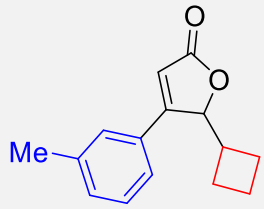
5-cyclopropyl-4-(*m*-tolyl)furan-2(5*H*)-one (±)-**11o**



(±)-**11o**

The general procedure for the synthesis of butenolides was followed as described using (±)-**S5** (0.92 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and *m*-tolyl boronic acid (2.24 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11o** as a white solid (0.88 g, 4.12 mmol, 75% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.32-7.37 (m, 3H), 7.29-7.31 (m, 1H), 6.23 (d, *J* = 1.5 Hz, 1H), 5.20 (dd, *J* = 6.3, 1.5 Hz, 1H), 2.42 (s, 3H), 1.03-1.12 (m, 1H), 0.61-0.71 (m, 2H), 0.47-0.55 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.9, 168.4, 139.0, 132.1, 130.5, 129.0, 128.2, 124.7, 114.0, 83.8, 21.5, 14.0, 4.2, 1.7. **IR** (neat) V_{max} (cm^{-1}): 2921, 1731, 1162, 1059. **HRMS** (ESI): calc for $C_{14}H_{14}O_2$ m/z: 214.0994 found $[M+H]^+$ 215.1068. **MP**: 55-57 °C.

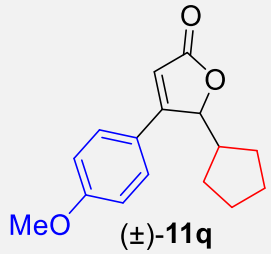
5-cyclobutyl-4-(*m*-tolyl)furan-2(5*H*)-one (±)-**11p**



(±)-**11p**

The general procedure for the synthesis of butenolides was followed as described using (±)-**S6** (1.00 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and *m*-tolyl boronic acid (2.24 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11p** as a colourless oil (0.99 g, 4.34 mmol, 79% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.32-7.36 (m, 1H), 7.27-7.30 (m, 1H), 7.20-7.23 (m, 2H), 6.22 (d, *J* = 1.4 Hz, 1H), 5.43 (dd, *J* = 3.5, 1.4 Hz, 1H), 2.77 (dh, *J* = 8.4, 3.3 Hz, 1H), 2.40 (s, 3H), 2.24-2.32 (m, 1H), 1.99-2.08 (m, 1H), 1.68-1.81 (m, 3H), 1.47-1.56 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.5, 166.8, 139.1, 132.1, 130.4, 129.1, 127.8, 124.3, 114.5, 83.4, 37.5, 24.6, 21.5, 20.4, 17.8. **IR** (neat) V_{max} (cm^{-1}): 2937, 1742, 1161, 1033. **HRMS** (ESI): calc for $C_{15}H_{16}O_2$ m/z: 228.1150 found $[M+H]^+$ 229.1224.

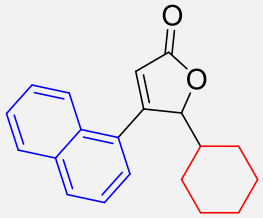
5-cyclopentyl-4-(4-methoxyphenyl)furan-2(5*H*)-one (±)-**11q**



(±)-**11q**

The general procedure for the synthesis of butenolides was followed as described using (±)-**S7** (1.08 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and 4-methoxylphenyl boronic acid (2.51 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11q** as a white solid (1.21 g, 4.68 mmol, 85% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.42 (d, *J* = 8.9 Hz, 2H), 6.97 (d, *J* = 8.9 Hz, 2H), 6.15 (d, *J* = 1.4 Hz, 1H), 5.56 (dd, *J* = 2.9, 1.4 Hz, 1H), 3.86 (s, 3H), 2.33 (pd, *J* = 8.9, 2.9 Hz, 1H), 1.78-1.91 (m, 2H), 1.64-1.72 (m, 1H), 1.56-1.62 (m, 1H), 1.48-1.54 (m, 1H), 13.4-1.42 (m, 1H), 1.12-1.19 (m, 1H), 1.04-1.09 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.8, 167.5, 162.0, 128.9, 123.1, 114.7, 112.7, 83.7, 55.6, 42.0, 29.7, 25.8, 25.5, 23.7. **IR** (neat) V_{max} (cm^{-1}): 2939, 1727, 1262, 1174. **HRMS** (ESI): calc for $C_{16}H_{18}O_3$ m/z: 258.1256 found $[M+H]^+$ 259.1330. **MP**: 108-110 ℃.

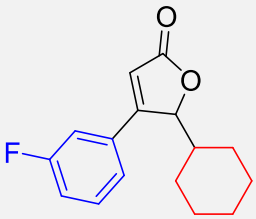
5-cyclohexyl-4-(naphthalen-1-yl)furan-2(5*H*)-one (±)-**11r**



(±)-**11r**

The general procedure for the synthesis of butenolides was followed as described using (±)-**S8** (1.16 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and Naphthalene-1-boronic acid (2.84 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11r** as a white solid (1.30 g, 4.46 mmol, 81% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.87-7.94 (m, 4H), 7.51-7.62 (m, 3H), 6.37 (d, *J* = 1.6 Hz, 1H), 5.53 (t, *J* = 1.6 Hz, 1H), 1.89-1.94 (m, 2H), 1.79-1.86 (m, 1H), 1.56-1.62 (m, 2H), 1.17-1.26 (m, 3H), 1.07-1.12 (m, 1H), 1.02-1.06 (m, 1H), 0.92-0.99 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.3, 166.9, 134.5, 133.0, 129.3, 128.9, 128.0, 127.3, 127.2, 124.1, 115.4, 86.3, 40.5, 30.6, 26.6, 25.9, 25.7, 23.8. **IR** (neat) V_{max} (cm^{-1}): 2924, 1719, 1447, 1171. **HRMS** (ESI): calc for $C_{20}H_{20}O_2$ m/z: 292.1463 found $[M+H]^+$ 293.1537. **MP**: 120-122˚C.

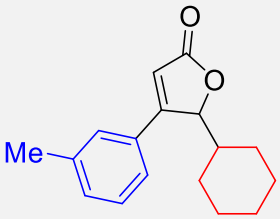
5-cyclohexyl-4-(3-fluorophenyl)furan-2(5*H*)-one (±)-**11s**



(±)-**11s**

The general procedure for the synthesis of butenolides was followed as described using (±)-**S8** (1.16 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and 3-fluorophenyl boronic acid (2.31 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL) at 65 °C. Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11s** as a white solid (0.86 g, 3.30 mmol, 60% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.46 (td, *J* = 8.0, 5.8 Hz, 1H), 7.22-7.25 (m, 1H), 7.17-7.22 (m, 1H), 7.13-7.16 (m, 1H), 6.27 (d, *J* = 1.6 Hz, 1H), 5.35 (t, *J* = 1.6 Hz, 1H), 1.77-1.87 (m, 3H), 1.57-1.65 (m, 3H), 1.22-1.26 (m, 1H), 1.14-1.17 (m, 1H), 1.04-1.12 (m, 1H), 0.97-1.03 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.7, 165.6 (d, J_{C-F} = 2.5 Hz, 1C), 163.0 (d, J_{C-F} = 248.5 Hz, 1C), 132.6 (d, J_{C-F} = 7.7 Hz, 1C), 131.1 (d, J_{C-F} = 8.3 Hz, 1C), 122.9 (d, J_{C-F} = 3.1 Hz, 1C), 118.2 (d, J_{C-F} = 21.2 Hz, 1C), 116.3, 114.1 (d, J_{C-F} = 22.4 Hz, 1C), 86.2, 40.3, 30.5, 26.5, 25.8, 25.7, 23.8. **^{19}F NMR** (377 MHz, $CDCl_3$) δ -110.93. **IR** (neat) V_{max} (cm^{-1}): 2931, 1720, 1449, 1161. **HRMS** (ESI): calc for $C_{16}H_{17}FO_2$ m/z: 260.1213 found $[M+H]^+$ 261.1287. **MP**: 95-97 °C.

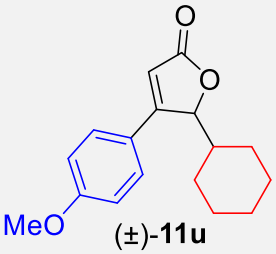
5-cyclohexyl-4-(*m*-tolyl)furan-2(5*H*)-one (±)-**11t**



(±)-**11t**

The general procedure for the synthesis of butenolides was followed as described using (±)-**S8** (1.16 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and *m*-tolyl boronic acid (2.24 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11t** as a white solid (0.99 g, 3.85 mmol, 70% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34-7.38 (m, 1H), 7.29-7.31 (m, 1H), 7.22-7.26 (m, 2H), 6.22 (d, *J* = 1.6 H, 1H), 5.37 (t, *J* = 1.9 Hz, 1H), 2.42 (s, 3H), 1.75-1.86 (m, 3H), 1.50-1.67 (m, 3H), 1.21-1.29 (m, 2H), 1.15-1.19 (m, 1H), 0.96-1.03 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.3, 167.3, 139.2, 132.1, 130.6, 129.2, 127.8, 124.3, 114.9, 86.3, 40.3, 30.6, 26.6, 25.9, 25.7, 23.8, 21.5. **IR** (neat) V_{max} (cm^{-1}): 2921, 1719, 1443, 1174. **HRMS** (ESI): calc for $C_{17}H_{20}O_2$ m/z: 256.1463 found $[M+H]^+$ 257.1537. **MP**: 75-77 °C.

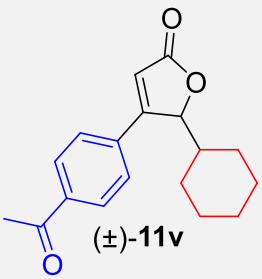
5-cyclohexyl-4-(4-methoxyphenyl)furan-2(5*H*)-one (±)-**11u**



(±)-**11u**

The general procedure for the synthesis of butenolides was followed as described using (±)-**S8** (1.16 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and 4-methoxyphenyl boronic acid (2.51 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11u** as a white solid (1.00 g, 3.69 mmol, 67% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.8 Hz, 2H), 6.98 (d, *J* = 8.8 Hz, 2H), 6.15 (d, *J* = 1.2 Hz, 1H), 5.34 (d, *J* = 1.7 Hz, 1H), 3.87 (s, 3H), 1.76-1.86 (m, 3H), 1.56-1.66 (m, 3H), 1.20-1.29 (m, 2H), 1.15-1.17 (m, 1H), 0.95-1.04 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 173.68, 166.57, 162.10, 128.94, 123.13, 114.79, 112.84, 86.16, 55.61, 40.68, 30.71, 26.70, 25.97, 25.82, 23.71. **IR** (neat) V_{max} (cm^{-1}): 2922, 1719, 1512, 1259, 1172. **HRMS** (ESI): calc for $C_{17}H_{20}O_3$ m/z: 272.1412 found $[M+H]^+$ 273.1490. **MP**: 110-112°C.

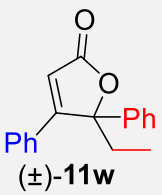
5-cyclohexyl-4-(4-acetylphenyl)furan-2(5*H*)-one (±)-**11v**



(±)-**11v**

The general procedure for the synthesis of butenolides was followed as described using (±)-**S8** (1.16 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and 4-acetylphenyl boronic acid (2.71 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). The crude sample of (±)-**11v** was obtained as a white solid (1.11 g, 3.90 mmol, 71% crude yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.03-8.07 (m, 2H), 7.53-7.57 (m, 2H), 6.35 (d, *J* = 1.6 Hz, 1H), 5.42 (dd, *J* = 2.5, 1.6 Hz, 1H), 2.65 (s, 3H), 1.73-1.87 (m, 3H), 1.57-1.64 (m, 3H), 1.23-1.28 (m, 2H), 1.13-1.15 (m, 1H), 0.94-1.02 (m, 2H). **IR** (neat) V_{max} (cm^{-1}): 2923, 1719, 1685, 1263, 1179. **HRMS** (ESI): calc for $C_{18}H_{20}O_3$ m/z: 284.1412 found $[M+H]^+$ 285.1486.

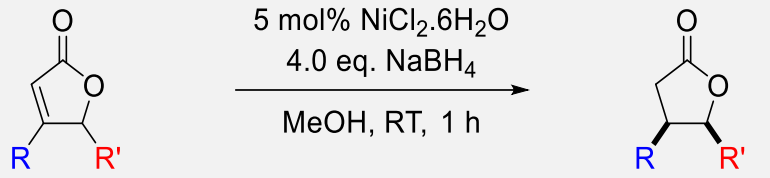
5-ethyl-4,5-diphenylfuran-2(5*H*)-one (±)-**11w**



(±)-**11w**

The general procedure for the synthesis of butenolides was followed as described using (±)-**S9** (1.28 g, 5.50 mmol, 1.0 eq.) in MeOH (10 mL), $Cu(OAc)_2$ (0.599 g, 3.3 mmol, 0.6 eq.) and phenyl boronic acid (2.01 g, 16.5 mmol, 3.0 eq.) in MeOH (30 mL). Purification by silica gel chromatography using 5-10% ethyl acetate in hexane produced (±)-**11w** as a white solid (0.81 g, 3.08 mmol, 56% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.42-7.44 (m, 1H), 7.40-7.42 (m, 1H), 7.38-7.40 (m, 2H), 7.36-7.38 (m, 2H), 7.29-7.33 (m, 2H), 7.21-7.23 (m, 2H), 6.45 (s, 1H), 2.65 (dq, *J* = 14.5, 7.3 Hz, 1H), 2.35 (dq, *J* = 14.5, 7.3 Hz, 1H), 0.88 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.4, 169.3, 138.1, 130.9, 130.5, 129.1, 129.0, 127.8, 126.3, 116.4, 91.7, 27.9, 7.4. **IR** (neat) V_{max} (cm^{-1}): 1735, 1446, 1222, 1065. **HRMS** (ESI): calc for $C_{18}H_{16}O_2$ m/z: 264.1150 found $[M+H]^+$ 265.1224. **MP**: 115-117˚C.

Synthesis of *cis*-β-aryl, γ-alkyl disubstituted γ-butyrolactones (12a-12v)



11a-11w *cis*-**12a-12v**

General experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones.

$NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.) was added, all at once, to a stirring light-green solution of pre-mixed $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.) and butenolide (0.75 mmol, 1.0 eq.) in MeOH (10 mL) at room temperature. A vigorous effervescences occurred, and the resulting black solution was stirred at room temperature under the atmosphere of nitrogen till completion (between 0.5 h-1 h, TLC control). Afterwards, the mixture was diluted with EtOAc (30 mL) and poured into a separating funnel which contains saturated aqueous solution of ammonium chloride (20 mL). The aqueous layer was further extracted with EtOAc (10 mL). The combined organic layers were washed with brine (30 mL), dried with $MgSO_4$, filtered, and solvent removed *in vacuo* to generate the crude sample. Purification by silica gel chromatography using 5-25% EtOAc in hexanes furnished β-aryl, γ-alkyl disubstituted γ-butyrolactones.

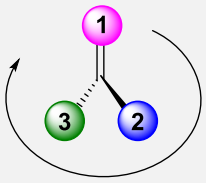
Note: The reaction works well either under the atmosphere of nitrogen or air. Figure **S1** shows formation of the nickel boride in the absence of a butenolide. Figure **S2** is indicative of no reaction taking place (and no effervescence), due to lack of formation of the key Ni-H specie needed for the reaction to occur. Figure **S3** is a representation of the reaction progressing well, and no formation of nickel boride. No reaction occurred when butenolide was added to the vessel shown in figure S1.

Fig S1: $NiCl_2.6H_2O$ (5 mol%) + $NaBH_4$ (4 eq.) in MeOH

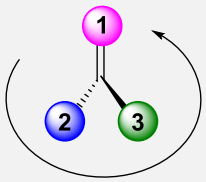
Fig S2: $NiCl_2.6H_2O$ (5 mol%) + $NaBH_4$ (4 eq.) + butenolide (1 eq.) in THF or PhMe

Fig S3: $NiCl_2.6H_2O$ (5 mol%) + $NaBH_4$ (4 eq.) + butenolide (1 eq.) in MeOH

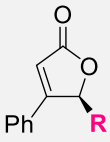
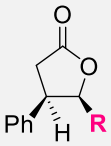
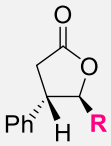
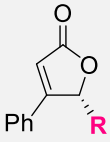
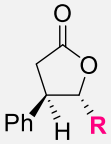
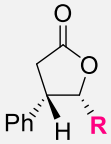
Figure S4



*looking at the **re** face*



*looking at the **si** face*

Starting Material		*Product of*	
		Re-facial attack	***Si***-facial attack
 **L**	→	 **M**	 **N**
+			
 **O**	→	 **P**	 **Q**
L + O	→	*four possible stereo-isomers*	

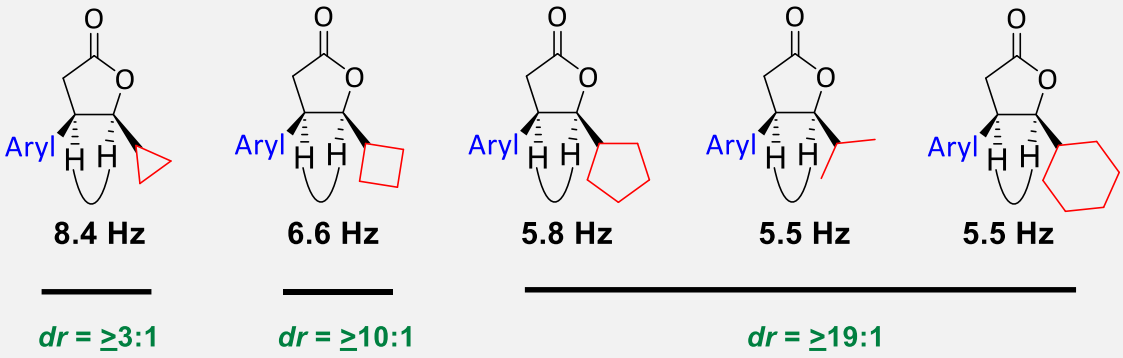
when **R** is *small, re- and si*-facial attack on **L**. Also, re- and *si*-facial attack on **O** = poor *dr*

when **R** is *large, re* face attack on **L**, but *si* face attack on **O** = excellent *dr*

when **R** is *large, hydride is delivered opposite to **R**-group* ONLY, therefore, ***M** and **Q** are generated.*

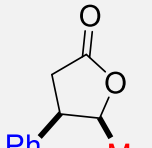
***M** and **Q** are enantiomers*

Figure S5: Observed trend in coupling constant. Aryl = Phenyl or substituted phenyl.



See page S105, S130 and S133 for NOESY for **12c**, **12g** and **12h** respectively.

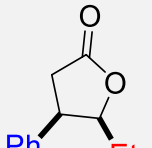
Dihydro-5-methyl-4-phenyl-2(3*H*)-furanone (±)-**12a**



(±)-**12a**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11a** (130 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 5-10% EtOAc in hexanes furnished (±)-**12a** (79.3 mg, 0.45 mmol, 60%) as colourless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33-7.38 (m, 2H), 7.28-7.32 (m, 1H), 7.12-7.15 (m, 2H), 4.93 (p, *J* = 6.6 Hz, 1H), 3.76 (dt, *J* = 8.5, 6.6 Hz, 1H), 2.95 (dd, *J* = 17.5, 8.5 Hz, 1H), 2.83 (dd, *J* = 17.5, 6.6 Hz, 1H), 1.01 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 176.7, 137.7, 128.9, 127.8, 127.7, 80.1, 44.9, 35.0, 16.8. The data obtained agrees with literature data.[5a]

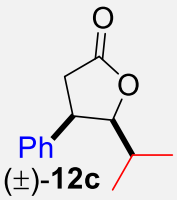
Dihydro-5-ethyl-4-phenyl-2(3*H*)-furanone (±)-**12b**



(±)-**12b**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11b** (140 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 5-10% EtOAc in hexanes furnished (±)-**12b** (88.5 mg, 0.46 mmol, 62%) as colourless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.32-7.36 (m, 2H), 7.27-7.31 (m, 1H), 7.12-7.15 (m, 2H), 4.63 (ddd, *J* = 9.3, 6.5, 4.6 Hz, 1H), 3.74 (ddd, *J* = 8.6, 6.5, 4.9 Hz, 1H), 2.98 (dd, *J* = 17.5, 8.6 Hz, 1H), 2.77 (dd, *J* = 17.5, 4.9 Hz, 1H), 1.32 (dt, *J* = 9.3, 7.4 Hz, 1H), 1.19-1.24 (m, 1H), 0.90 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 176.9, 138.1, 128.9, 127.9, 127.7, 85.8, 44.6, 36.0, 24.6, 10.4. The data obtained agrees with literature data.[5b]

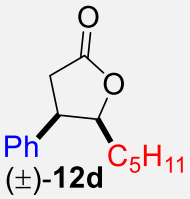
Dihydro-5-(1-methylethyl)-4-phenyl-2(3*H*)-furanone (±)-**12c**



(±)-**12c**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11c** (150 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 5-10% EtOAc in hexanes furnished (±)-**12c** (125.6 mg, 0.62 mmol, 82%) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.21-7.29 (m, 3H), 7.09-7.12 (m, 2H), 4.18 (dd, *J* = 10.1, 5.5 Hz, 1H), 3.56 (ddd, *J* = 8.6, 5.5, 1.6 Hz, 1H), 2.99 (dd, *J* = 17.5, 8.6 Hz, 1H), 2.56 (dd, *J* = 17.5, 1.6 Hz, 1H), 1.53 (dp, *J* = 10.1, 6.6 Hz, 1H), 0.95 (d, *J* = 6.6 Hz, 3H), 0.66 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 177.2, 139.2, 128.8, 128.1, 127.6, 90.1, 44.3, 38.8, 28.9, 20.0, 17.7. **IR** (neat) V_{max} (cm^{-1}): 2967, 1764, 1468, 1140. **HRMS** (ESI): calc for $C_{13}H_{16}O_2$ m/z: 204.1150 found $[M+H]^+$ 205.1224. **MP**: 95-97˚C.

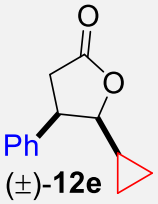
Dihydro-5-pentyl-4-phenyl-2(3*H*)-furanone (±)-**12d**



(±)-**12d**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11d** (170 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 5-10% EtOAc in hexanes furnished (±)-**12d** (106.3 mg, 0.45 mmol, 61%) as colourless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.32-7.36 (m, 2H), 7.27-7.30 (m, 1H), 7.12-7.15 (m, 2H), 4.71 (ddd, *J* = 9.0, 6.4, 4.2 Hz, 1H), 3.72 (ddd, *J* = 8.6, 6.5, 4.9 Hz, 1H), 2.97 (dd, *J* = 17.5, 8.6 Hz, 1H), 2.77 (dd, *J* = 17.5, 4.9 Hz, 1H), 1.22-1.44 (m, 1H), 1.11-1.20 (m, 1H), 0.81 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 177.0, 138.2, 128.9, 127.9, 127.7, 84.4, 44.7, 35.9, 31.5, 31.2, 25.6, 22.5, 14.0. The data obtained agrees with literature data.[5c]

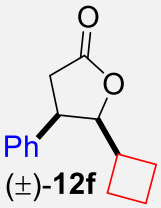
Dihydro-5-(cyclopropyl)-4-phenyl-2(3*H*)-furanone (±)-**12e**



(±)-**12e**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11e** (150 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 5-10% EtOAc in hexanes furnished (±)-**12e** (91.0 mg, 0.45 mmol, 60%) as white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33-7.37 (m, 2H), 7.27-7.32 (m, 1H), 7.21-7.23 (m, 2H), 4.01 (dd, *J* = 8.4, 6.6 Hz, 1H), 3.79 (dd, *J* = 8.4, 5.6 Hz, 1H), 2.95 (dd, *J* = 17.5, 8.4 Hz, 1H), 2.86 (dd, *J* = 17.5, 5.6 Hz, 1H), 0.47-0.57 (m, 2H), 0.40-0.46 (m, 1H), 0.27-0.34 (m, 1H), 0.10-0.17 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 176.8, 138.3, 128.8, 128.0, 127.6, 89.1, 44.9, 35.7, 11.5, 4.0, 2.3. **IR** (neat) V_{max} (cm^{-1}): 2970, 1755, 1178, 1141. **HRMS** (ESI): calc for $C_{13}H_{14}O_2$ m/z: 202.0994 found $[M+H]^+$ 203.1068. **MP**: 105-107˚C.

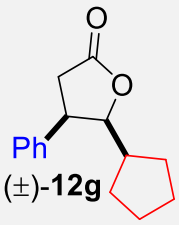
Dihydro-5-(cyclobutyl)-4-phenyl-2(3*H*)-furanone (±)-**12f**



(±)-**12f**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11f** (160 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 5-10% EtOAc in hexanes furnished (±)-**12f** (126.5 mg, 0.59 mmol, 78%) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.28-7.34 (m, 3H), 7.09-7.12 (m, 2H), 4.67 (dd, *J* = 8.6, 6.6, Hz, 1H), 3.74 (ddd, *J* = 8.6, 6.6, 5.3 Hz, 1H), 2.94 (dd, *J* = 17.5, 8.6 Hz, 1H), 2.78 (dd, *J* = 17.5, 5.2 Hz, 1H), 2.13-2.23 (m, 1H), 1.63-1.78 (m, 4H), 1.47-1.55 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 177.1, 137.9, 128.8, 127.8, 127.6, 87.7, 43.8, 36.0, 35.8, 25.0, 24.4, 18.5. **IR** (neat) V_{max} (cm^{-1}): 2978, 1759, 1169, 1184. **HRMS** (ESI): calc for $C_{14}H_{16}O_2$ m/z: 216.1150 found $[M+H]^+$ 217.1224. **MP**: 55-57˚C.

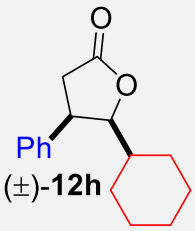
Dihydro-5-(cyclopentyl)-4-phenyl-2(3*H*)-furanone (±)-**12g**



(±)-**12g**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11g** (170 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 5-10% EtOAc in hexanes furnished (±)-**12g** (150.3 mg, 0.65 mmol, 87%) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.28-7.35 (m, 3H), 7.14-7.17 (m, 2H), 4.43 (dd, *J* = 10.0, 5.8 Hz, 1H), 3.64 (ddd, *J* = 8.6, 5.8, 2.4 Hz, 1H), 3.04 (dd, *J* = 17.5, 8.6 Hz, 1H), 2.69 (dd, *J* = 17.5, 2.4 Hz, 1H), 1.65-1.84 (m, 2H), 1.50-1.59 (m, 3H), 1.31-1.44 (m, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 177.4, 139.3, 128.8, 128.2, 127.6, 89.3, 44.7, 40.3, 38.0, 30.9, 27.9, 25.6, 25.3. **IR** (neat) V_{max} (cm^{-1}): 2964, 1760, 1112, 1024. **HRMS** (ESI): calc for $C_{15}H_{18}O_2$ m/z: 230.1307 found $[M+H]^+$ 231.1381. **MP**: 75-77˚C.

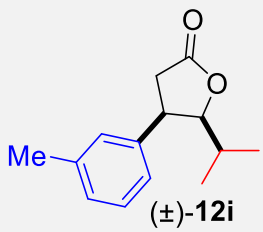
Dihydro-5-(cyclohexyl)-4-phenyl-2(3*H*)-furanone (±)-**12h**



(±)-**12h**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11h** (180 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 5-10% EtOAc in hexanes furnished (±)-**12h** (168.6 mg, 0.69 mmol, 92%) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.28-7.35 (m, 3H), 7.15-7.17 (m, 2H), 4.32 (dd, *J* = 9.9, 5.5 Hz, 1H), 3.62 (ddd, *J* = 8.5, 5.5, 1.7 Hz, 1H), 3.03 (dd, *J* = 17.4, 8.5 Hz, 1H), 2.61 (dd, *J* = 17.4, 1.7 Hz, 1H), 1.97-1.99 (m, 1H), 1.65-1.66 (m, 1H), 1.47-1.55 (m, 2H), 1.25-1.30 (m, 2H), 1.03-1.08 (m, 3H), 0.83-0.88 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 177.1, 139.1, 128.7, 127.9, 127.4, 88.7, 44.0, 38.5, 37.8, 29.9, 27.7, 26.1, 25.1, 25.0. **IR** (neat) V_{max} (cm^{-1}): 2926, 1760, 1112, 1078. **HRMS** (ESI): calc for $C_{16}H_{20}O_2$ m/z: 244.1463 found $[M+H]^+$ 245.1537. **MP**: 75-77˚C.

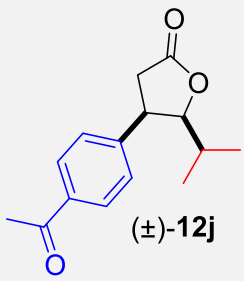
5-isopropyl-4-(*m*-tolyl)dihydrofuran-2(3*H*)-one (±)-**12i**



(±)-**12i**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11i** (160 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 5-10% EtOAc in hexanes furnished (±)-**12i** (144.1 mg, 0.66 mmol, 88%) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.16-7.22 (m, 1H), 7.07-7.10 (m, 1H), 6.95-6.97 (m, 2H), 4.24 (dd, *J* = 10.1, 5.5 Hz, 1H), 3.58 (ddd, *J* = 8.6, 5.5, 1.7 Hz, 1H), 3.04 (dd, *J* = 17.5, 8.6 Hz, 1H), 2.61 (dd, *J* = 17.5, 1.7 Hz, 1H), 2.33 (s, 3H), 1.62 (dp, *J* = 10.1, 6.5 Hz, 1H), 1.02 (d, *J* = 6.5 Hz, 3H), 0.73 (d, *J* = 6.5 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 177.3, 139.2, 138.5, 128.7, 128.3, 125.2, 90.1, 44.2, 38.8, 28.8, 21.6, 20.0, 17.8. **IR** (neat) V_{max} (cm^{-1}): 2961, 1774, 1473, 1141. **HRMS** (ESI): calc for $C_{14}H_{18}O_2$ m/z: 218.1307 found $[M+H]^+$ 219.1381. **MP**: 60-62˚C

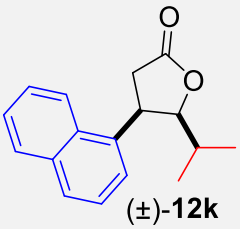
4-(4-acetylphenyl)-5-isopropyldihydrofuran-2(3*H*)-one (±)-**12j**



(±)-**12j**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (28.4 mg, 0.75 mmol, 1.0 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11j** (180 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 10-20% EtOAc in hexanes furnished (±)-**12j** (110.8 mg, 0.45 mmol, 60%) as white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.92 (d, *J* = 8.4 Hz, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 4.27 (dd, *J* = 10.2, 5.5 Hz, 1H), 3.70 (ddd, *J* = 8.7, 5.5, 1.5 Hz, 1H), 3.09 (dd, *J* = 17.5, 8.6 Hz, 1H), 2.64 (dd, *J* = 17.5, 1.7 Hz, 1H), 2.60 (s, 3H), 1.56 (dp, *J* = 10.2, 6.5 Hz, 1H), 1.03 (d, *J* = 6.5 Hz, 3H), 0.72 (d, *J* = 6.5 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.6, 176.7, 144.8, 136.7, 129.0, 128.5, 89.2, 44.4, 38.8, 29.0, 26.8, 20.1, 17.8. **IR** (neat) V_{max} (cm^{-1}): 2967, 1760, 1681, 1269, 1176. **HRMS** (ESI): calc for $C_{15}H_{18}O_3$ m/z: 246.1256 found $[M+H]^+$ 247.1330. **MP**: 150-152˚C

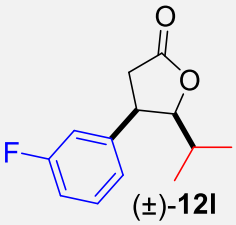
5-isopropyl-4-(naphthalen-1-yl)dihydrofuran-2(3*H*)-one (±)-**12k**



(±)-**12k**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11k** (190 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 5-10% EtOAc in hexanes furnished (±)-**12k** (182.5 mg, 0.72 mmol, 92%) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.76–7.86 (m, 3H), 7.62 (d, *J* = 1.9 Hz, 1H), 7.44–7.55 (m, 2H), 7.30 (dd, *J* = 8.5, 1.9 Hz, 1H), 4.33 (dd, *J* = 10.1, 5.5 Hz, 1H), 3.81 (ddd, *J* = 8.6, 5.5, 1.7 Hz, 1H), 3.14 (dd, *J* = 17.5, 8.6 Hz, 1H), 2.71 (dd, *J* = 17.5, 1.7 Hz, 1H), 1.64 (dp, *J* = 10.1, 6.6 Hz, 1H), 1.03 (d, *J* = 6.6 Hz, 3H), 0.74 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 177.2, 136.7, 133.3, 132.7, 128.7, 127.9, 127.7, 127.0, 126.6, 126.2, 125.9, 90.1, 44.5, 38.9, 29.0, 20.0, 17.9. **IR** (neat) V_{max} (cm^{-1}): 2970, 1761, 1412, 1174. **HRMS** (ESI): calc for $C_{17}H_{18}O_2$ m/z: 254.1307 found $[M+H]^+$ 255.1381. **MP**: 155-157˚C

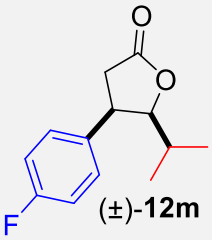
4-(3-fluorophenyl)-5-isopropyldihydrofuran-2(3*H*)-one (±)-**12l**



(±)-**12l**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11l** (170 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 5-10% EtOAc in hexanes furnished (±)-**12l** (151.7 mg, 0.68 mmol, 91%) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.30 (td, *J* = 8.0, 6.0 Hz, 1H), 6.93–7.02 (m, 2H), 6.89 (dt, *J* = 9.7, 2.1 Hz, 1H), 4.24 (dd, *J* = 10.1, 5.5 Hz, 1H), 3.62 (ddd, *J* = 8.6, 5.5, 1.6 Hz, 1H), 3.06 (dd, *J* = 17.5, 8.6 Hz, 1H), 2.61 (dd, *J* = 17.5, 1.6 Hz, 1H), 1.62 (dp, *J* = 10.1, 6.6 Hz, 1H), 1.03 (d, *J* = 6.6 Hz, 3H), 0.74 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 176.7, 162.9 (d, J_{C-F} = 247.0 Hz, 1C), 141.7 (d, J_{C-F} = 7.0 Hz, 1C), 130.5 (d, J_{C-F} = 8.4 Hz, 1C), 123.7 (d, J_{C-F} = 2.9 Hz, 1C), 115.2 (d, J_{C-F} = 21.7 Hz, 1C), 114.7 (d, J_{C-F} = 21.0 Hz, 1C), 89.7, 44.1 (d, J_{C-F} = 1.7 Hz, 1C), 38.7, 28.8, 20.0, 17.8. **^{19}F NMR** (377 MHz, $CDCl_3$) δ -112.0. **IR** (neat) V_{max} (cm^{-1}): 2930, 1748, 1487, 1170. **HRMS** (ESI): calc for $C_{13}H_{15}FO_2$ m/z: 222.1056 found $[M+H]^+$ 223.1130. **MP**: 71-73˚C

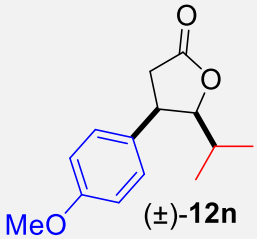
4-(4-fluorophenyl)-5-isopropyldihydrofuran-2(3*H*)-one (±)-**12m**



(±)-**12m**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11m** (170 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 5-10% EtOAc in hexanes furnished (±)-**12m** (156.7 mg, 0.71 mmol, 94%) as white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.12-7.17 (m, 2H), 6.99-7.04 (m, 2H), 4.23 (dd, *J* = 10.2, 5.4 Hz, 1H), 3.62 (ddd, *J* = 8.6, 5.4, 1.5 Hz, 1H), 3.06 (dd, *J* = 17.5, 8.6 Hz, 1H), 2.58 (dd, *J* = 17.5, 1.5 Hz, 1H), 1.51–1.64 (m, 1H), 1.03 (d, *J* = 6.5 Hz, 3H), 0.72 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 176.9, 162.2 (d, J_{C-F} = 246.6 Hz, 1C), 135.0 (d, J_{C-F} = 3.4 Hz, 1C), 129.6 (d, J_{C-F} = 7.9 Hz, 1C), 115.8 (d, J_{C-F} = 21.3 Hz, 1C), 89.9, 43.6, 39.0, 28.8, 20.0, 17.6. **^{19}F NMR** (377 MHz, $CDCl_3$) δ -114.6. **IR** (neat) V_{max} (cm^{-1}): 2974, 1751, 1506, 1204, 1160. **HRMS** (ESI): calc for $C_{13}H_{15}FO_2$ m/z: 222.1056 found $[M+H]^+$ 223.1130. **MP**: 81-83˚C

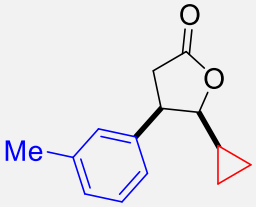
5-isopropyl-4-(4-methoxyphenyl)dihydrofuran-2(3*H*)-one (±)-**12n**



(±)-**12n**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11n** (170 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 10-15% EtOAc in hexanes furnished (±)-**12n** (166.9 mg, 0.71 mmol, 95%) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.08 (d, *J* = 8.8 Hz, 2H), 6.85 (d, *J* = 8.8 Hz, 2H), 4.21 (dd, *J* = 10.1, 5.5 Hz, 1H), 3.80 (s, 3H), 3.58 (ddd, *J* = 8.6, 5.5, 1.6 Hz, 1H), 3.03 (dd, *J* = 17.4, 8.6 Hz, 1H), 2.58 (dd, *J* = 17.4, 1.6 Hz, 1H), 1.59 (dp, *J* = 10.1, 6.6 Hz, 1H), 1.01 (d, *J* = 6.6 Hz, 3H), 0.72 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 177.4, 159.0, 131.2, 129.1, 114.2, 90.3, 55.3, 43.5, 39.0, 28.9, 20.0, 17.7. **IR** (neat) V_{max} (cm^{-1}): 2988, 1759, 1514, 1174. **HRMS** (ESI): calc for $C_{14}H_{18}O_3$ m/z: 234.1256 found $[M+H]^+$ 235.1130. **MP**: 97-99˚C

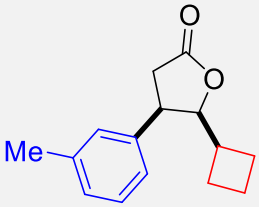
5-cyclopropyl-4-(*m*-tolyl)dihydrofuran-2(3*H*)-one (±)-**12o**



(±)-**12o**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11o** (160 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 5-10% EtOAc in hexanes furnished (±)-**12o** (100.6 mg, 0.47 mmol, 62%) as colourless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.21-7.25 (m, 1H), 7.09-7.11 (m, 1H), 7.00-7.03 (m, 2H), 3.96-4.03 (m, 1H), 3.75 (ddd, *J* = 8.4, 6.5, 5.2 Hz, 1H), 2.93 (dd, *J* = 17.5, 8.4 Hz, 1H), 2.84 (dd, *J* = 17.5, 5.2 Hz, 1H), 2.35 (s, 3H), 0.83-0.95 (m, 1H), 0.48-0.57 (m, 2H), 0.40-0.46 (m, 1H), 0.28-0.35 (m, 1H), 0.11-0.17 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 177.0, 138.47, 138.40, 128.6, 128.3, 125.1, 89.2, 44.9, 35.8, 21.6, 11.4, 4.0, 2.3. **IR** (neat) V_{max} (cm^{-1}): 2922, 1769, 1163. **HRMS** (ESI): calc for $C_{14}H_{16}O_2$ m/z: 216.1150 found $[M+H]^+$ 217.1224.

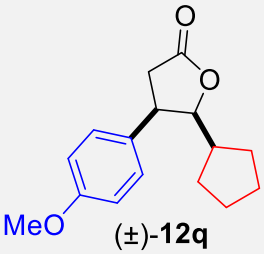
5-cyclobutyl-4-(*m*-tolyl)dihydrofuran-2(3*H*)-one (±)-**12p**



(±)-**12p**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11p** (170 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 5-10% EtOAc in hexanes furnished (±)-**12p** (108.8 mg, 0.47 mmol, 63%) as colourless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.18-7.22 (m, 1H), 7.07-7.10 (m, 1H), 6.89-6.91 (m, 2H), 4.66 (dd, *J* = 8.6, 6.6 Hz, 1H), 3.70 (ddd, *J* = 8.6, 6.6, 5.2 Hz, 1H), 2.92 (dd, *J* = 17.5, 8.6 Hz, 1H), 2.76 (dd, *J* = 17.5, 5.2 Hz, 1H), 2.33 (s, 3H), 2.14-2.24 (m, 1H), 1.81-1.90 (m, 1H), 1.65-1.78 (m, 4H), 1.52-1.56 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 177.2, 138.4, 137.8, 128.6, 128.5, 128.3, 124.9, 87.8, 43.7, 36.0, 35.8, 25.1, 24.4, 21.5, 18.5. **IR** (neat) V_{max} (cm^{-1}): 2936, 1772, 1165. **HRMS** (ESI): calc for $C_{15}H_{18}O_2$ m/z: 230.1307 found $[M+H]^+$ 231.1381.

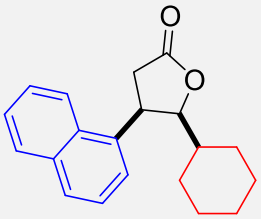
5-cyclopentyl-4-(4-methoxyphenyl)dihydrofuran-2(3*H*)-one (±)-**12q**



(±)-**12q**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11q** (190 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 10-15% EtOAc in hexanes furnished (±)-**12q** (173.8 mg, 0.67 mmol, 89%) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.07 (d, *J* = 8.8 Hz, 2H), 6.85 (d, *J* = 8.8 Hz, 2H), 4.39 (dd, *J* = 9.9, 5.7 Hz, 1H), 3.80 (s, 3H), 3.59 (ddd, *J* = 8.4, 5.7, 2.4 Hz, 1H), 3.01 (dd, *J* = 17.5, 8.4 Hz, 1H), 2.64 (dd, *J* = 17.5, 2.4 Hz, 1H), 1.74–1.84 (m, 1H), 1.67-1.73 (m, 1H), 1.47–1.60 (m, 2H), 1.31–1.45 (m, 4H), 1.02–1.09 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 177.5, 159.0, 131.2, 129.1, 114.1, 89.5, 55.3, 43.9, 40.3, 38.1, 30.9, 27.9, 25.6, 25.3. **IR** (neat) V_{max} (cm^{-1}): 2954, 1740, 1512, 1179. **HRMS** (ESI): calc for $C_{16}H_{20}O_3$ m/z: 260.1412 found $[M+H]^+$ 261.1486. **MP**: 93-95˚C

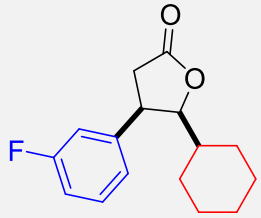
5-cyclohexyl-4-(naphthalen-1-yl)dihydrofuran-2(3*H*)-one (±)-**12r**



(±)-**12r**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11r** (220 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 5-10% EtOAc in hexanes furnished (±)-**12r** (214.2 mg, 0.73 mmol, 97%) as white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80-7.85 (m, 3H), 7.61-7.62 (m, 1H), 7.46-7.53 (m, 2H), 7.30 (dd, *J* = 8.5, 1.9 Hz, 1H), 4.40 (dd, *J* = 10.0, 5.5 Hz, 1H), 3.80 (ddd, *J* = 8.5, 5.5, 1.7 Hz, 1H), 3.11 (dd, *J* = 17.5, 8.5 Hz, 1H), 2.68 (dd, *J* = 17.5, 1.7 Hz, 1H), 2.01-2.04 (m, 1H), 1.63-1.65 (m, 1H), 1.50-1.54 (m, 3H), 1.31-1.41 (m, 1H), 1.00-1.14 (m, 3H), 0.74-0.94 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 177.2, 136.8, 133.4, 132.7, 128.7, 127.9, 127.8, 126.9, 126.5, 126.2, 126.0, 88.9, 44.3, 38.8, 38.0, 30.1, 27.9, 26.2, 25.17, 25.12. **IR** (neat) V_{max} (cm^{-1}): 2936, 1770, 1180. **HRMS** (ESI): calc for $C_{20}H_{22}O_2$ m/z: 294.1620 found $[M+H]^+$ 295.1694. **MP**: 135-137˚C

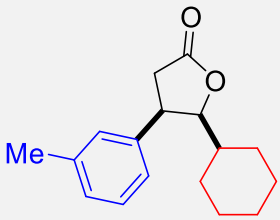
5-cyclohexyl-4-(3-fluorophenyl)dihydrofuran-2(3*H*)-one (±)-**12s**



(±)-**12s**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11s** (200 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 5-10% EtOAc in hexanes furnished (±)-**12s** (186.9 mg, 0.71 mmol, 95%) as white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.27-7.33 (m, 1H), 6.94-7.01 (m, 2H), 6.87-6.90 (m, 1H), 4.31 (dd, *J* = 10.0, 5.5 Hz, 1H), 3.62 (ddd, *J* = 8.5, 5.5, 1.8 Hz, 1H), 3.03 (dd, *J* = 17.5, 8.5 Hz, 1H), 2.58 (dd, *J* = 17.4, 1.8 Hz, 1H), 1.95-2.01 (m, 1H), 1.66-1.69 (m, 1H), 1.43–1.60 (m, 3H), 1.30-1.35 (m, 1H), 0.99-1.14 (m, 3H), 0.81-0.94 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 176.5, 162.9 (d, J_{C-F} = 246.9 Hz, 1C), 141.7 (d, J_{C-F} = 7.0 Hz, 1C), 130.5 (d, J_{C-F} = 8.3 Hz, 1C), 123.7 (d, J_{C-F} = 2.9 Hz, 1C), 115.8 (d, J_{C-F} = 21.7 Hz, 1C), 114.6 (d, J_{C-F} = 20.9 Hz, 1C), 88.5, 43.99, 43.98, 38.5, 37.9, 30.1, 27.8, 26.2, 25.2. **^{19}F NMR** (377 MHz, $CDCl_3$) δ -112.0. **IR** (neat) V_{max} (cm^{-1}): 2927, 1755, 1588, 1194. **HRMS** (ESI): calc for $C_{16}H_{19}FO_2$ m/z: 262.1369 found $[M+H]^+$ 263.1443. **MP**: 96-98˚C

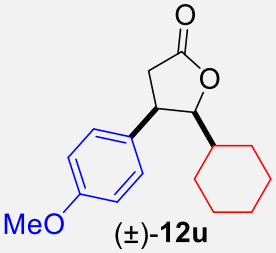
5-cyclohexyl-4-(*m*-tolyl)dihydrofuran-2(3*H*)-one (±)-**12t**



(±)-**12t**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11t** (190 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 5-10% EtOAc in hexanes furnished (±)-**12t** (180.2 mg, 0.69 mmol, 93%) as colourless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.19-7.25 (m, 1H), 7.09-7.13 (m, 1H), 6.97-6.99 (m, 2H), 4.33 (dd, *J* = 9.9, 5.5 Hz, 1H), 3.60 (ddd, *J* = 8.5, 5.5, 1.8 Hz, 1H), 3.03 (dd, *J* = 17.4, 8.5 Hz, 1H), 2.62 (dd, *J* = 17.4, 1.8 Hz, 1H), 2.36 (s, 3H), 2.00-2.02 (m, 1H), 1.65-1.69 (m, 1H), 1.50–1.59 (m, 3H), 1.31-1.41 (m, 1H), 1.03-1.14 (m, 3H), 0.82-0.97 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 177.4, 139.2, 138.5, 128.69, 128.63, 128.3, 125.2, 88.8, 44.1, 38.6, 37.9, 30.1, 27.9, 26.2, 25.2, 25.1, 21.6. **IR** (neat) V_{max} (cm^{-1}): 2922, 1773, 1179. **HRMS** (ESI): calc for $C_{17}H_{22}O_2$ m/z: 258.1620 found $[M+H]^+$ 259.1694.

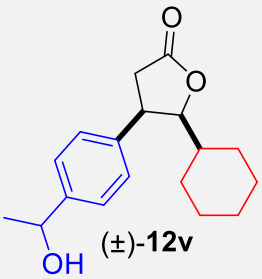
5-cyclohexyl-4-(4-methoxyphenyl)dihydrofuran-2(3*H*)-one (±)-**12u**



(±)-**12u**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11u** (200 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 10-15% EtOAc in hexanes furnished (±)-**12u** (197.5 mg, 0.72 mmol, 96%) as yellow solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.07 (d, *J* = 8.8 Hz, 2H), 6.85 (d, *J* = 8.8 Hz, 2H), 4.28 (dd, *J* = 10.0, 5.4 Hz, 1H), 3.81 (s, 3H), 3.57 (ddd, *J* = 8.5, 5.5, 1.7 Hz, 1H), 3.00 (dd, *J* = 17.4, 8.5 Hz, 1H), 2.56 (dd, *J* = 17.4, 1.7 Hz, 1H), 1.96-1.99 (m, 1H), 1.61-1.67 (m, 1H), 1.46–1.57 (m, 3H), 1.25-1.32 (m, 1H), 1.03-1.12 (m, 3H), 0.82-0.91 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 177.4, 158.9, 131.2, 129.0, 114.1, 89.0, 55.3, 43.4, 38.8, 37.9, 30.0, 27.8, 26.2, 25.27, 25.22. **IR** (neat) V_{max} (cm^{-1}): 2930, 1758, 1511, 1179. **HRMS** (ESI): calc for $C_{17}H_{22}O_3$ m/z: 274.1569 found $[M+H]^+$ 275.1643. **MP**: 102-104˚C

5-cyclohexyl-4-(4-(1-hydroxyethyl)phenyl)dihydrofuran-2(3*H*)-one (±)-**12v**



(±)-**12v**

The general experimental procedure for the Nickel-hydride catalysed 1,4-reduction of β-aryl, γ-alkyl disubstituted α,β-unsaturated lactones procedure was followed, using $NaBH_4$ (113.5 mg, 3.00 mmol, 4 eq.), $NiCl_2.6H_2O$ (8.91 mg, 0.0375 mmol, 0.05 eq.), butenolide (±)-**11v** (210 mg, 0.75 mmol, 1.0 eq.) and MeOH (10 mL). Purification by silica gel chromatography using 15-25% EtOAc in hexanes furnished (±)-**12v** (95.2 mg, 0.33 mmol, 44%) as a white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.32 (d, *J* = 8.3 Hz, 2H), 7.15 (d, *J* = 8.3 Hz, 2H), 4.91 (qd, *J* = 6.5, 2.0 Hz, 1H), 4.31 (dd, *J* = 10.0, 5.4 Hz, 1H), 3.61 (ddd, *J* = 8.5, 5.4, 1.6 Hz, 1H), 3.02 (dd, *J* = 17.4, 8.5 Hz, 1H), 2.58 (dd, *J* = 17.4, 1.7 Hz, 1H), 1.98-2.04 (m, 1H), 1.65-1.67 (m, 1H), 1.49–1.56 (m, 3H), 1.50 (d, *J* = 6.5 Hz, 3H), 1.28-1.34 (m, 1H), 1.04-1.13 (m, 3H), 0.86-0.91 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 177.2, 145.1, 138.4, 128.2, 125.9, 88.8, 70.1, 43.9, 38.7, 37.9, 37.8, 30.1, 27.8, 26.2, 25.3, 25.2. **IR** (neat) V_{max} (cm^{-1}): 3274, 2924, 1754, 1189. **HRMS** (ESI): calc for $C_{18}H_{24}O_3$ m/z: 288.1725 found $[M+H]^+$ 289.1799. **MP**: 72-74˚C

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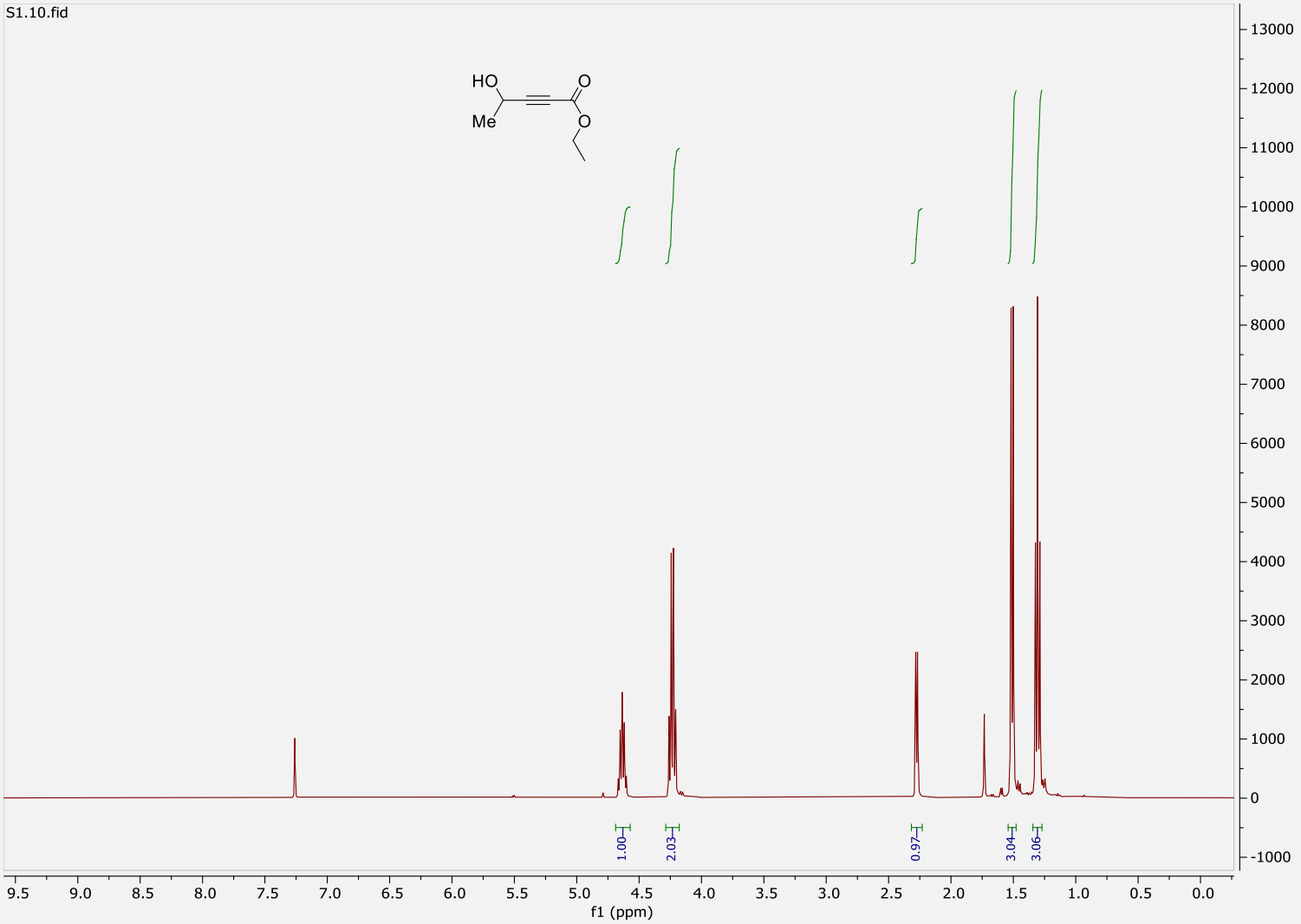
^{1}H and ^{13}C

NMR Spectra

For

Alkynoates and Butenolides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11000
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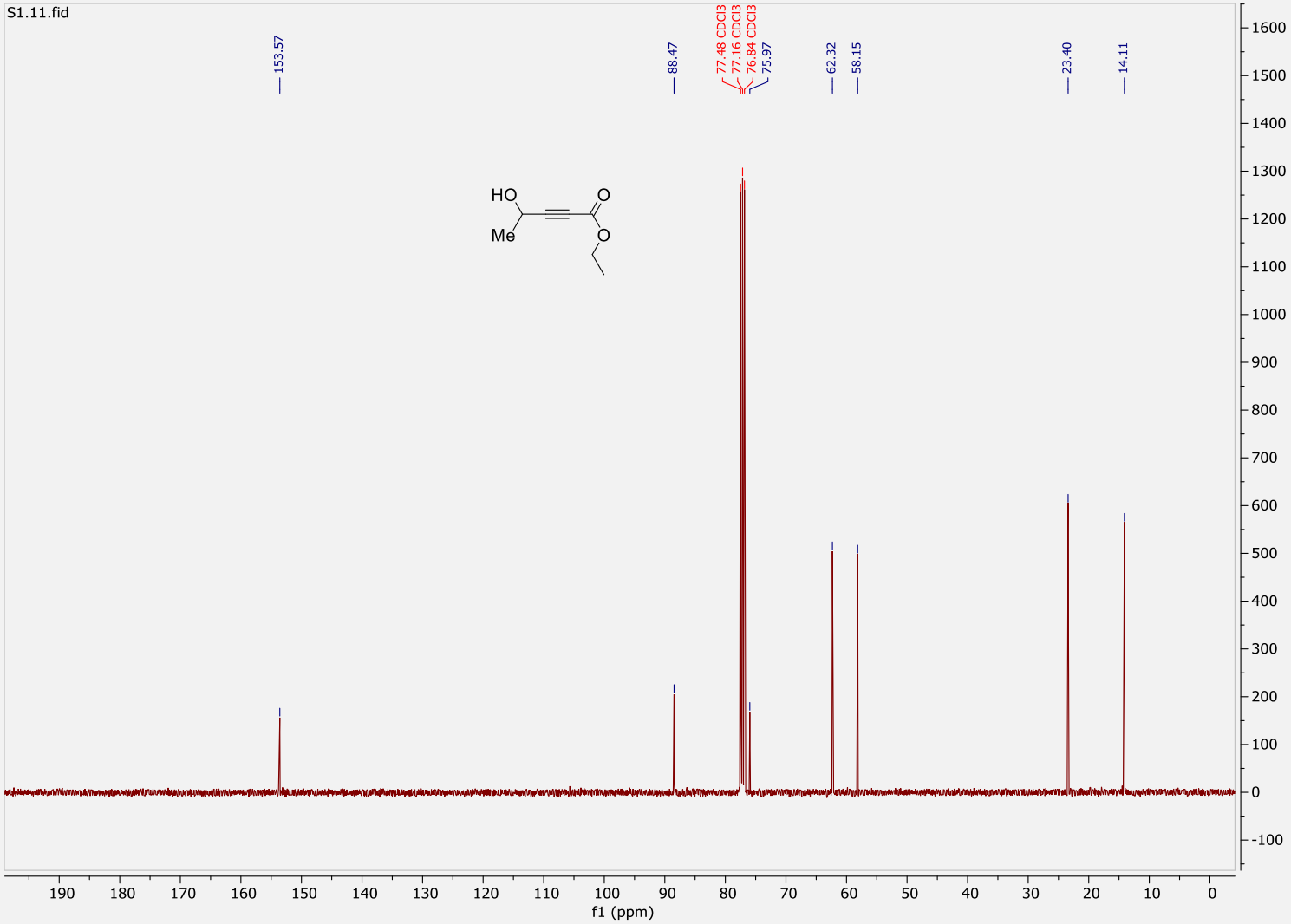
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
1.00
2.03
0.97
3.04
3.06
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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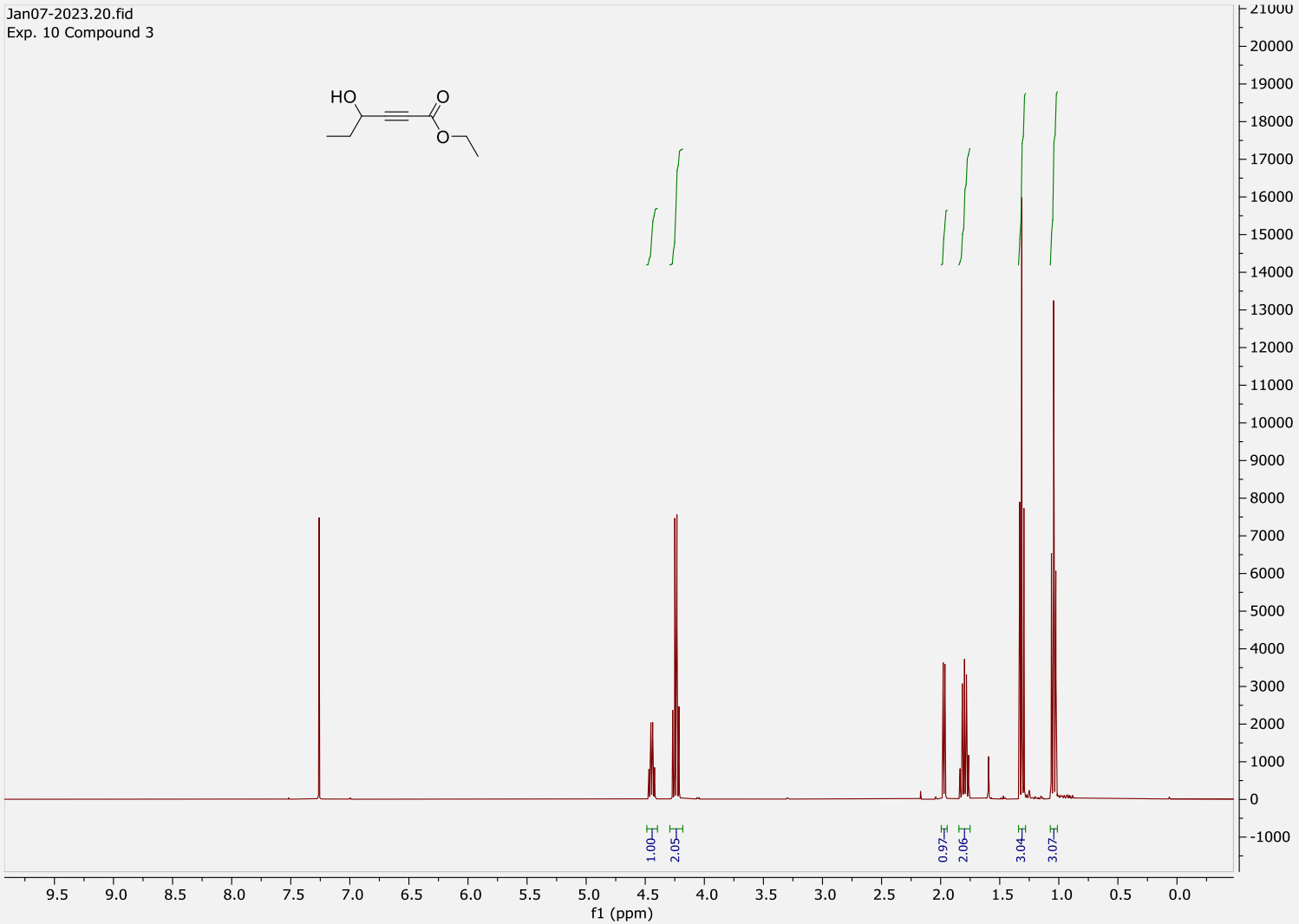
88.47
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
75.97
62.32
58.15
23.40
14.11
HO
Me
O
O
f1 (ppm)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O
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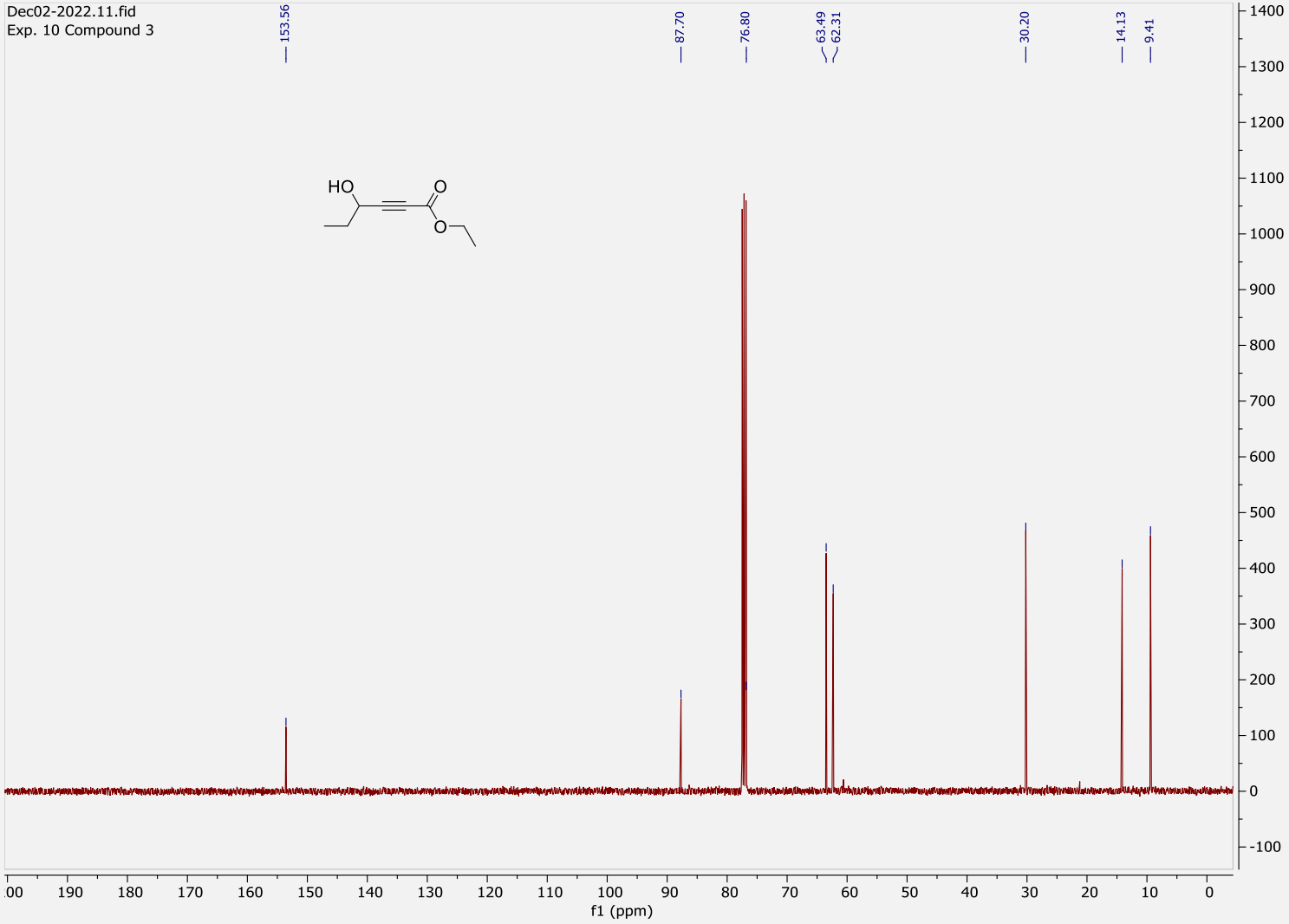
2.06
3.04
3.07
f1 (ppm)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63.49
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30.20
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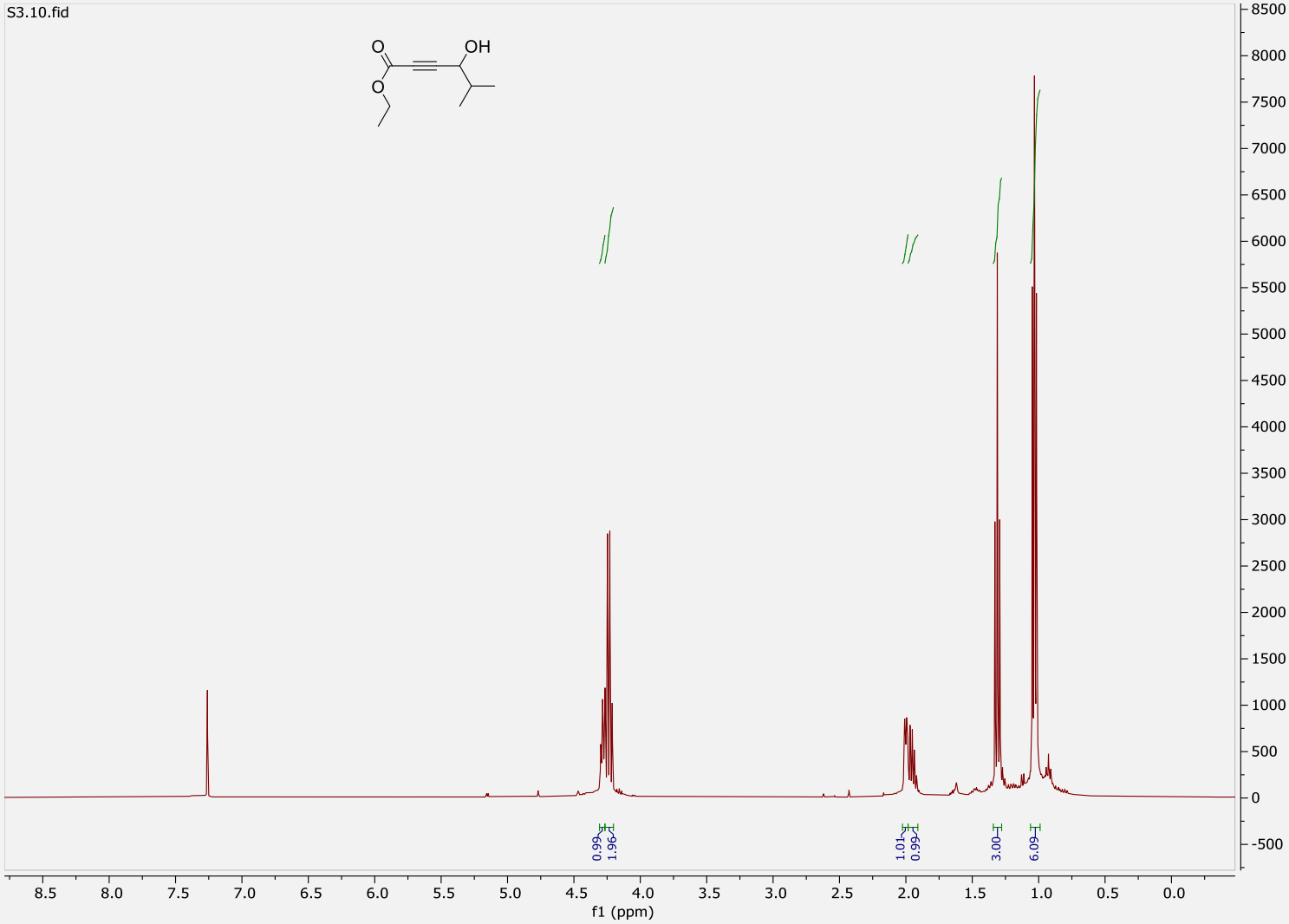
O
O
f1 (ppm)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1.96
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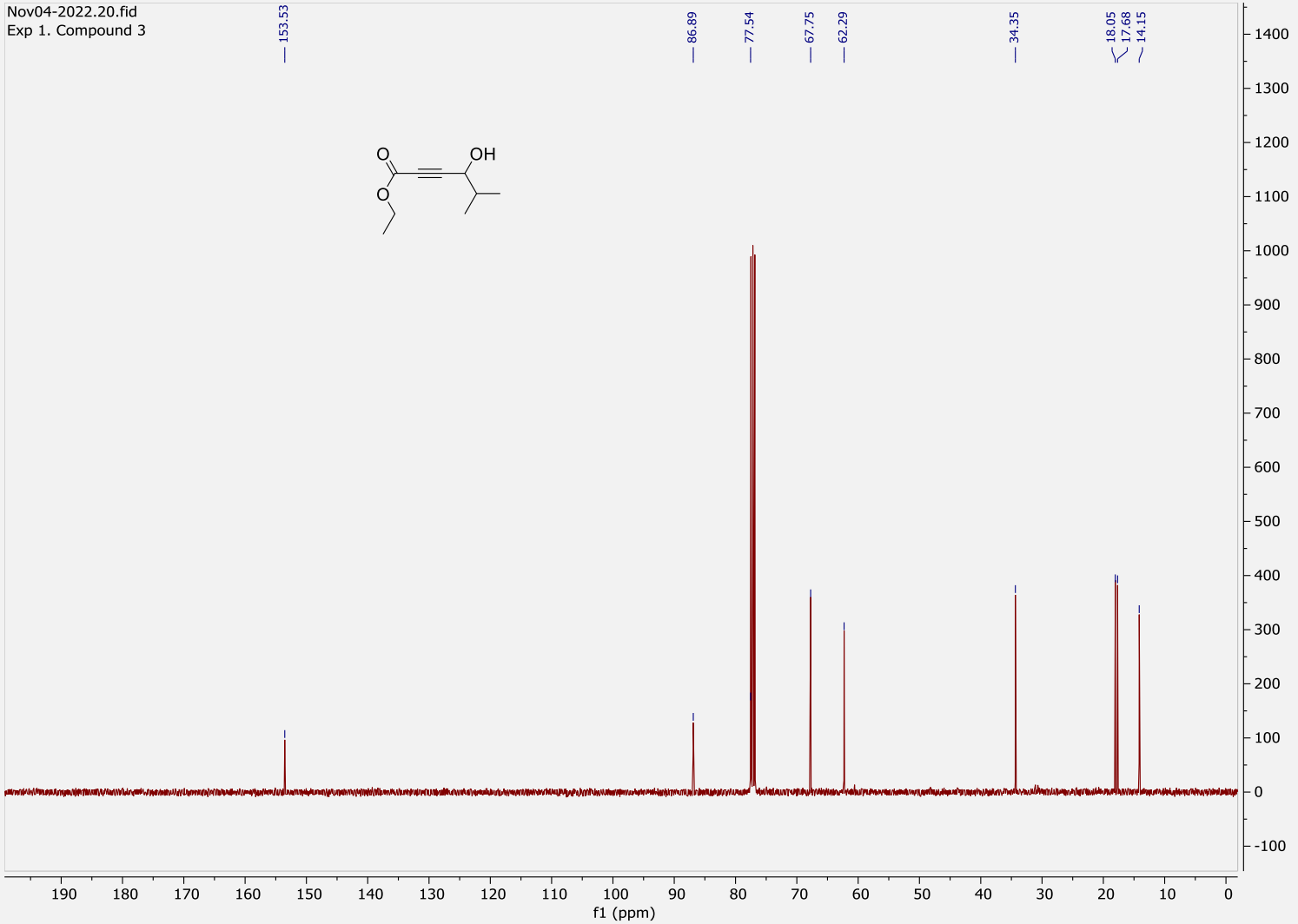
6.09
f1 (ppm)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77.54
67.75
62.29
34.35
18.05
17.68
14.15
O
OH
O
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
190
180
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110
100
90
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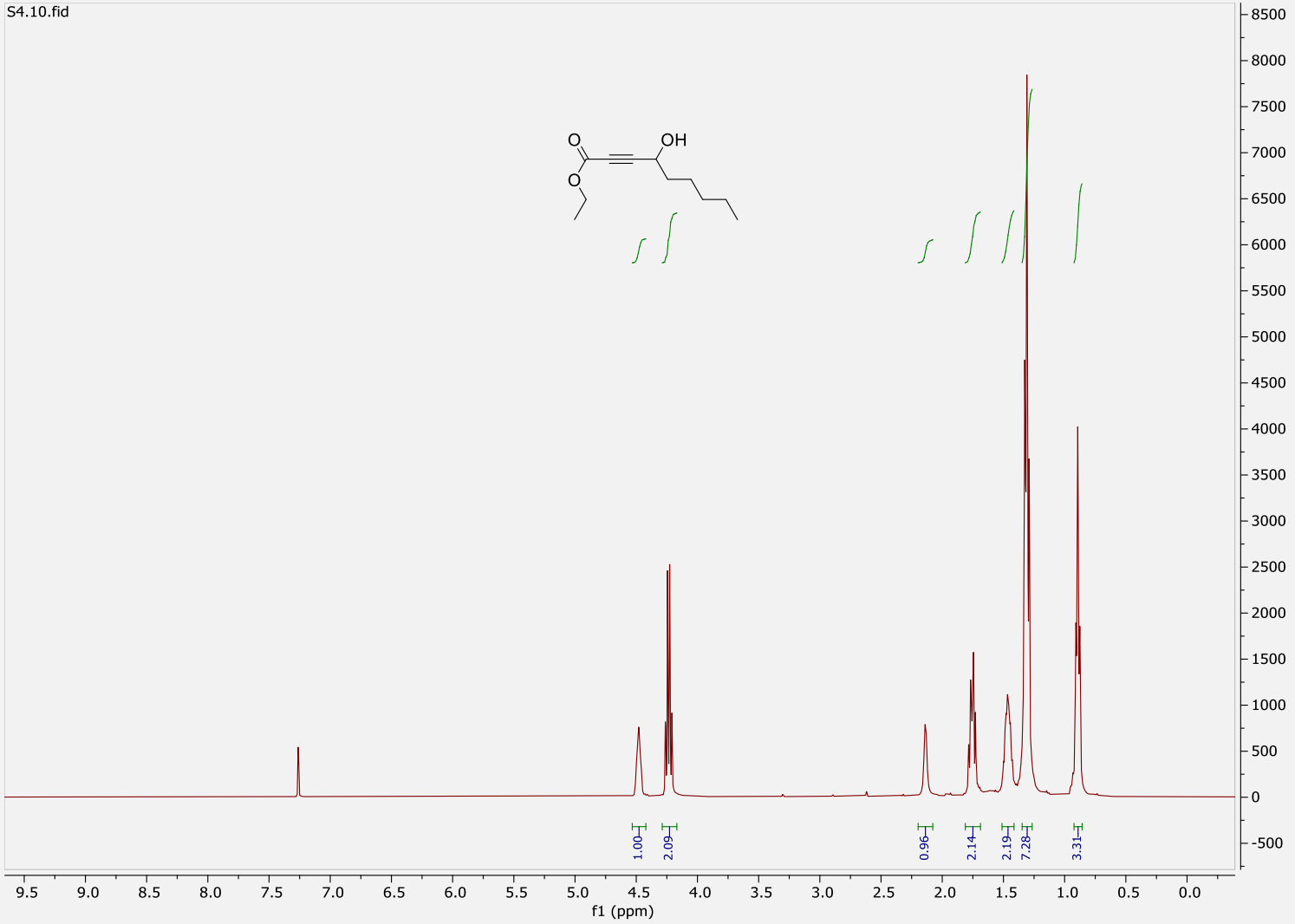
70
60
50
40
30
20
10
0
f1 (ppm)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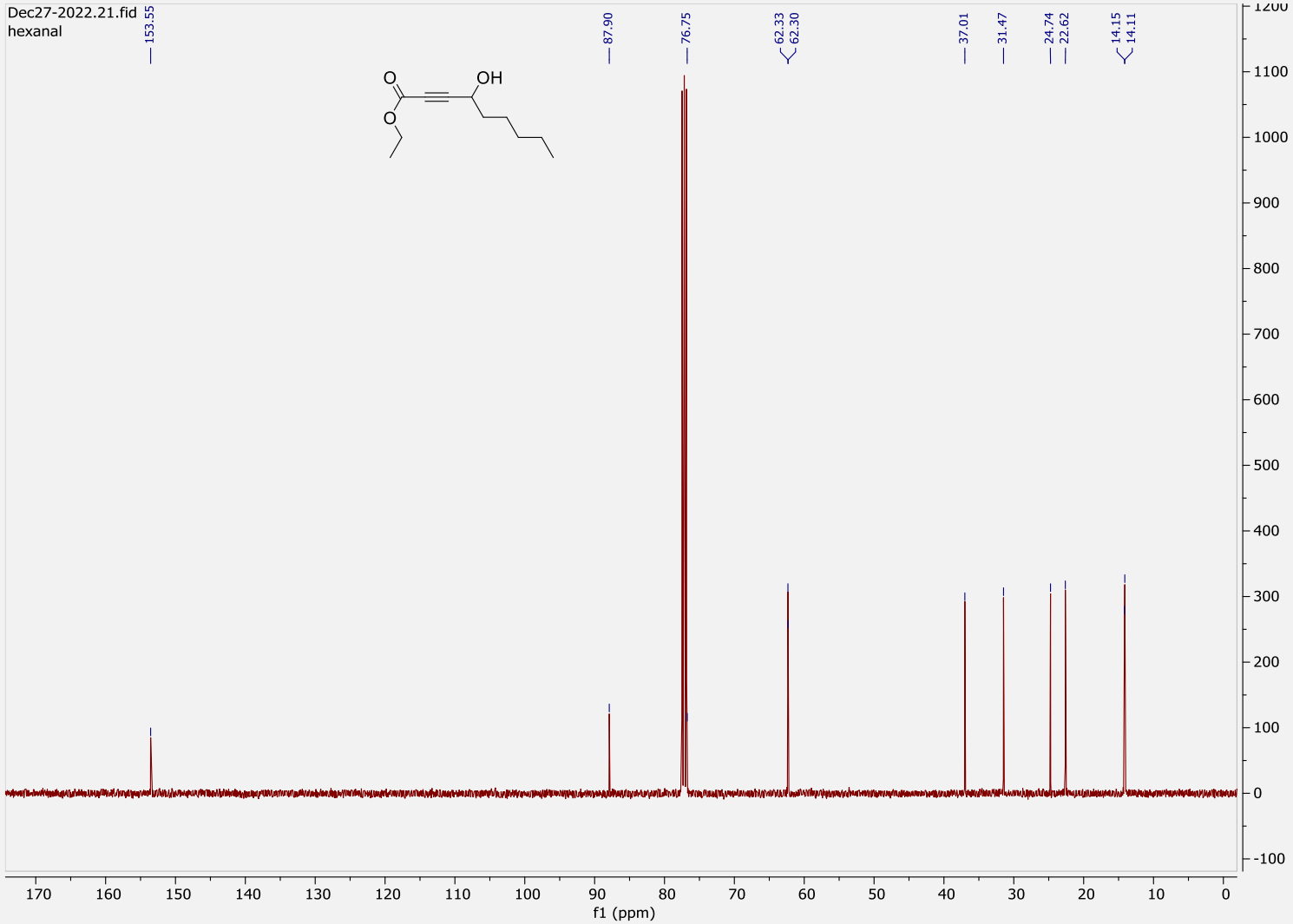
f1 (ppm)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62.30
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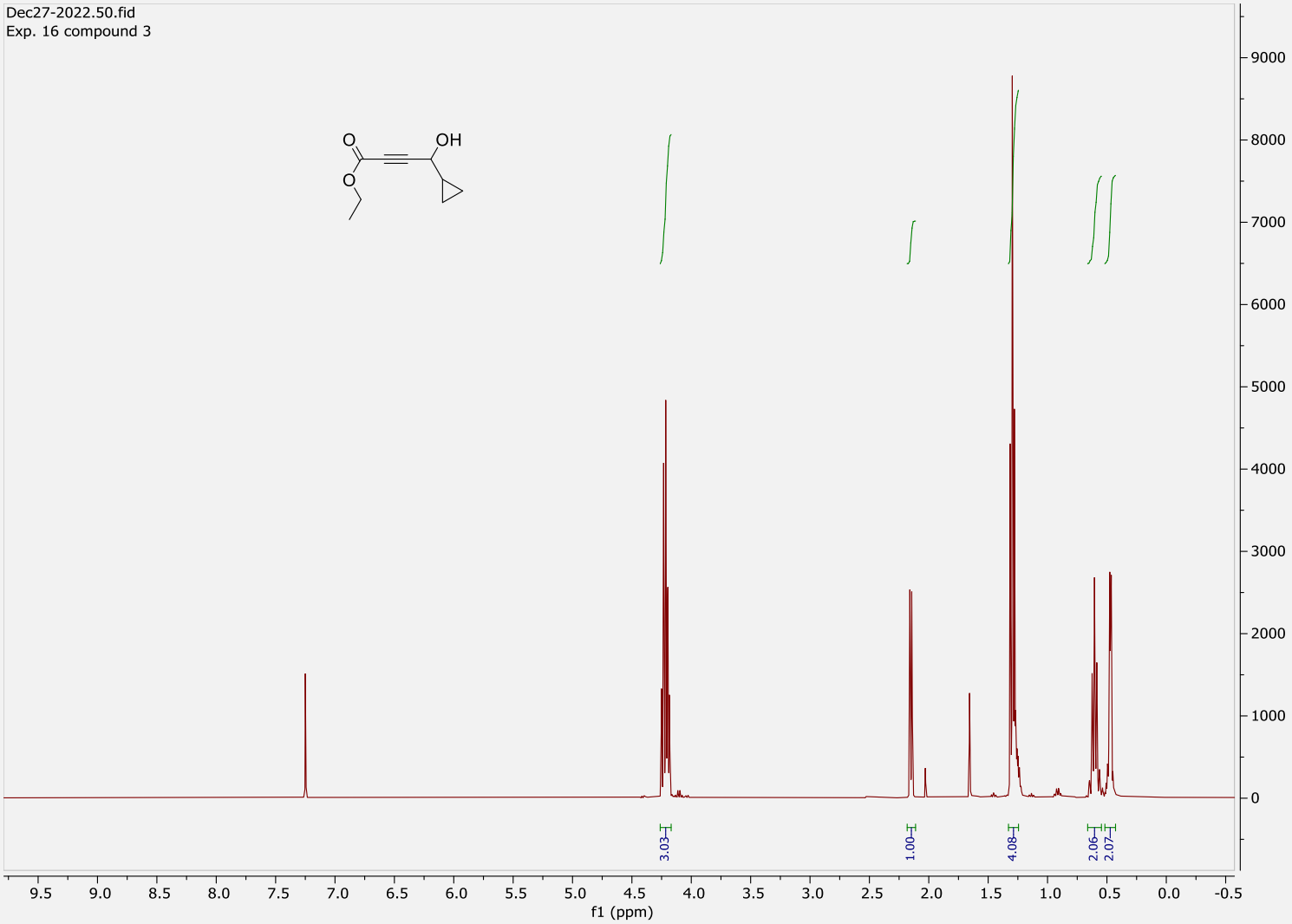
31.47
24.74
22.62
14.15
14.11
O
OH
O
f1 (ppm)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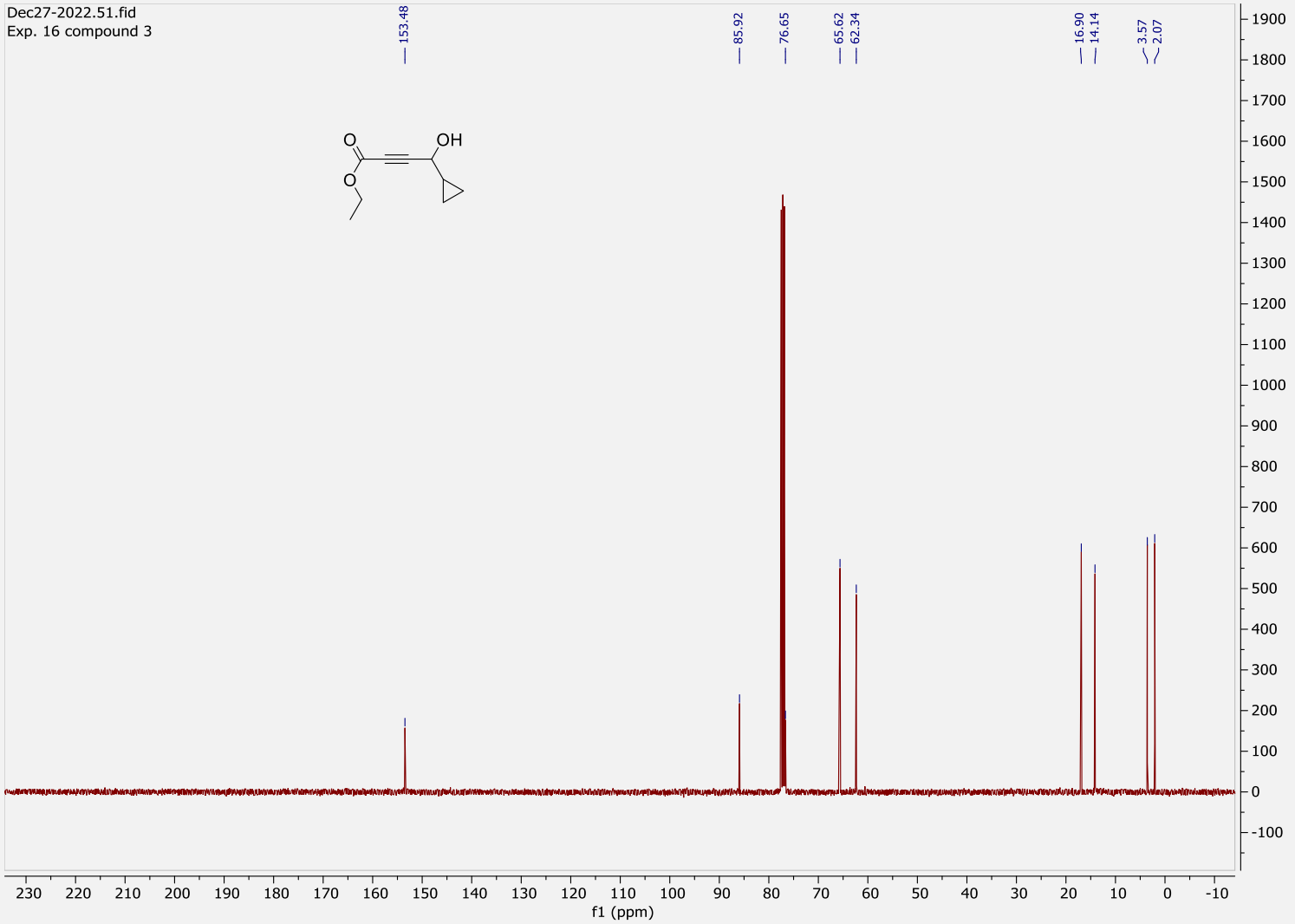
7000
6000
5000
4000
3000
2000
1000
0
3.03
1.00
4.08
2.06
2.07
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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65.62
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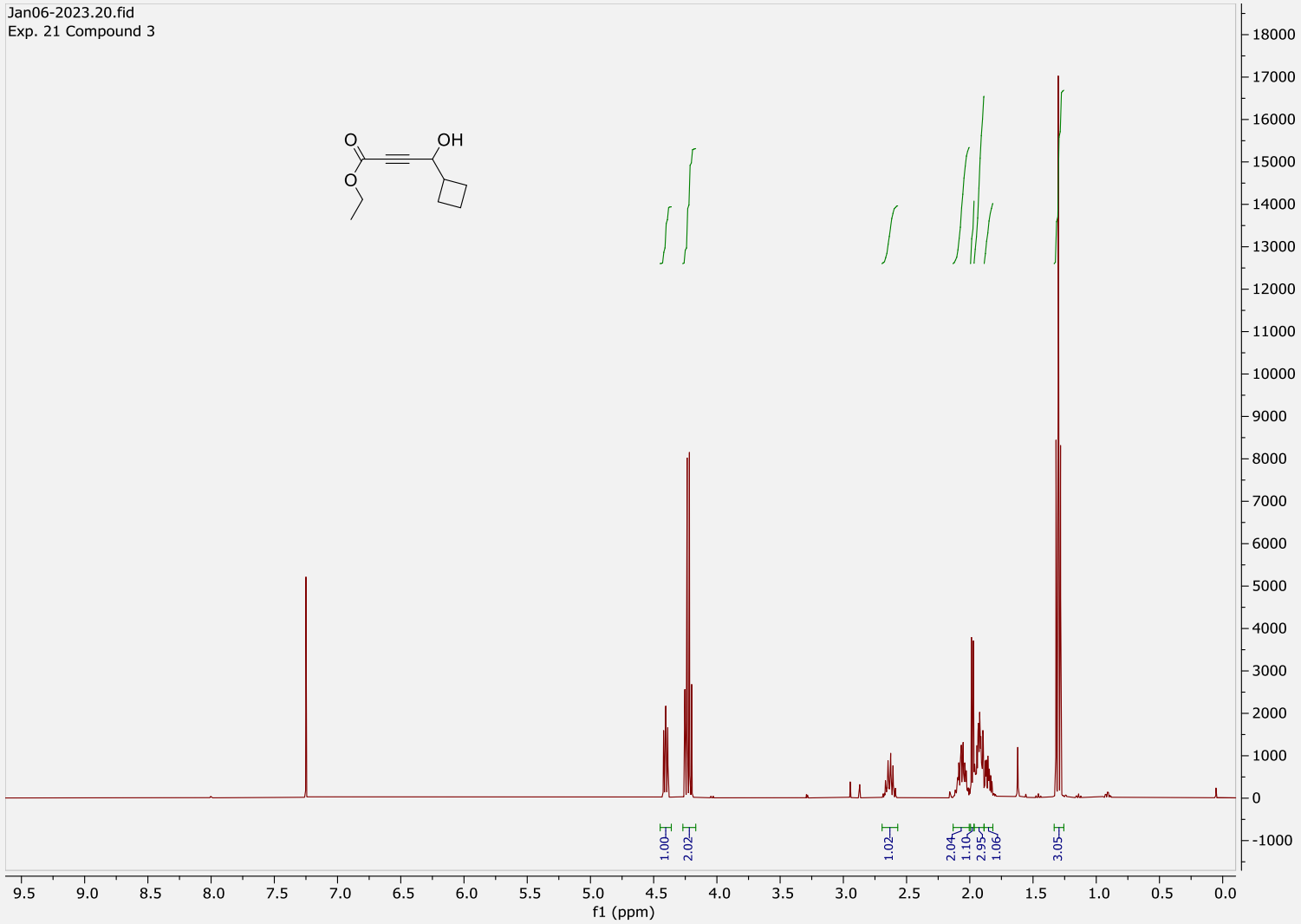
16.90
14.14
3.57
2.07
O
OH
O
f1 (ppm)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OH
O
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
1.00
2.02
1.02
2.04
1.10
2.95
1.06
3.05
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
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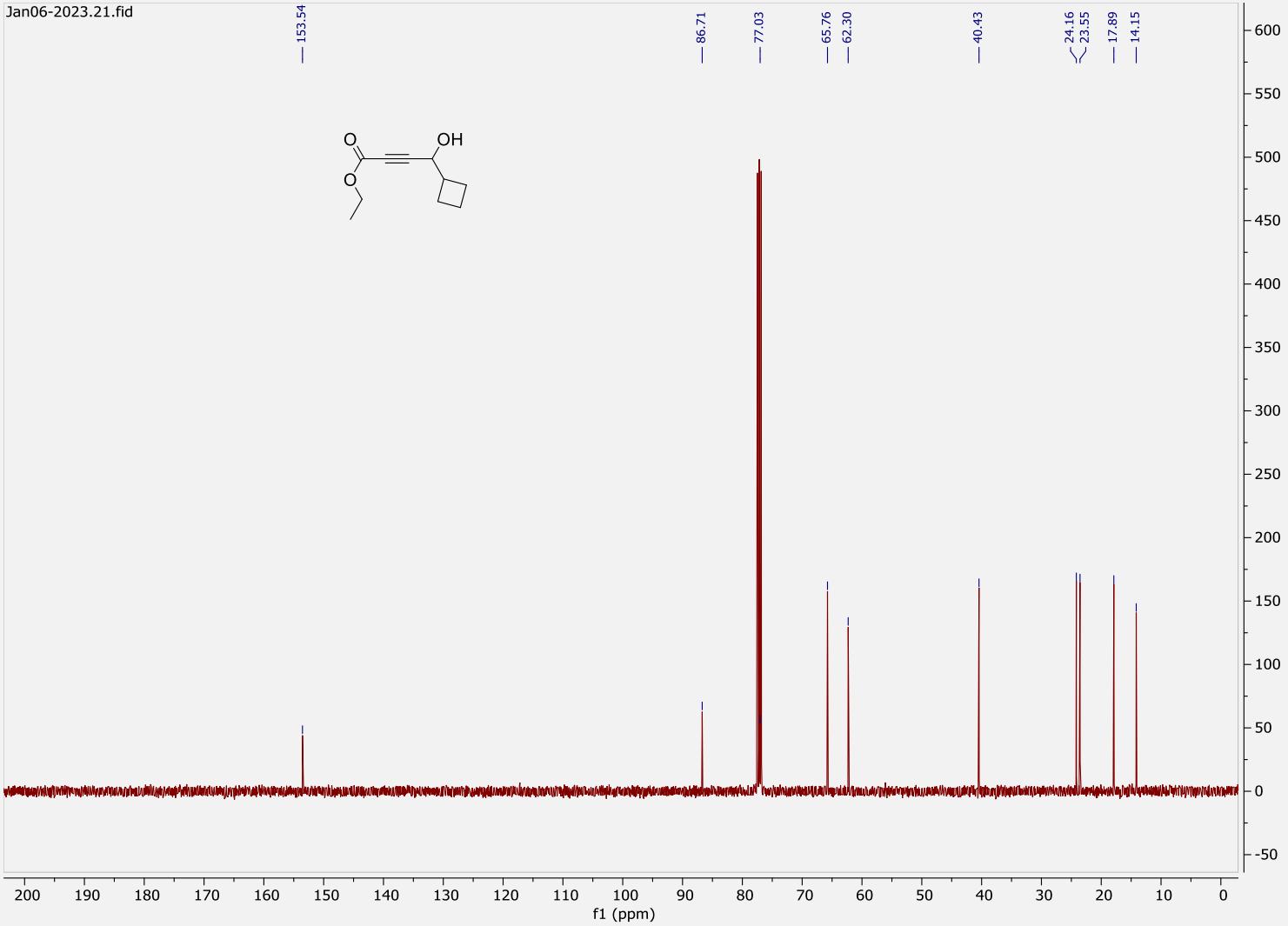
1.0
0.5
0.0
f1 (ppm)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23.55
17.89
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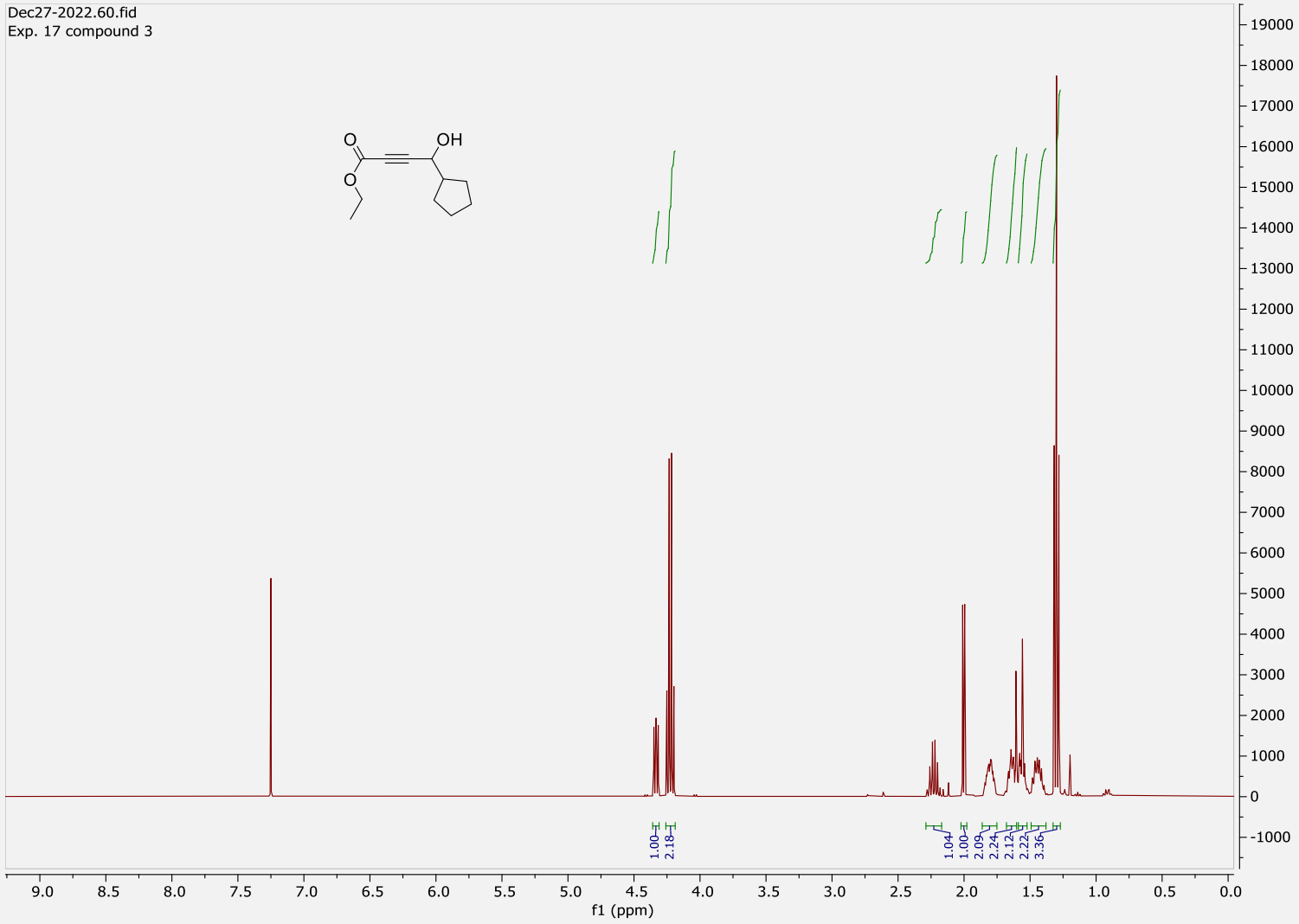
O
OH
O
f1 (ppm)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OH
O
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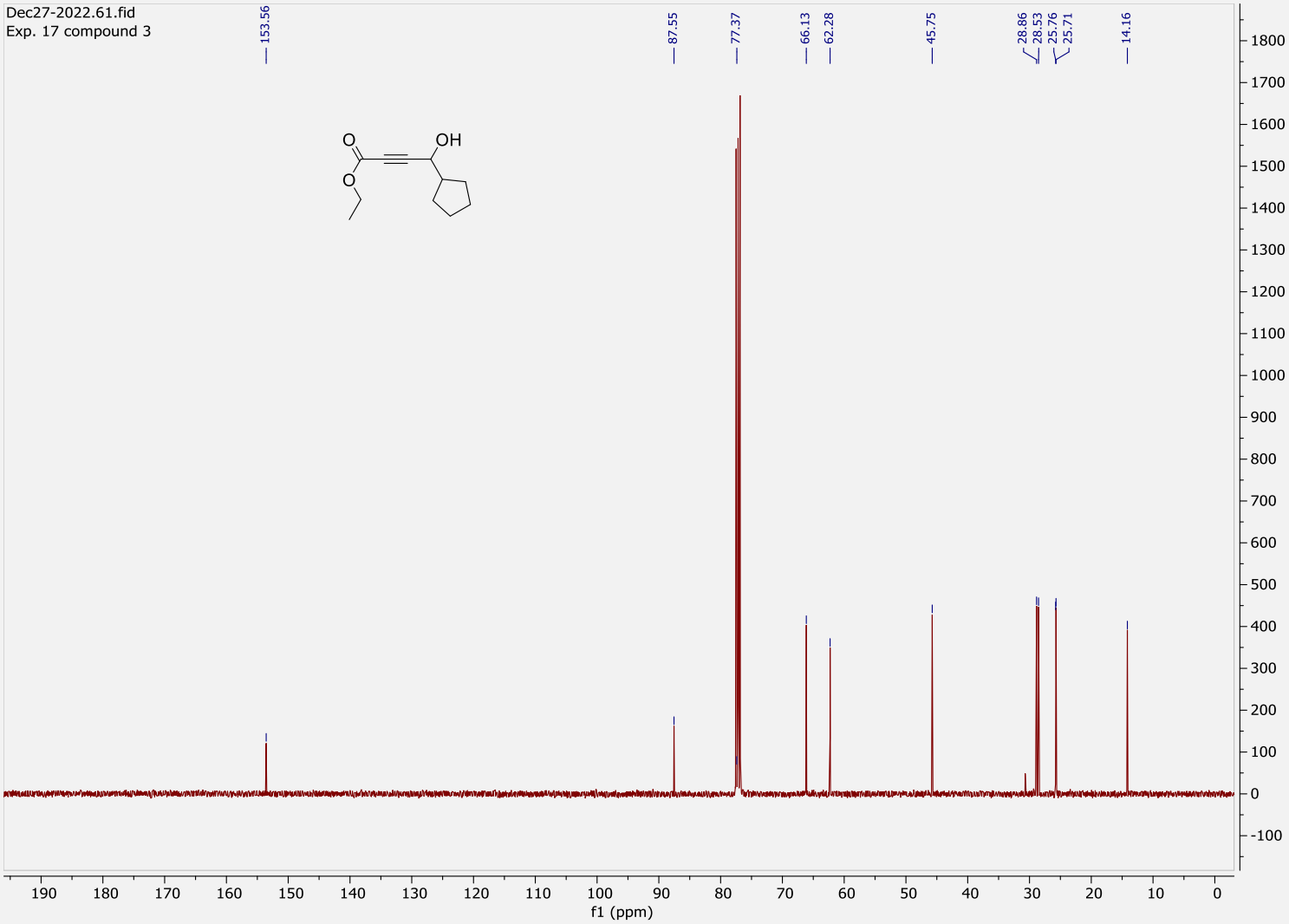
1.00
2.09
2.24
2.12
2.22
3.36
f1 (ppm)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87.55
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66.13
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45.75
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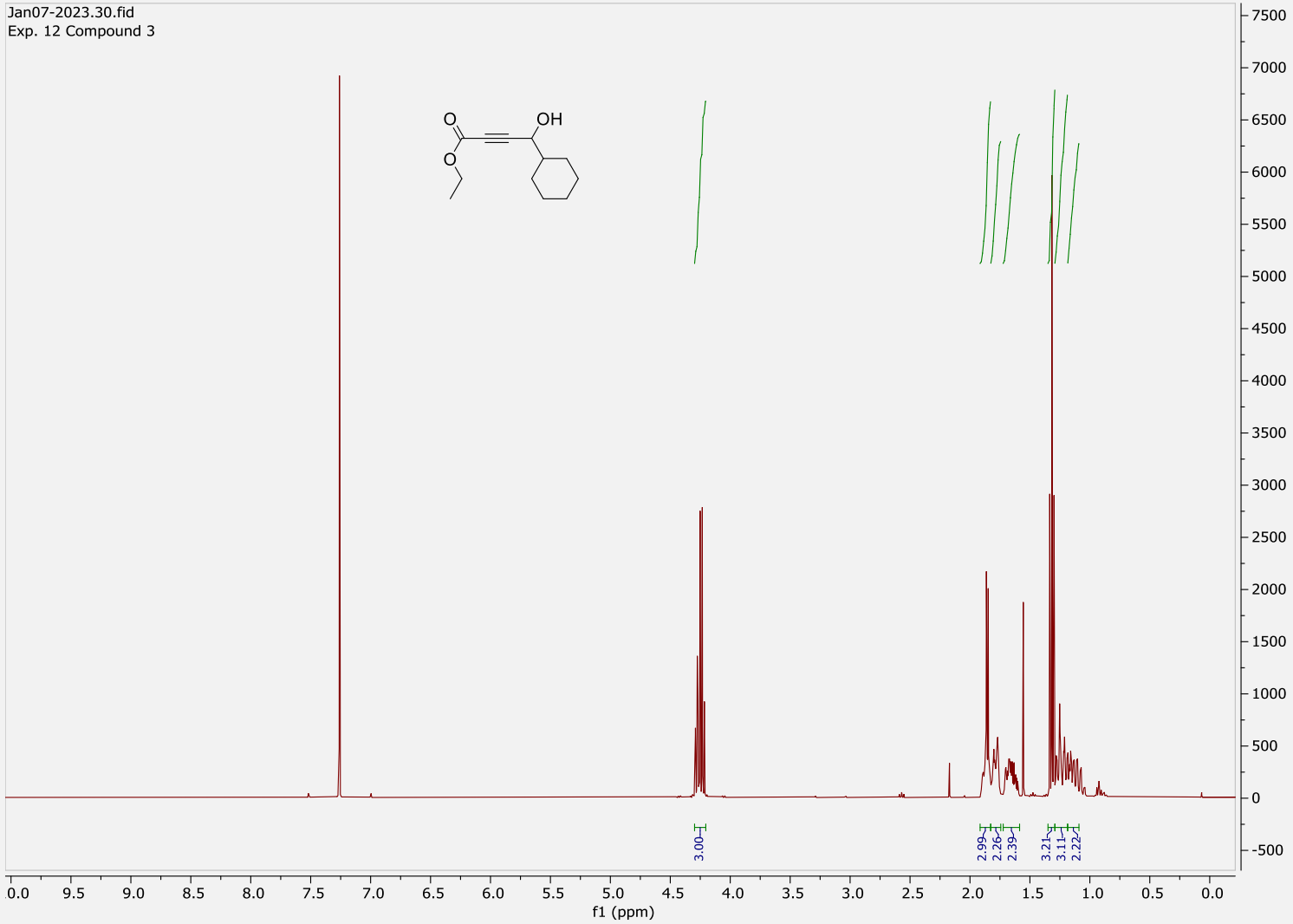
25.76
25.71
14.16
O
OH
O
f1 (ppm)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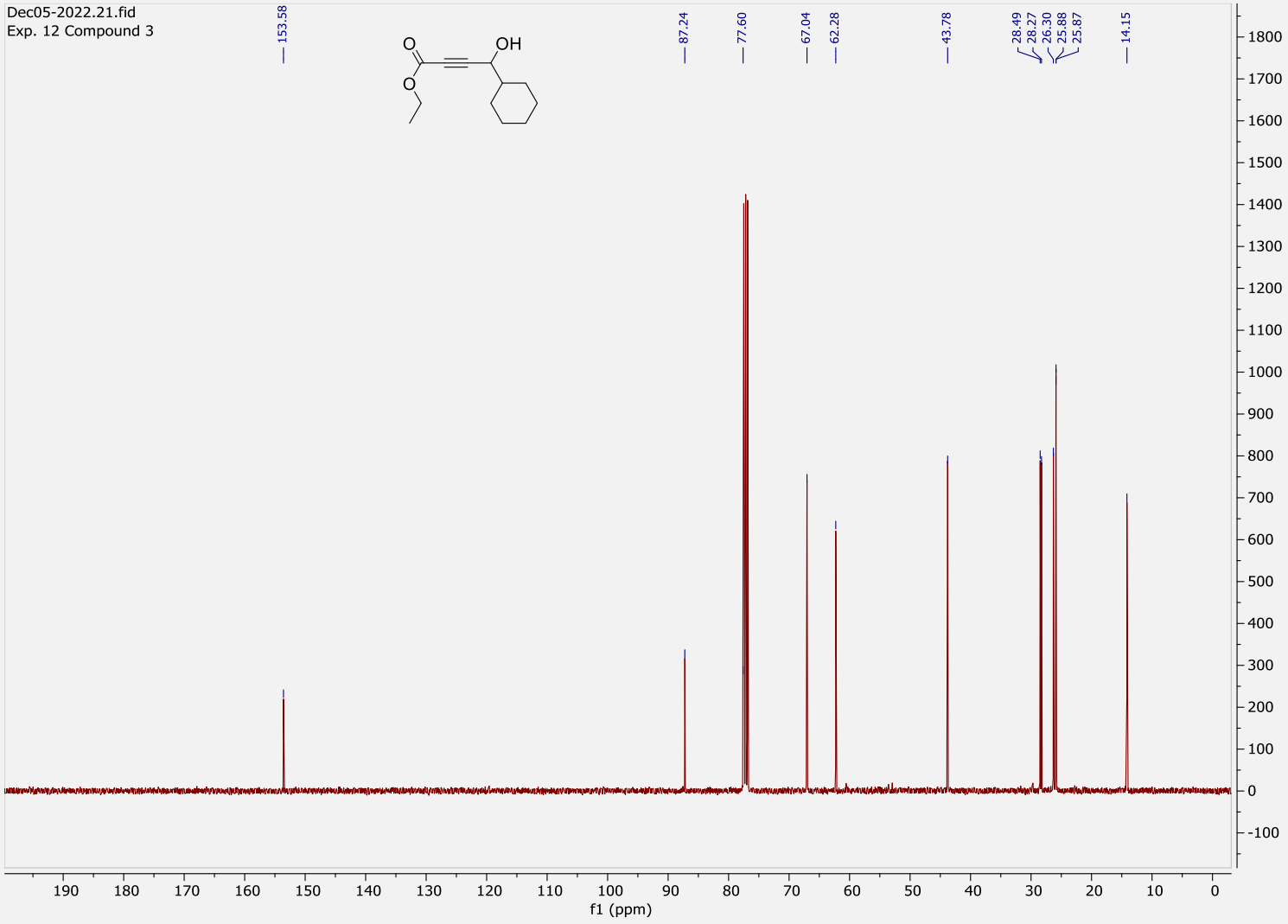
2.39
3.21
3.11
2.22
f1 (ppm)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43.78
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28.27
26.30
25.88
25.87
14.15
O
OH
O
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1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
190
180
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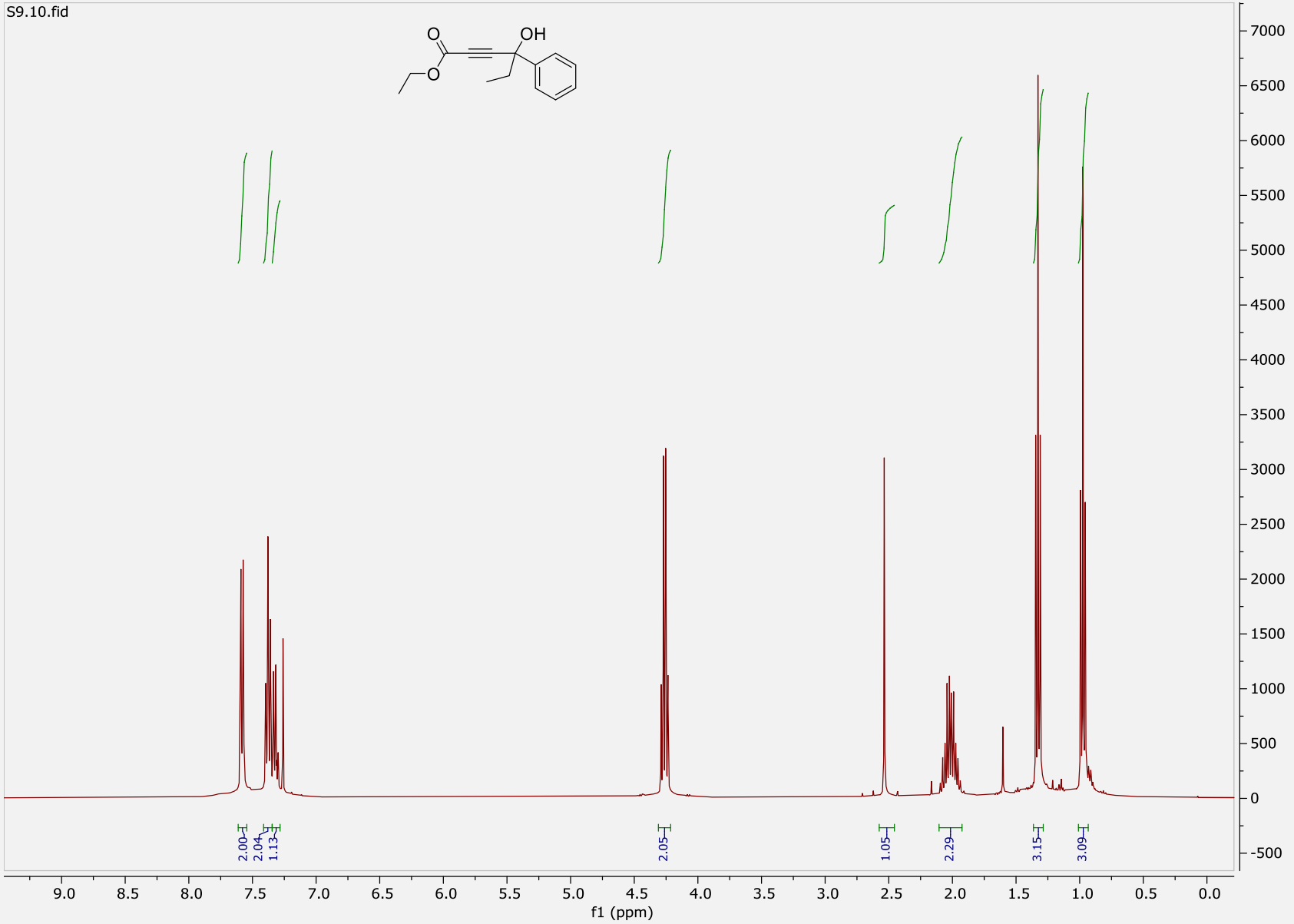
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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OH
O
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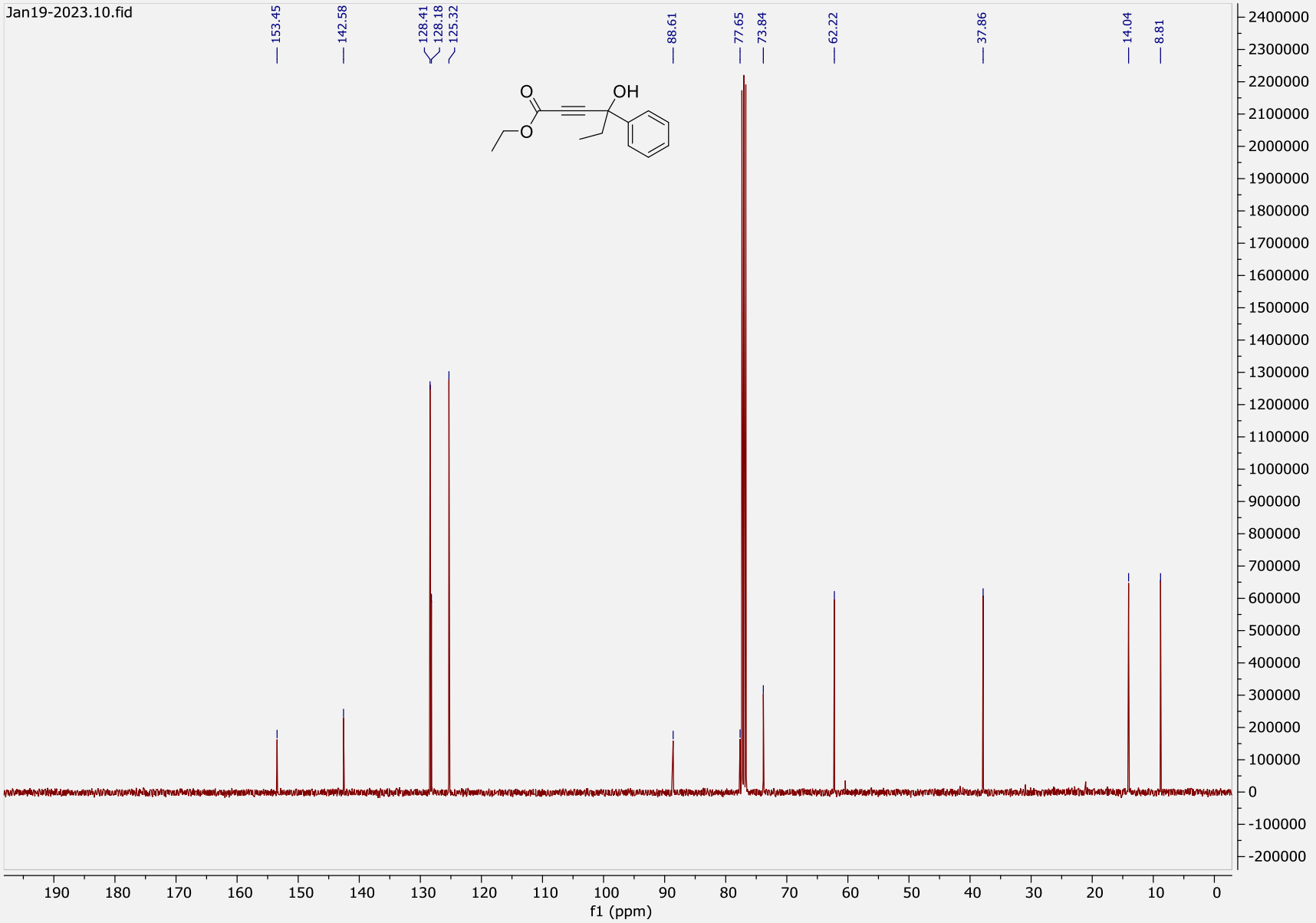
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
2.00
2.04
1.13
2.05
1.05
2.29
3.15
3.09
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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73.84
62.22
37.86
14.04
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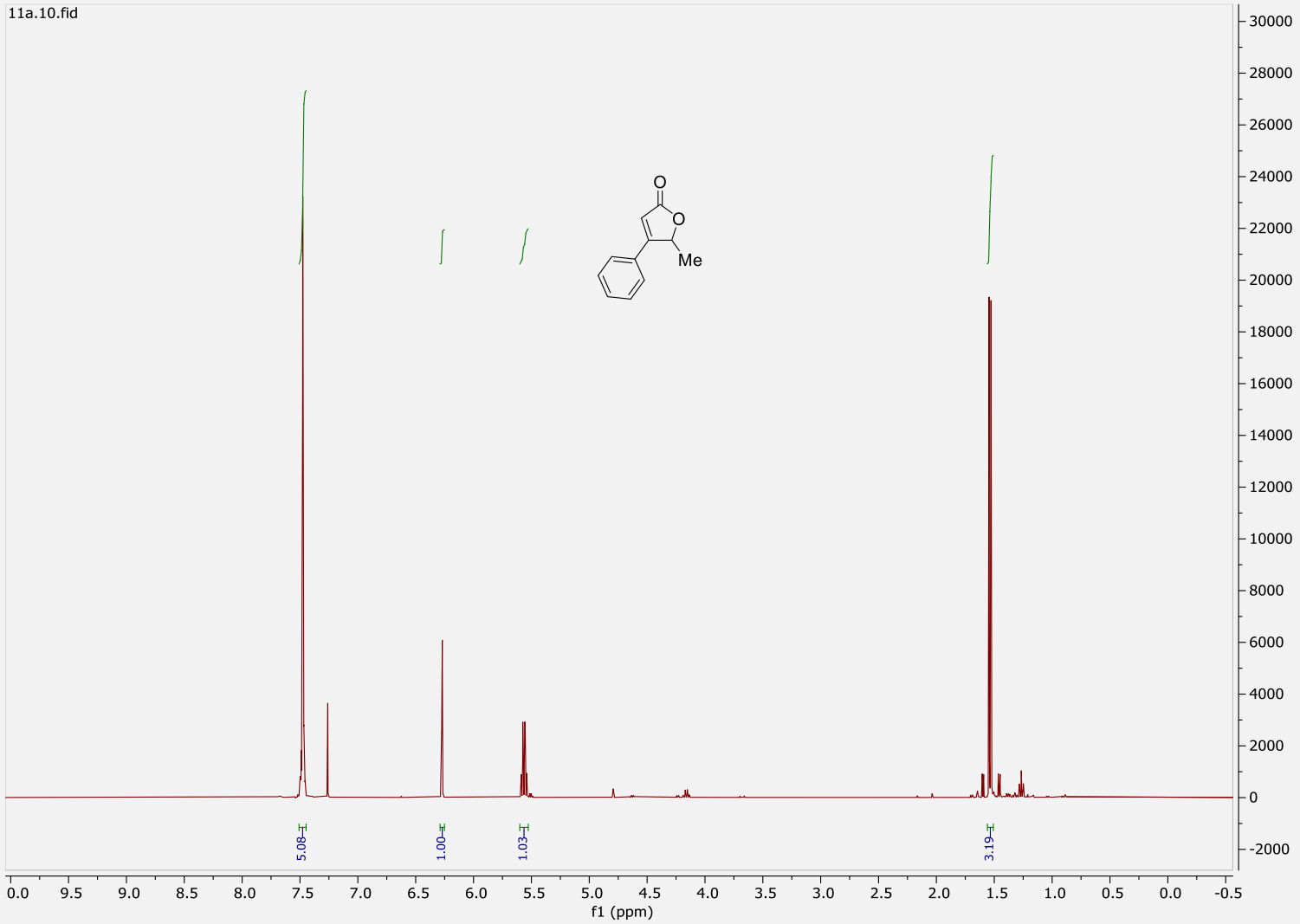
O
OH
O
f1 (ppm)


11a.10.fid
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O
Me
5.08
1.00
1.03
3.19
f1 (ppm)


11a.11.fid
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169.01
131.43
130.08
129.37
127.33
113.88
78.76
19.94
19.70
rac-
O
O
Me
2500
2000
1500
1000
500
0
190
180
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150
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130
120
110
100
90
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70
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50
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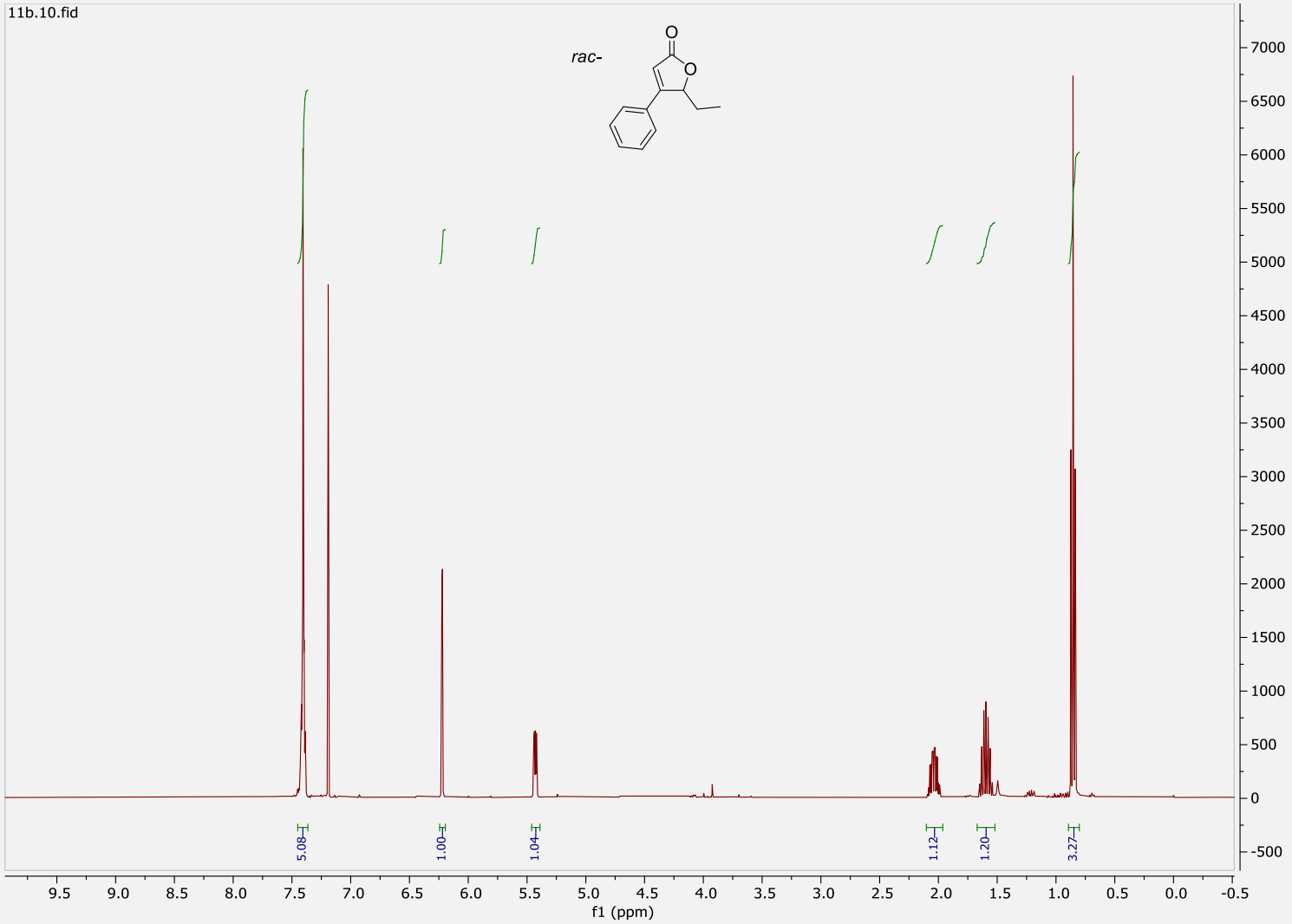
10
0
f1 (ppm)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5500
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4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
5.08
1.00
1.04
1.12
1.20
3.27
9.5
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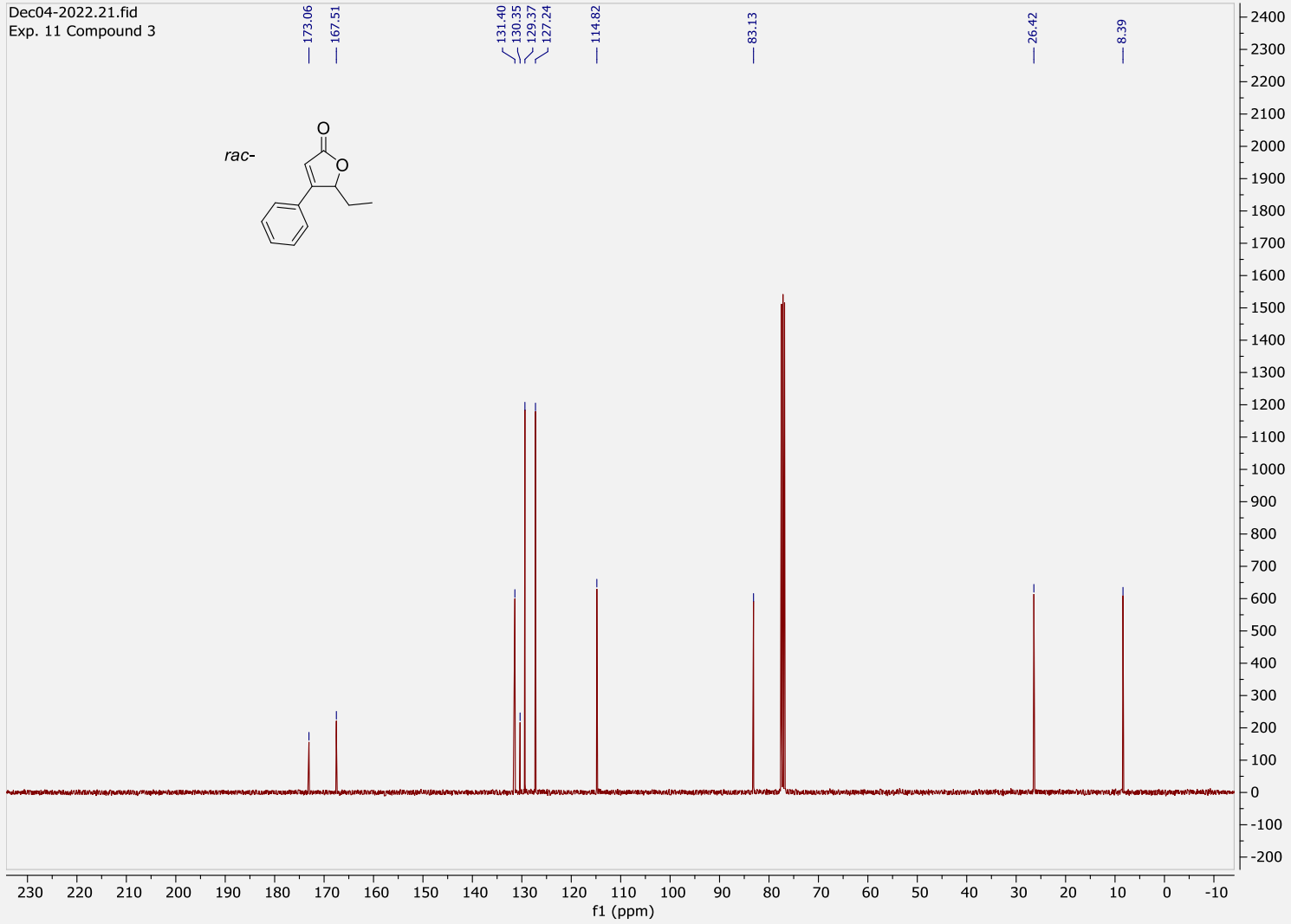
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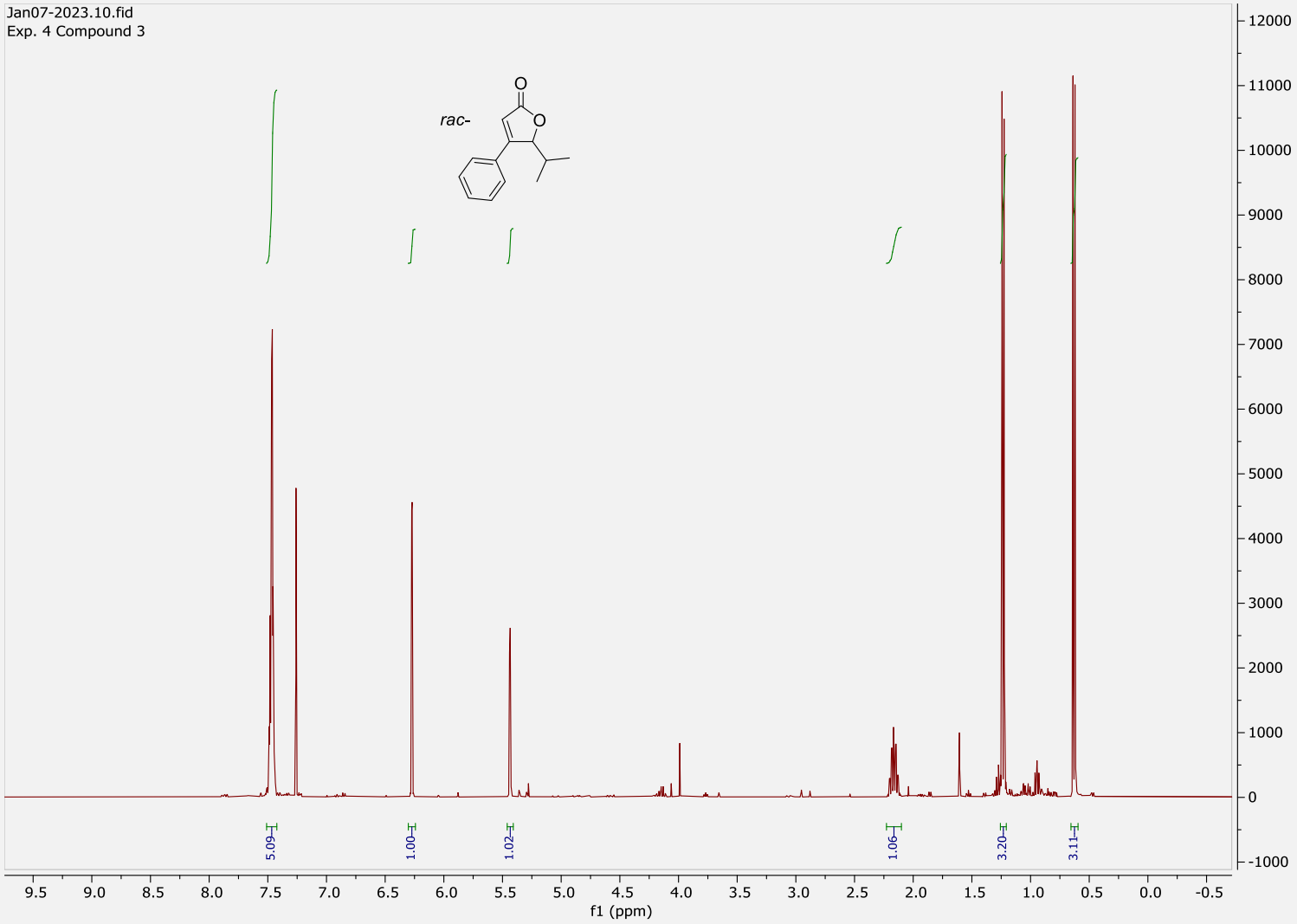
8.39
rac-
O
O
f1 (ppm)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O
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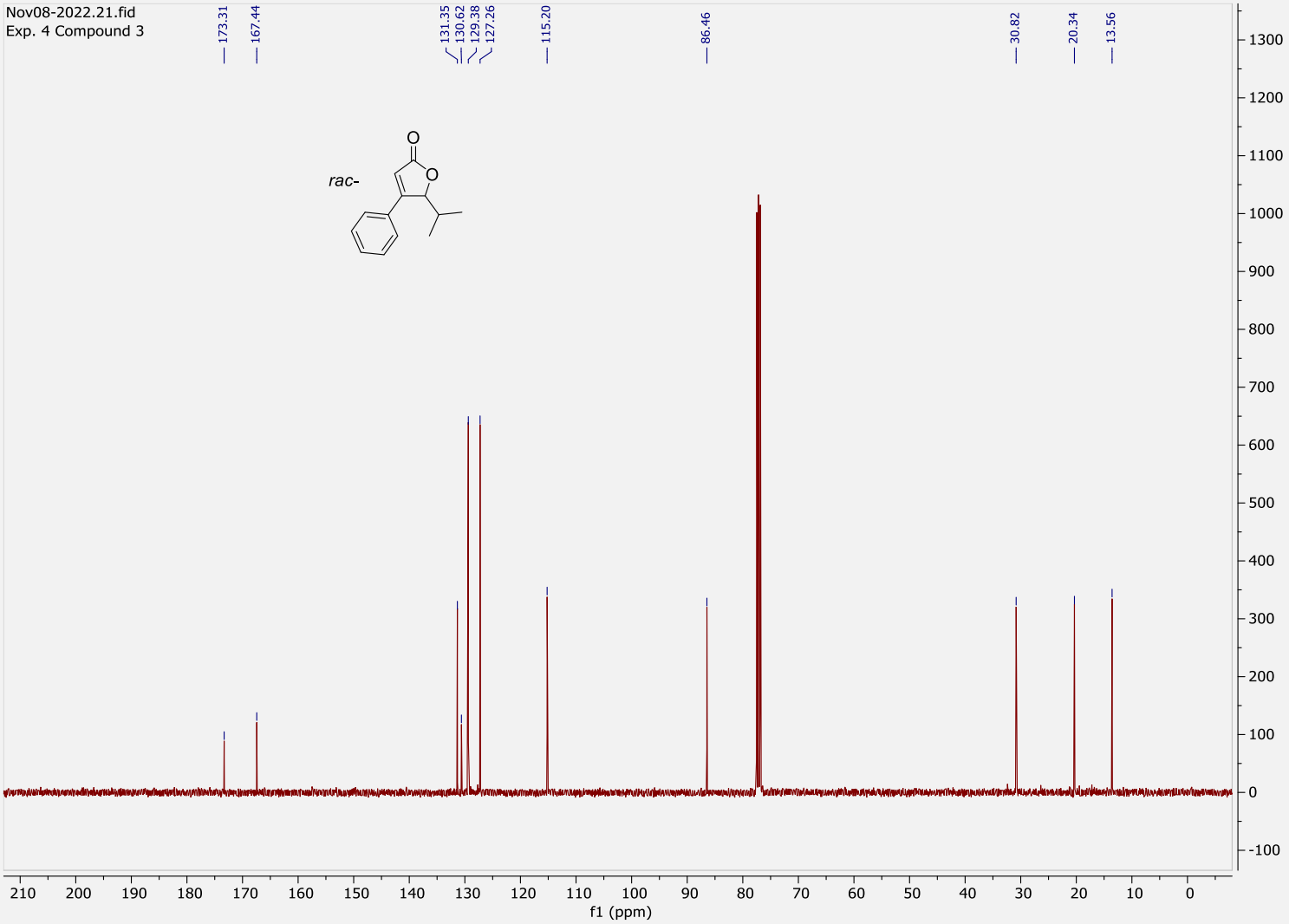
1.00
1.02
1.06
3.20
3.11
f1 (ppm)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130.62
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127.26
115.20
86.46
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20.34
13.56
rac-
O
O
1300
1200
1100
1000
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800
700
600
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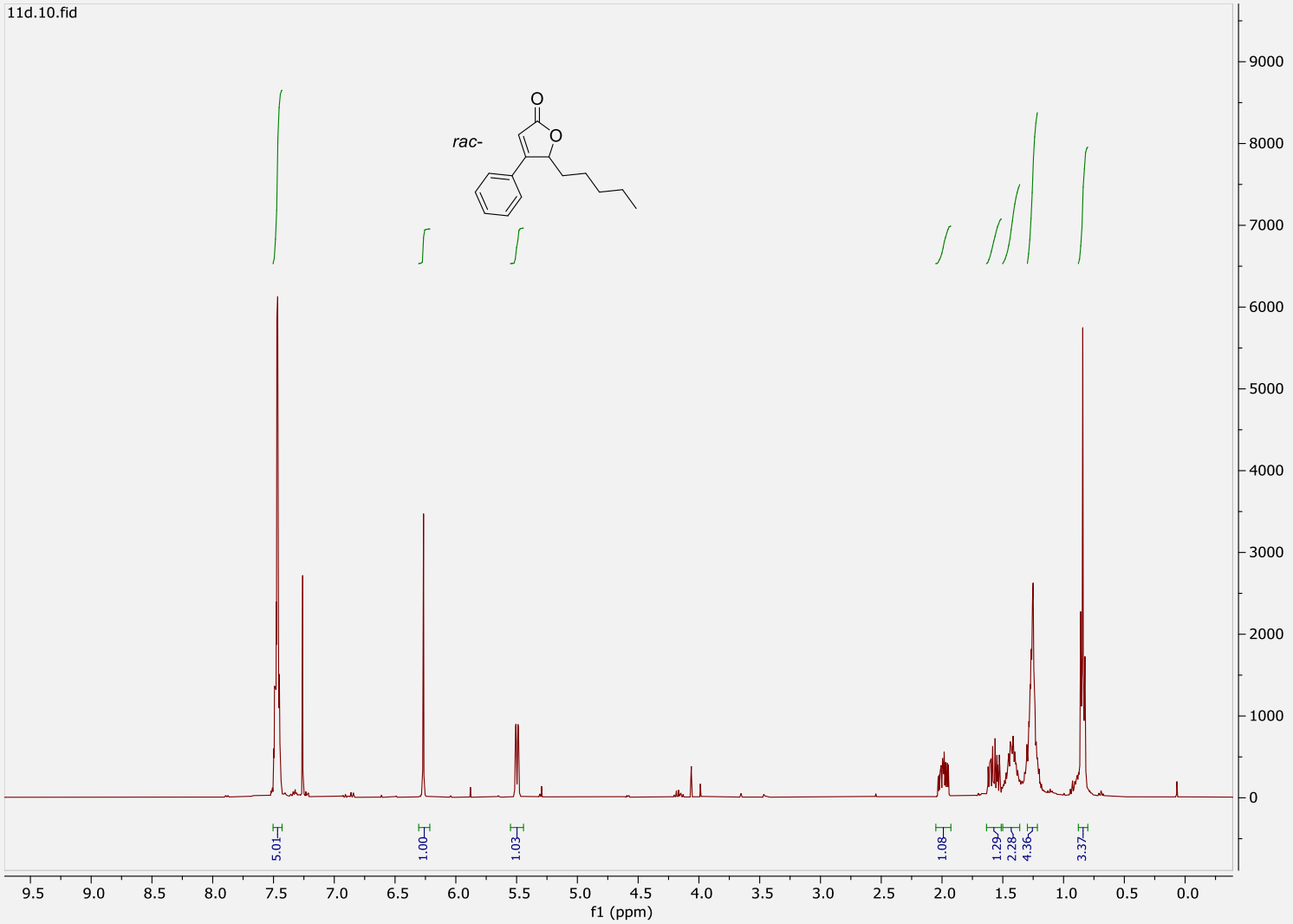
f1 (ppm)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rac-
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O
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1000
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1.00
1.03
1.08
1.29
2.28
4.36
3.37
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.0
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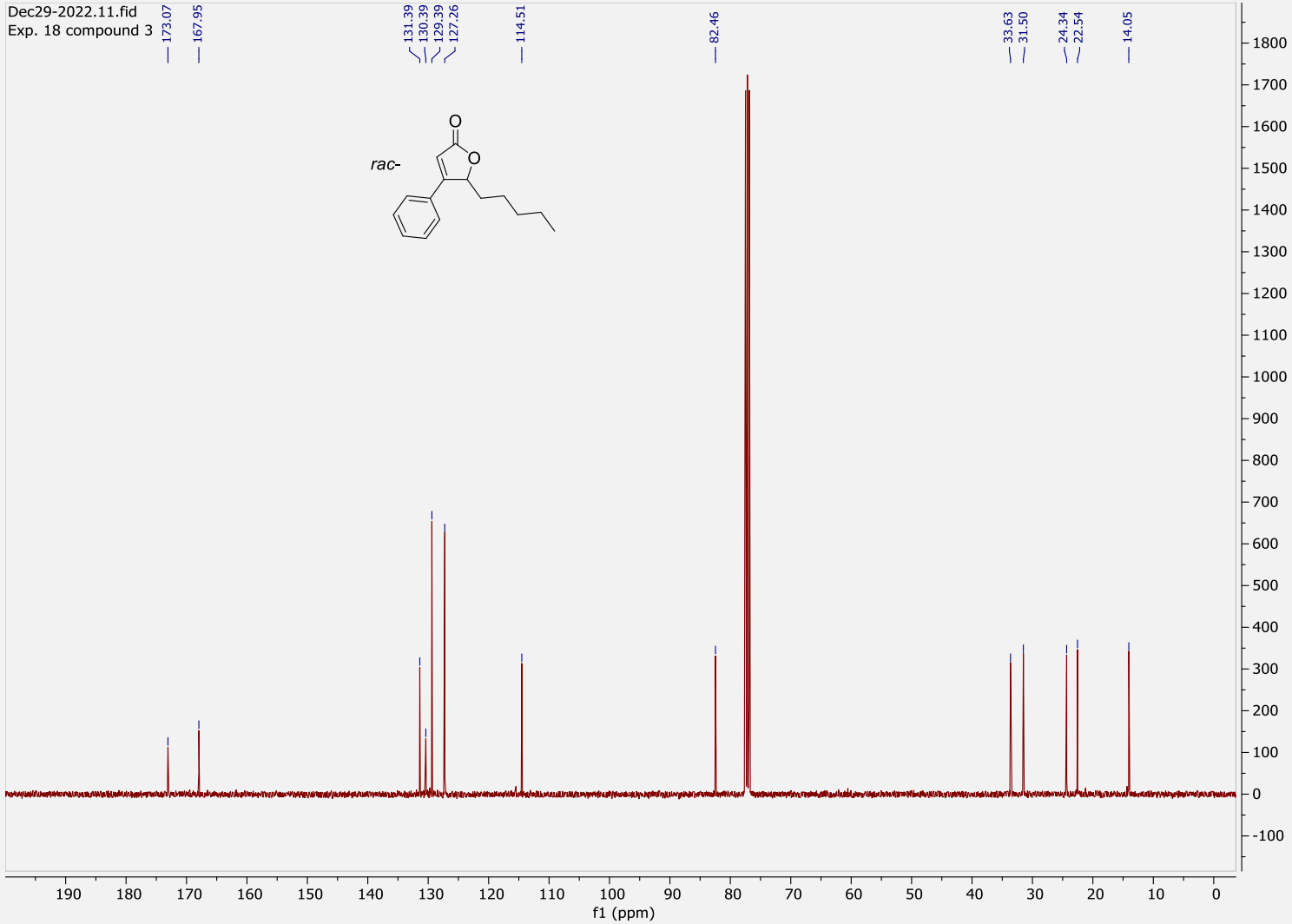
0.0
f1 (ppm)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114.51
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31.50
24.34
22.54
14.05
rac-
O
O
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
190
180
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120
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100
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70
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50
40
30
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10
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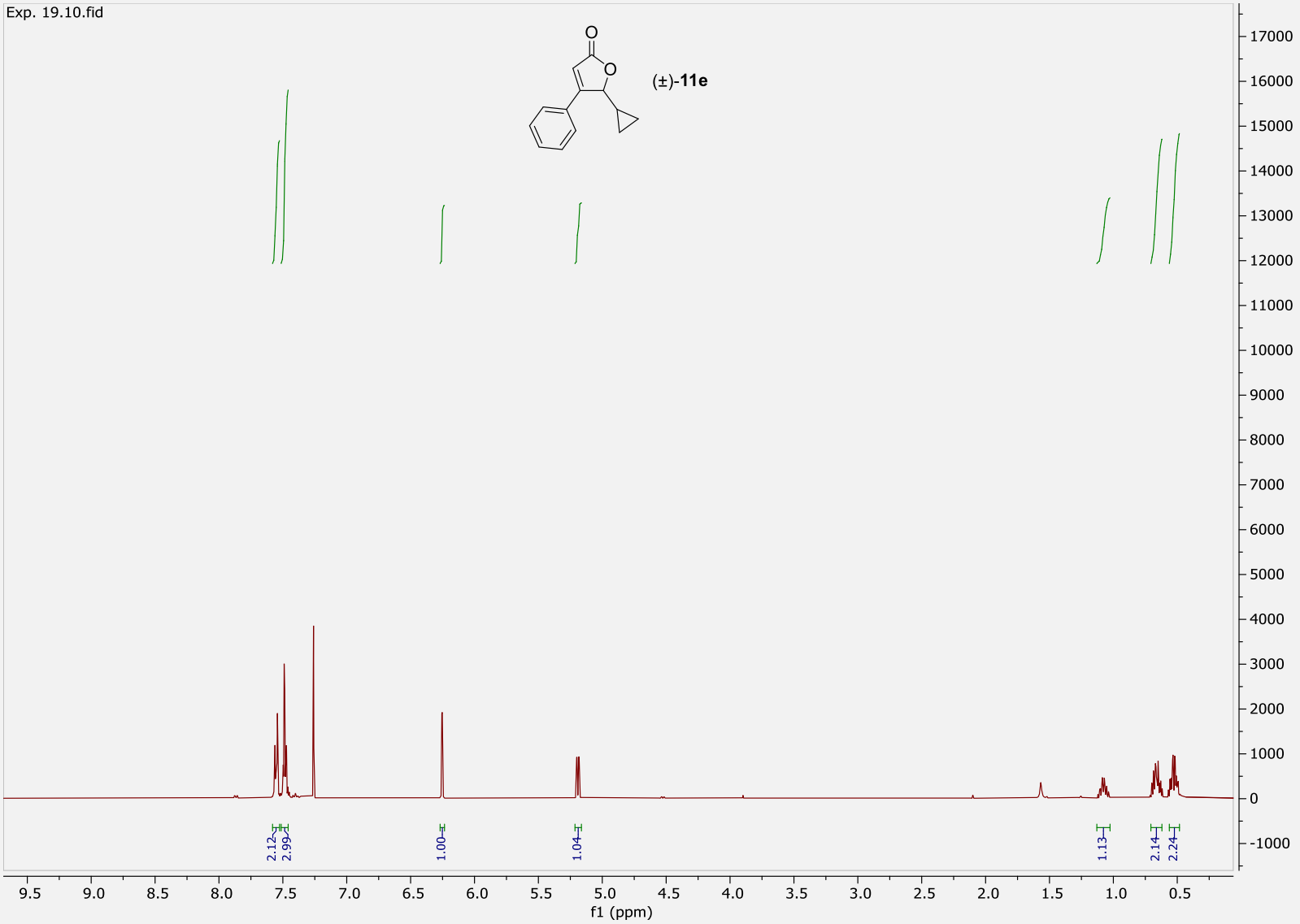
f1 (ppm)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(±)-11e
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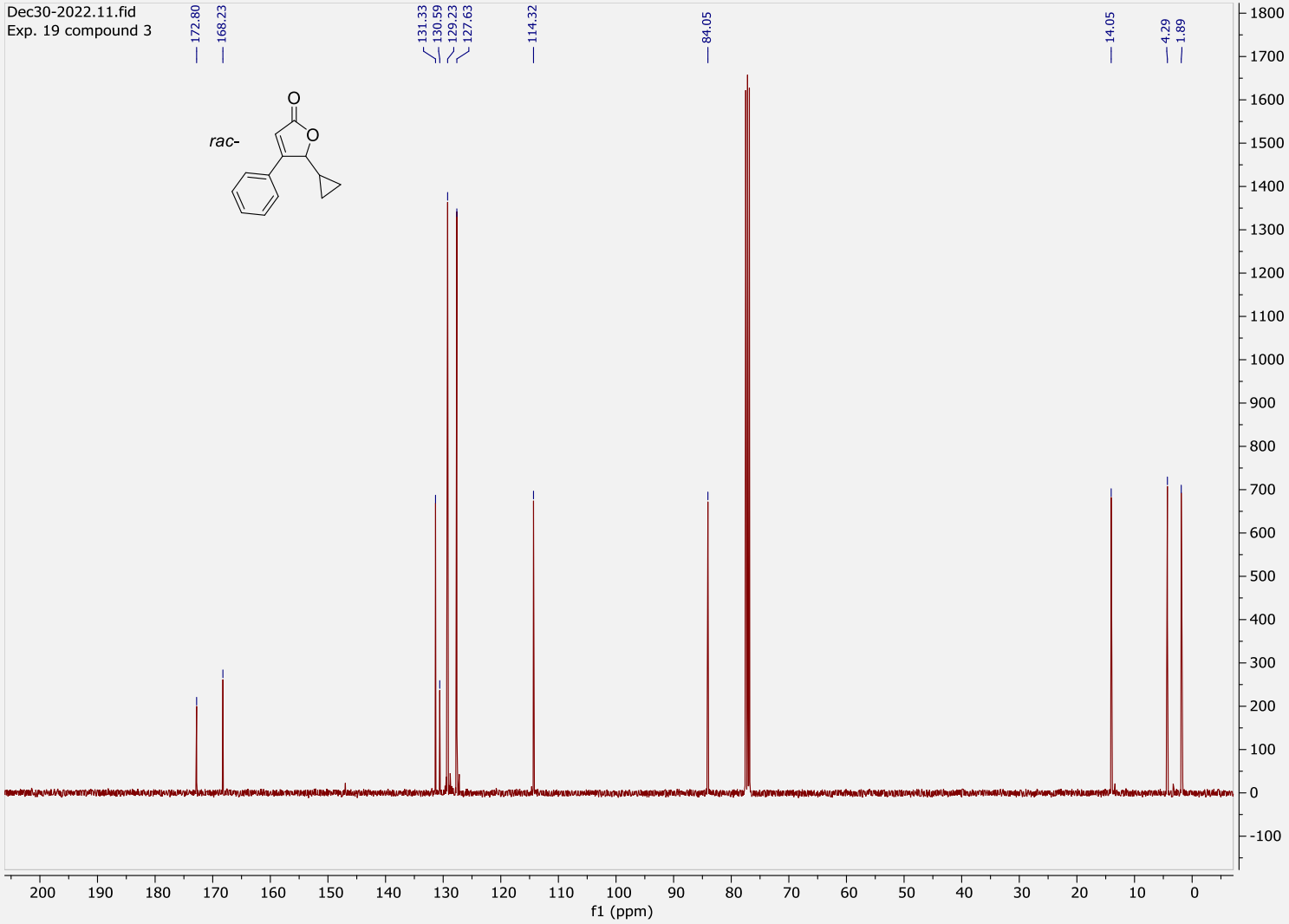
1.00
1.04
1.13
2.14
2.24
f1 (ppm)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127.63
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14.05
4.29
1.89
rac-
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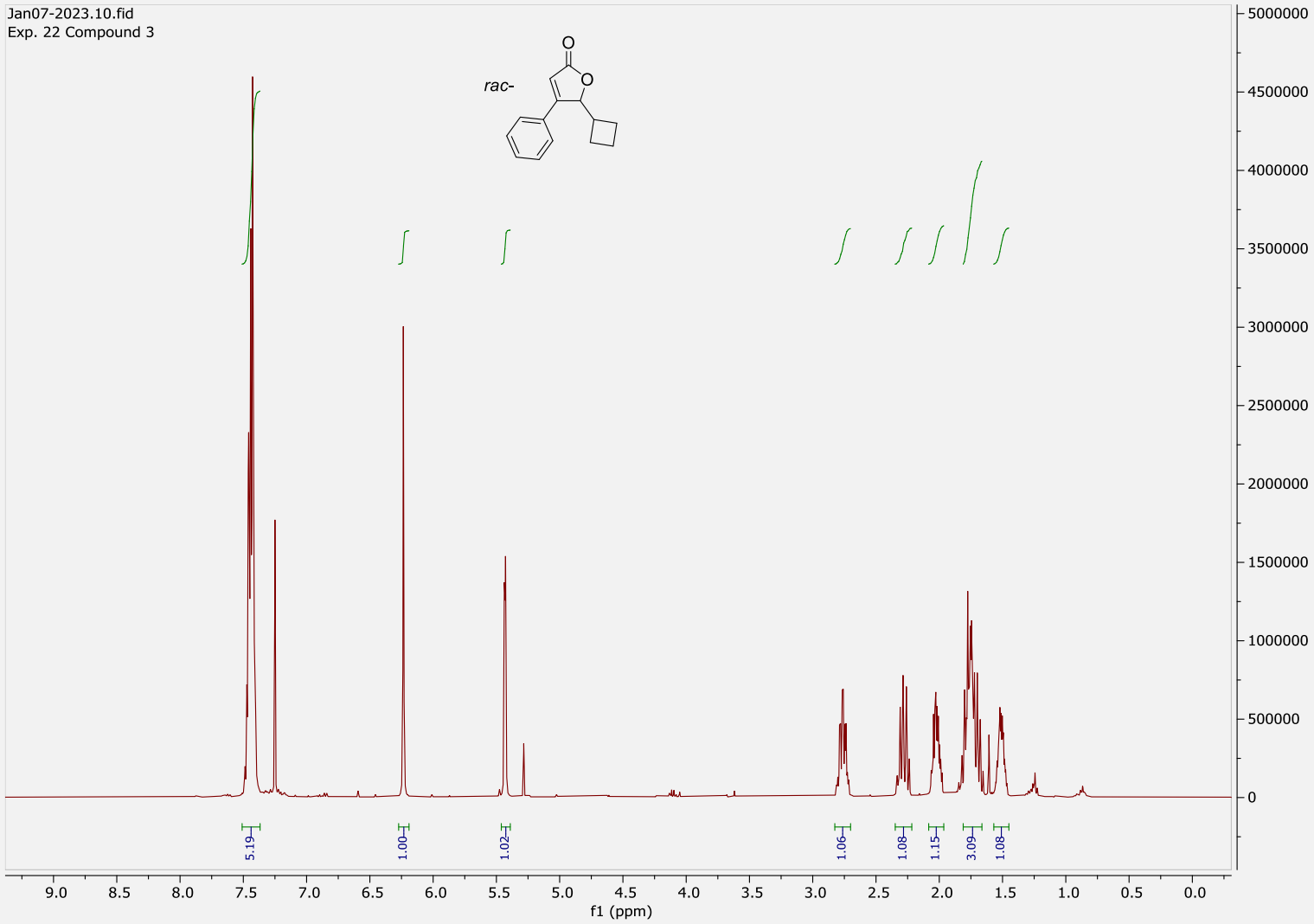
O
f1 (ppm)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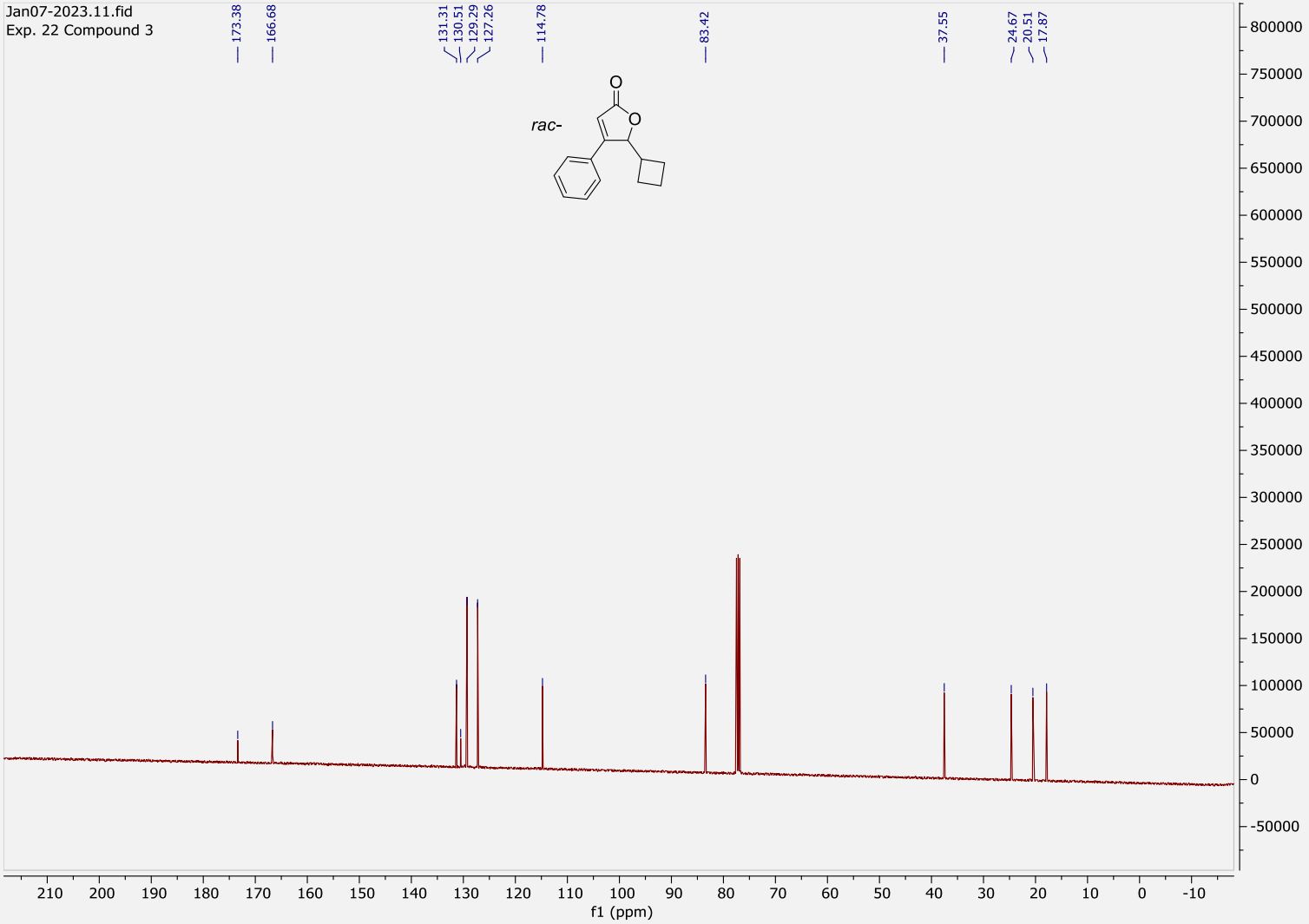
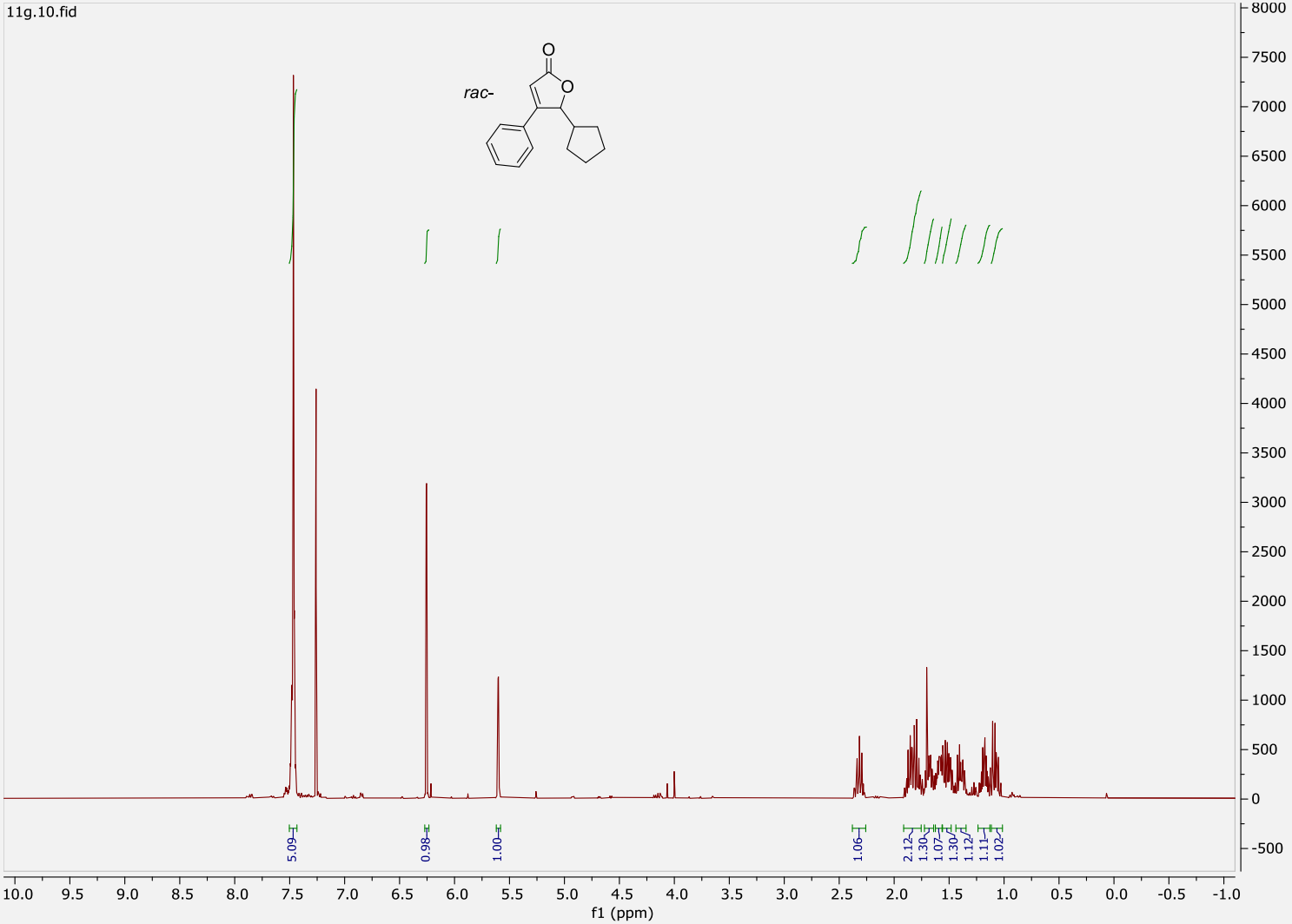
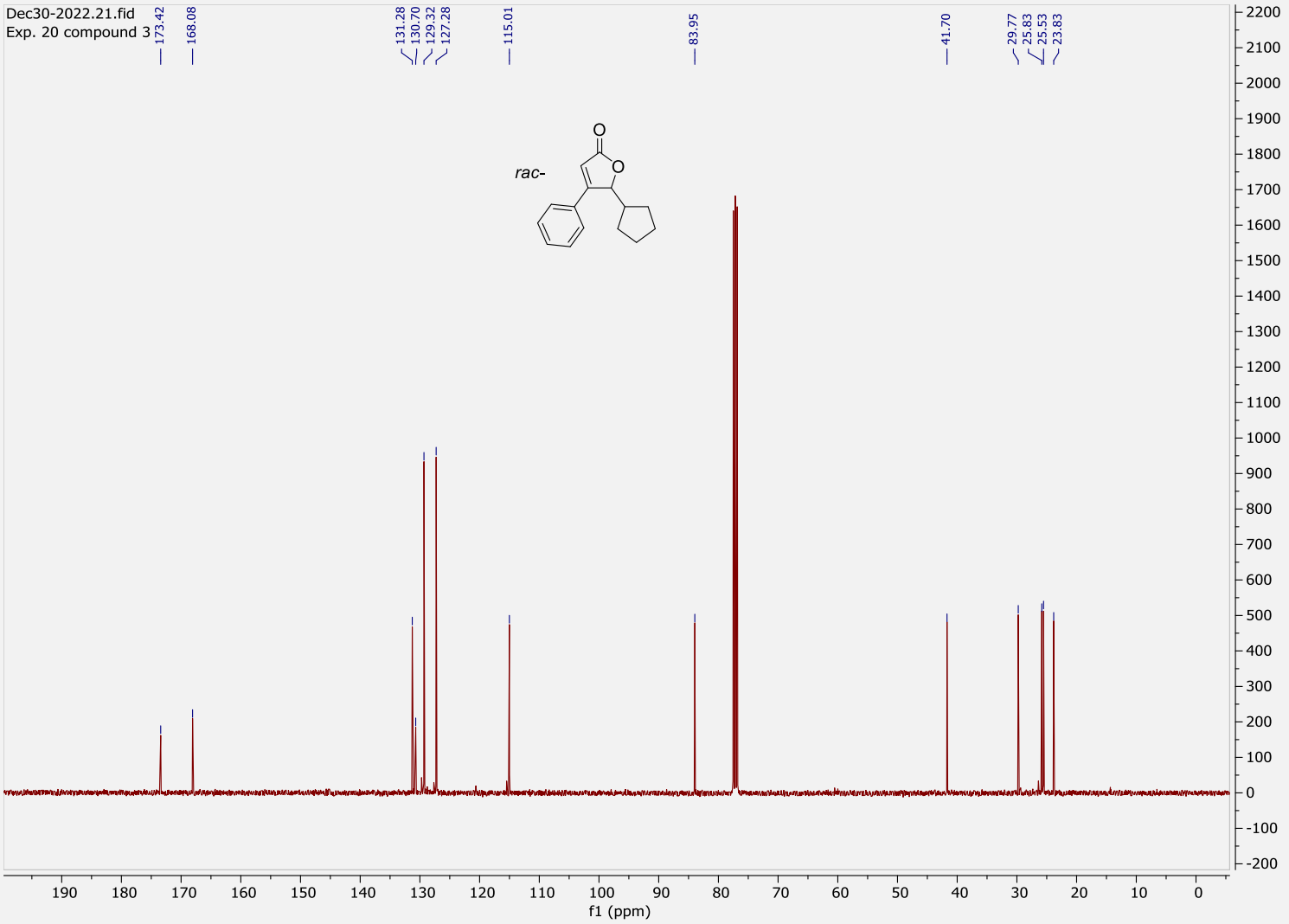
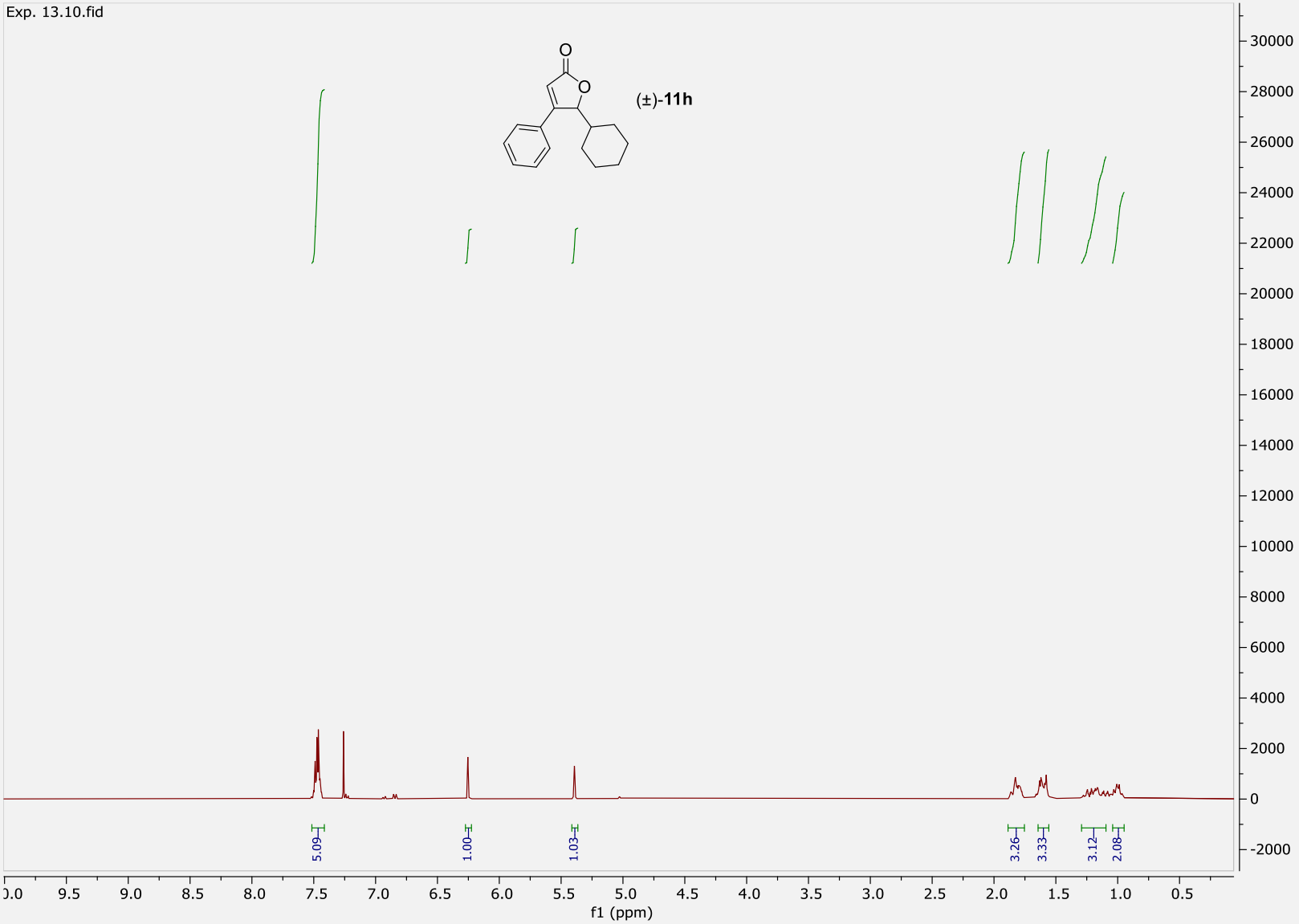
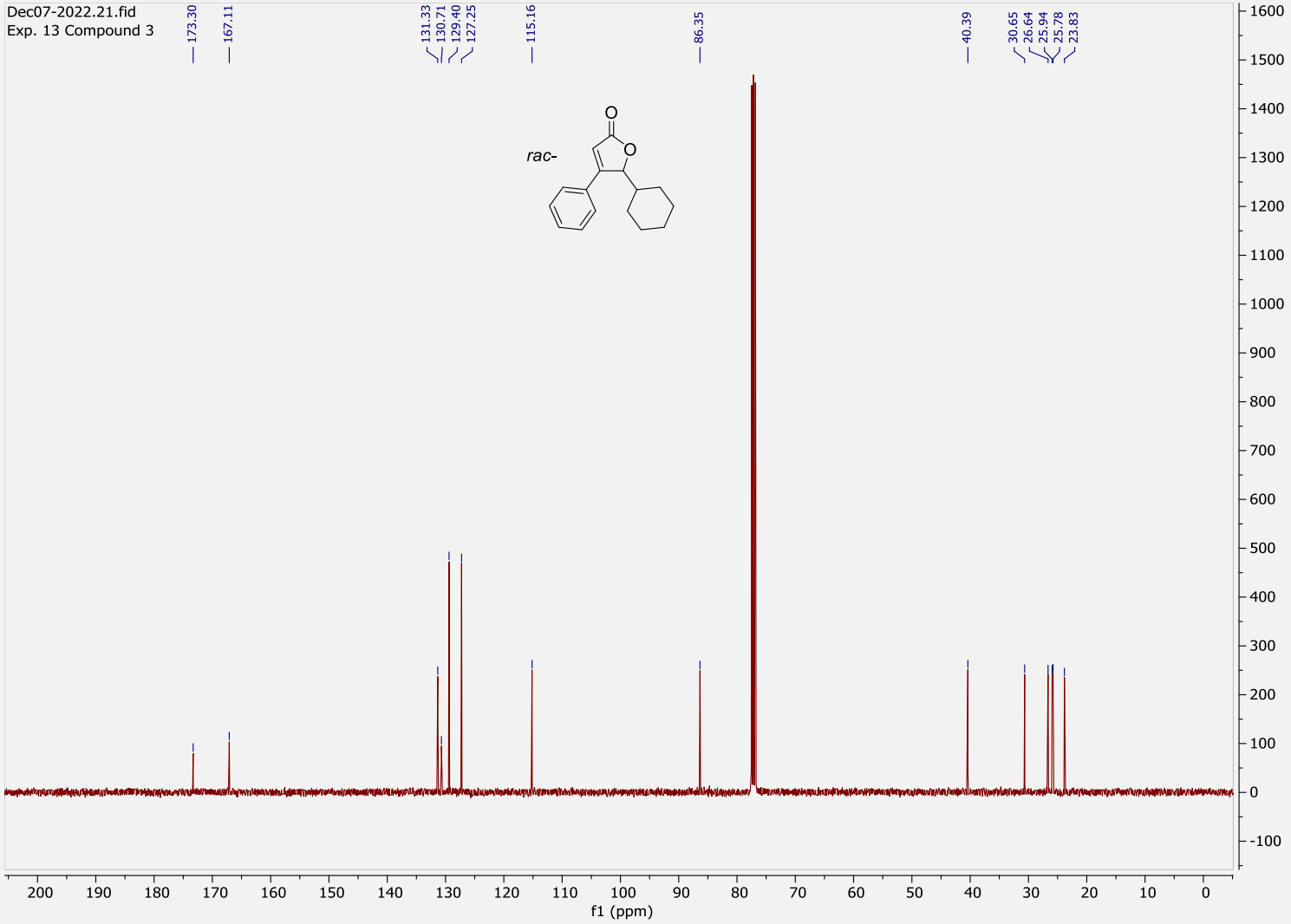
rac-
O
O
5.19
1.00
1.02
1.06
1.08
1.15
3.09
1.08
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
5000000
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0

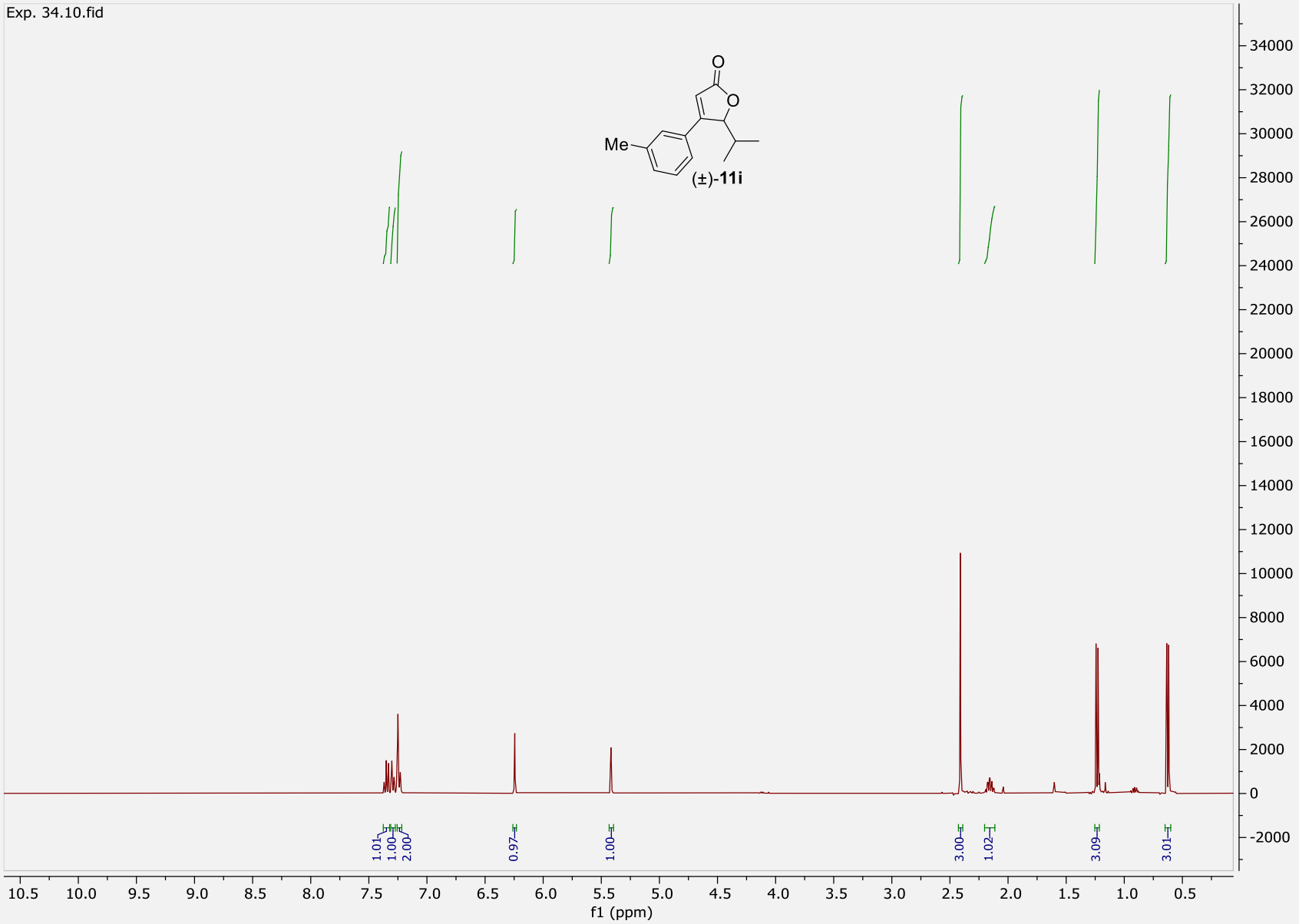

Jan07-2023.11.fid
Exp. 22 Compound 3
173.38
166.68
131.31
130.51
129.29
127.26
114.78
83.42
37.55
24.67
20.51
17.87
rac-
O
O
800000
750000
700000
650000
600000
550000
500000
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
-50000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

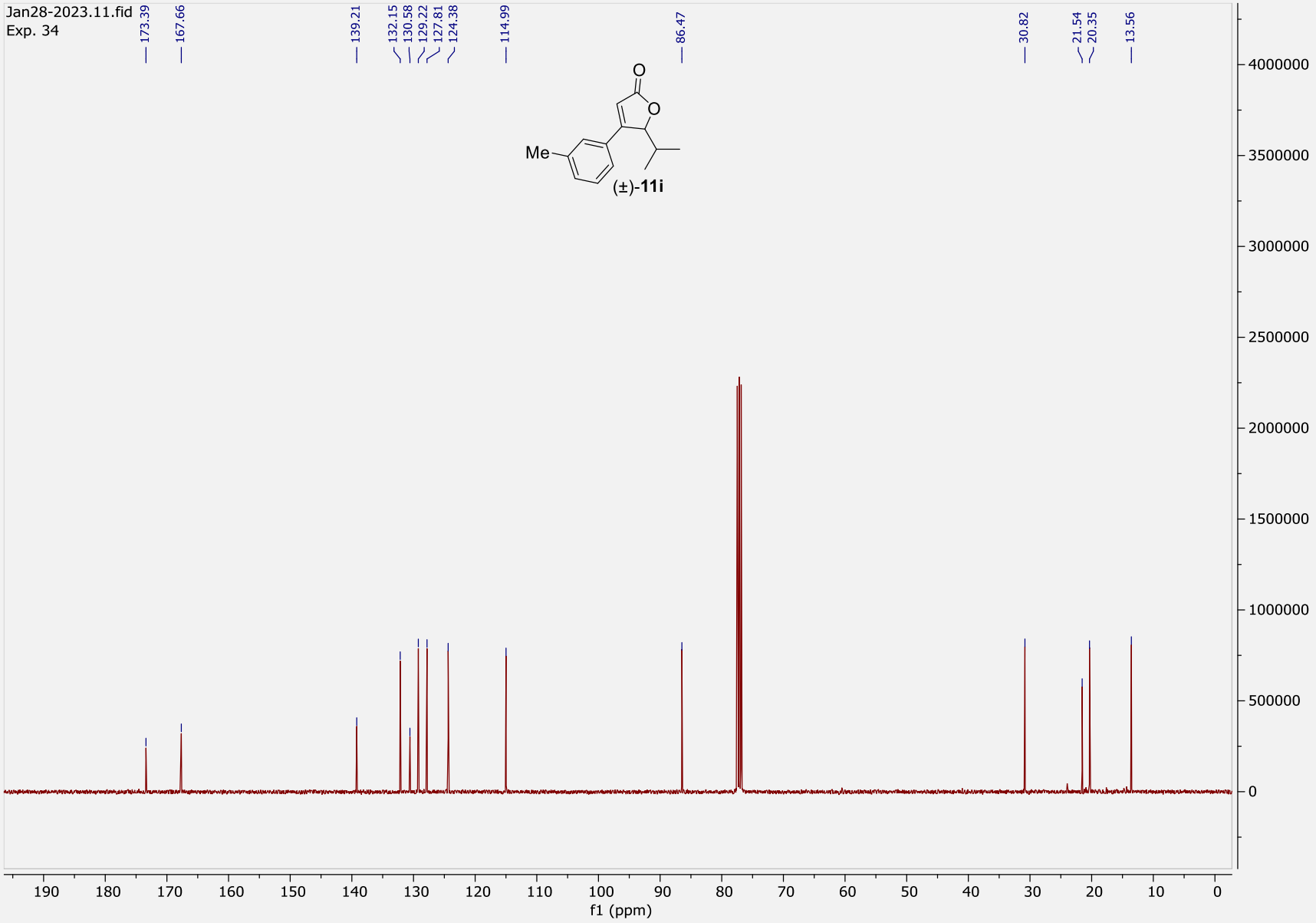

11g.10.fid
rac-
O
O
8000
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
5.09
0.98
1.00
1.06
2.12
1.30
1.07
1.30
1.12
1.11
1.02
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

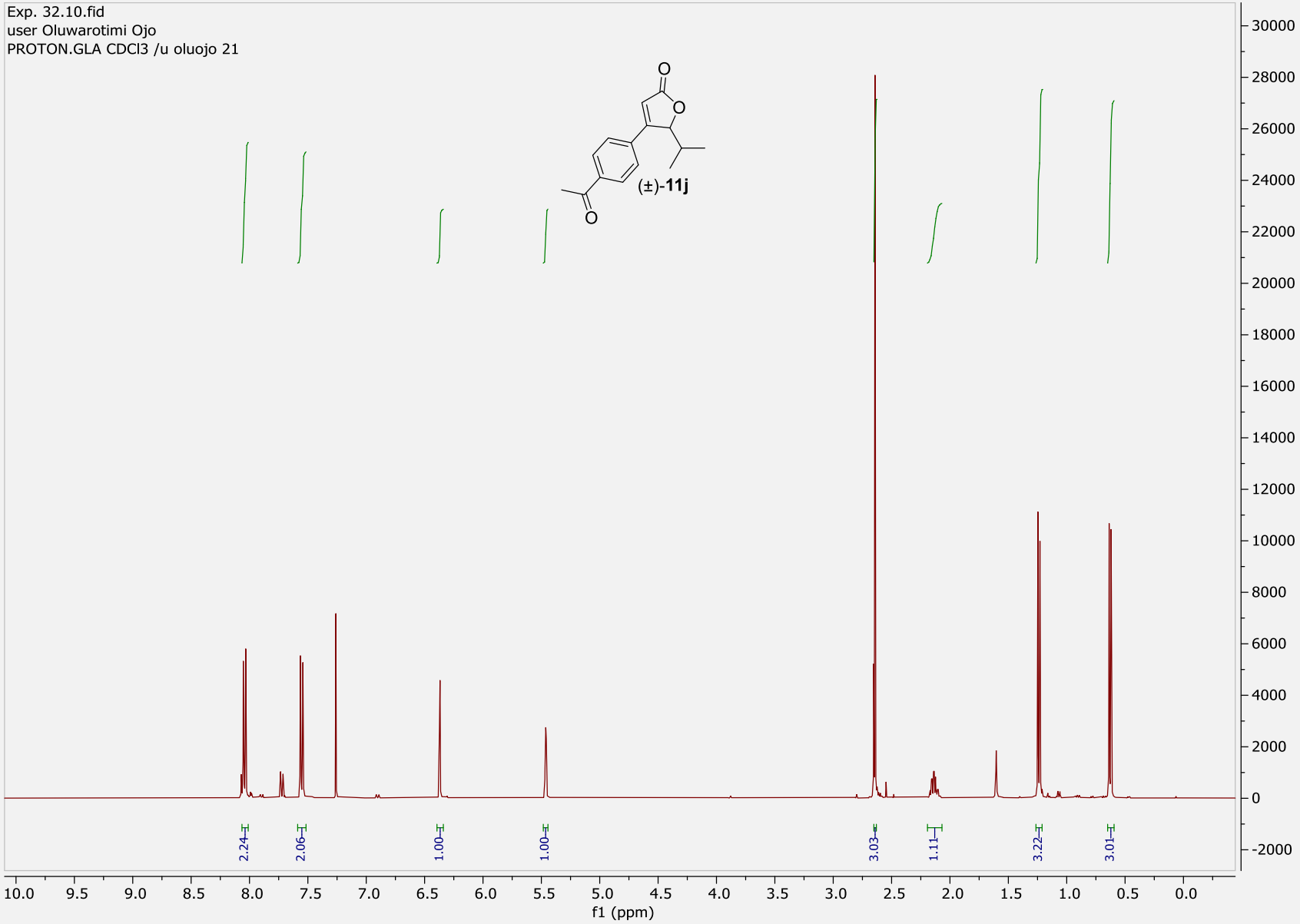

Dec30-2022.21.fid
Exp. 20 compound 3
173.42
168.08
131.28
130.70
129.32
127.28
115.01
83.95
41.70
29.77
25.83
25.53
23.83
rac-
O
O
f1 (ppm)

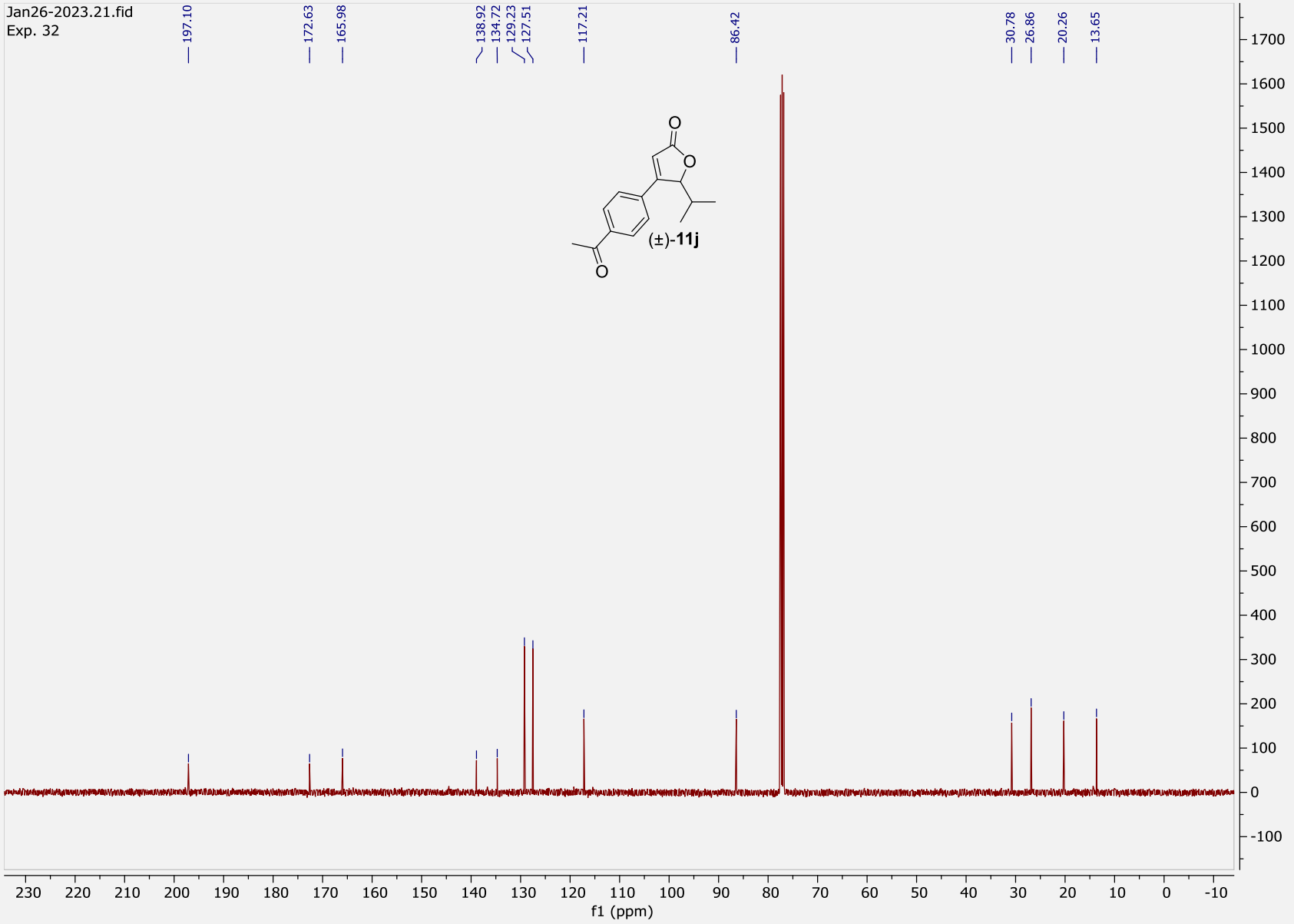

Exp. 13.10.fid
(±)-11h
5.09
1.00
1.03
3.26
3.33
3.12
2.08
f1 (ppm)

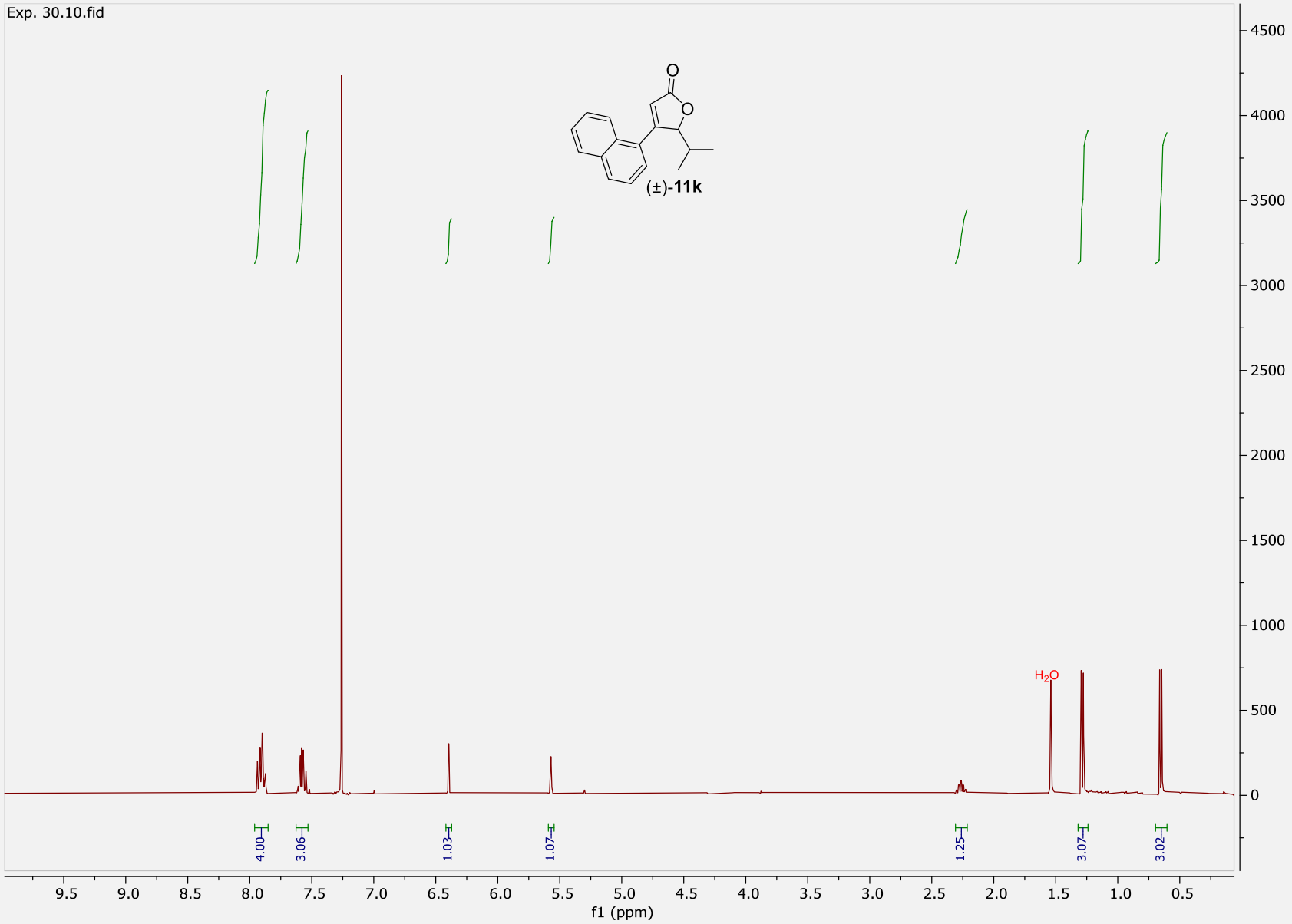

Dec07-2022.21.fid
Exp. 13 Compound 3
173.30
167.11
131.33
130.71
129.40
127.25
115.16
86.35
40.39
30.65
26.64
25.94
25.78
23.83
rac-
O
O
f1 (ppm)

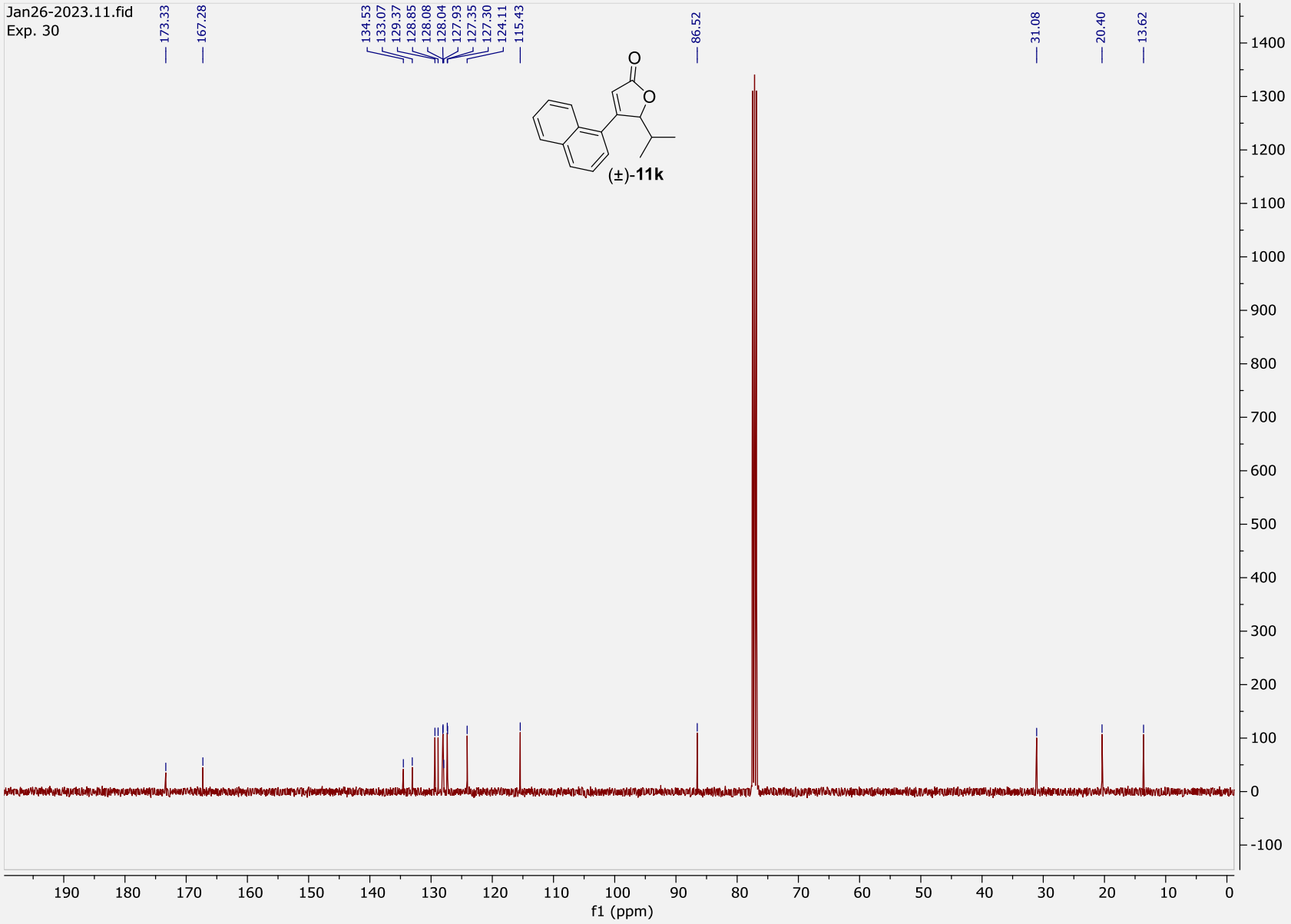

Exp. 34.10.fid
O
O
Me
(±)-11i
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
1.01
1.00
2.00
0.97
1.00
3.00
1.02
3.09
3.01
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

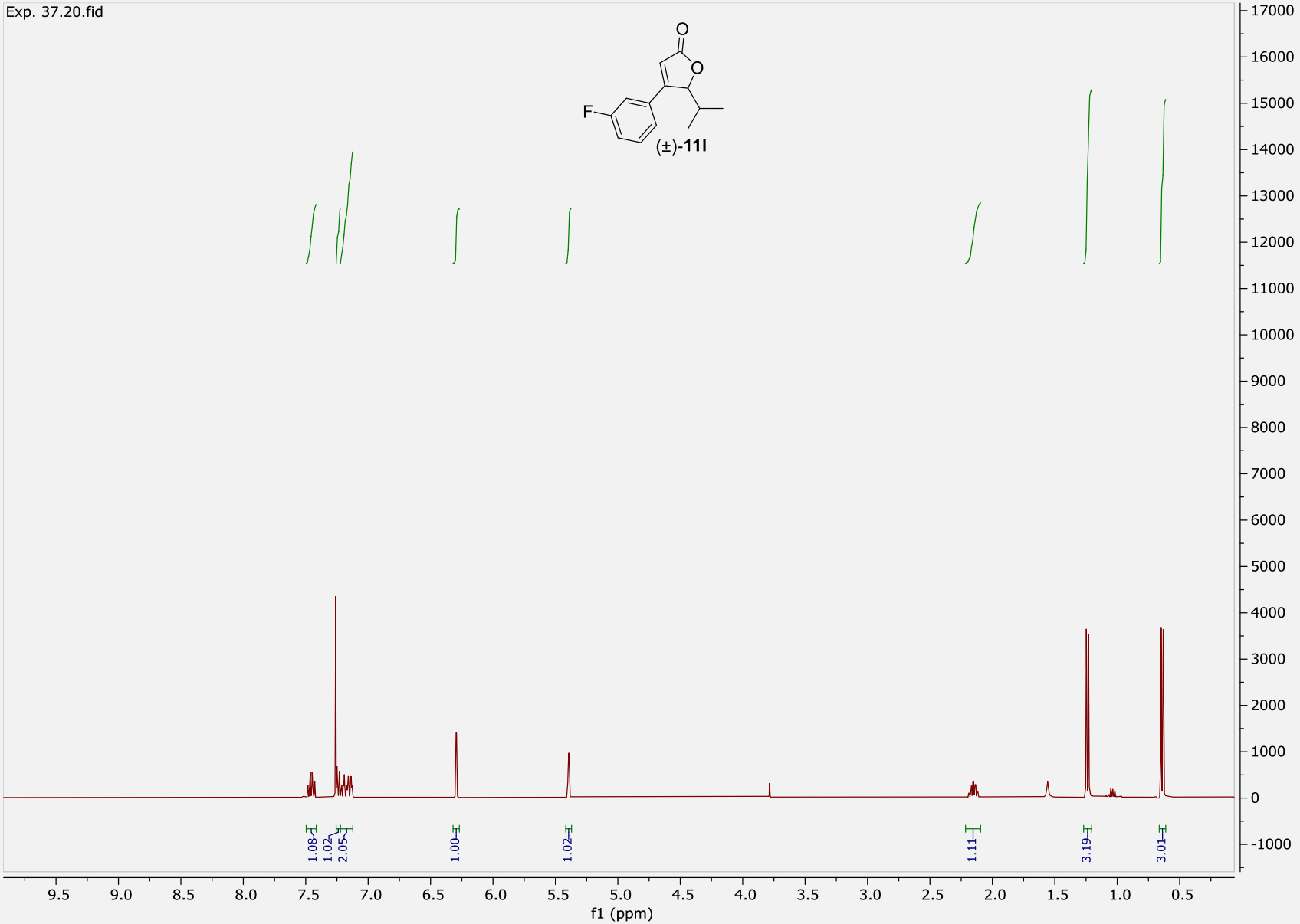

Jan28-2023.11.fid
Exp. 34
173.39
167.66
139.21
132.15
130.58
129.22
127.81
124.38
114.99
86.47
30.82
21.54
20.35
13.56
Me
(±)-11i
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

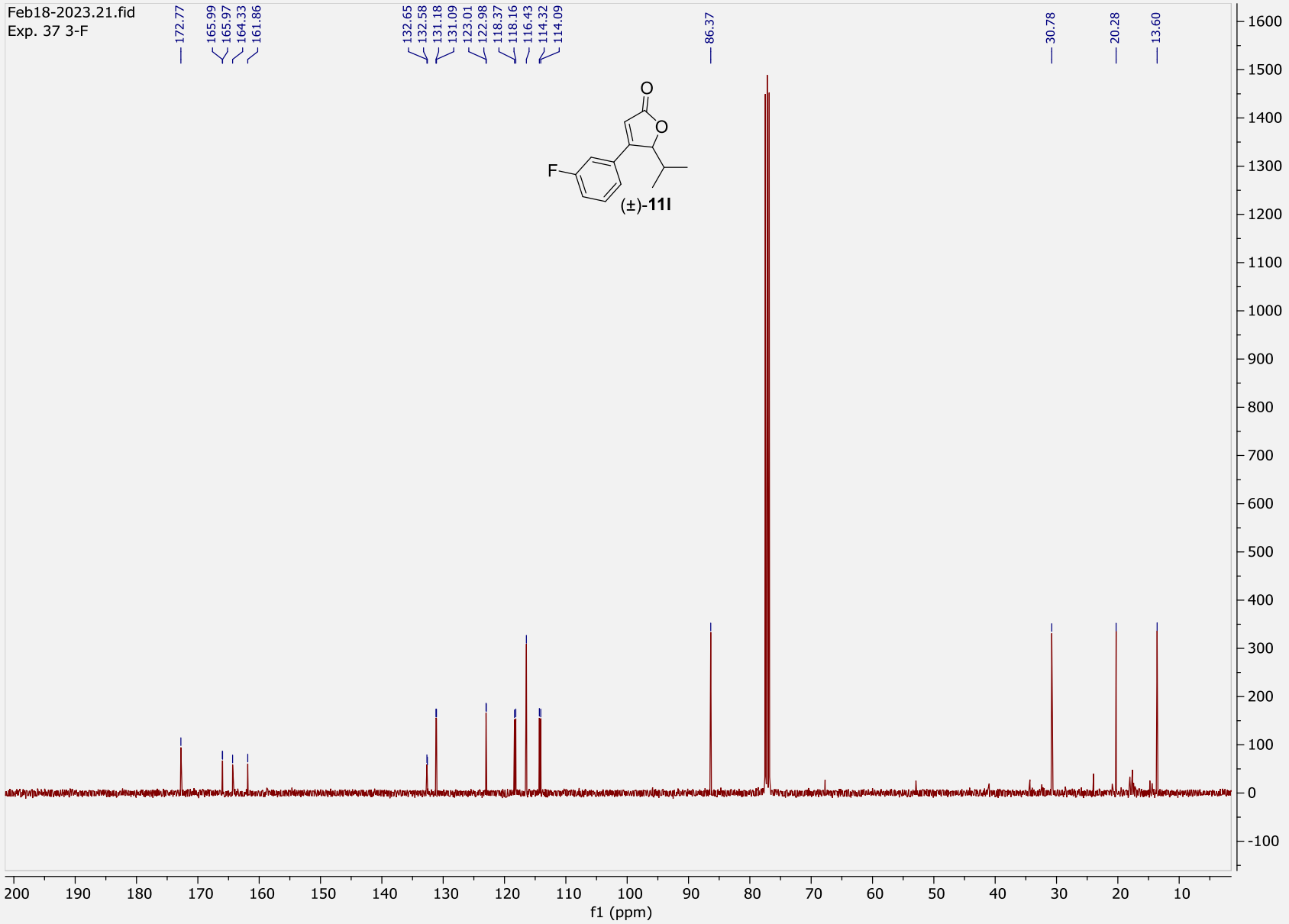

Exp. 32.10.fid
user Oluwarotimi Ojo
PROTON.GLA CDCl3 /u oluojo 21
(±)-11j
2.24
2.06
1.00
1.00
3.03
1.11
3.22
3.01
f1 (ppm)

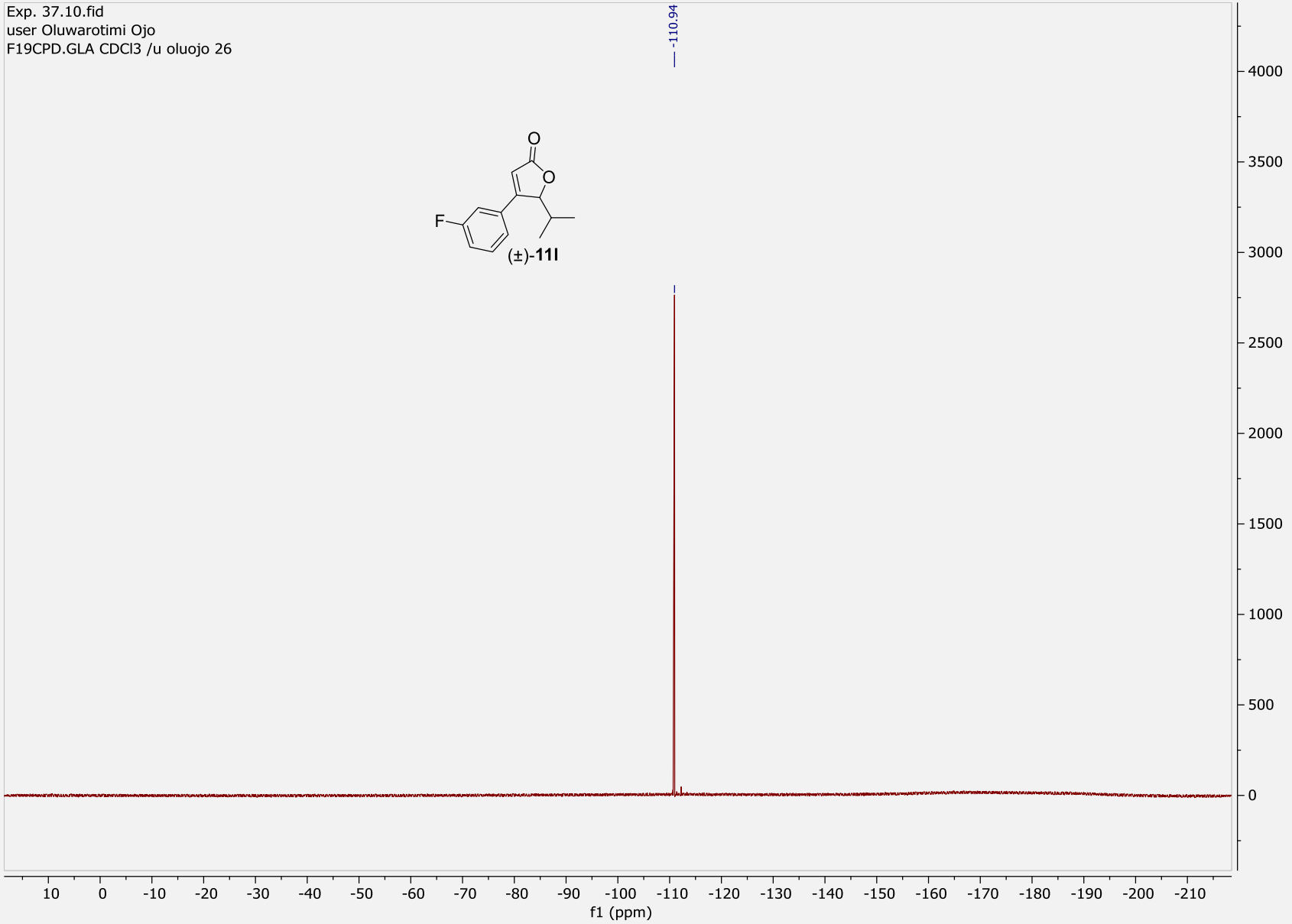

Jan26-2023.21.fid
Exp. 32
197.10
172.63
165.98
138.92
134.72
129.23
127.51
117.21
86.42
30.78
26.86
20.26
13.65
(±)-11j
f1 (ppm)

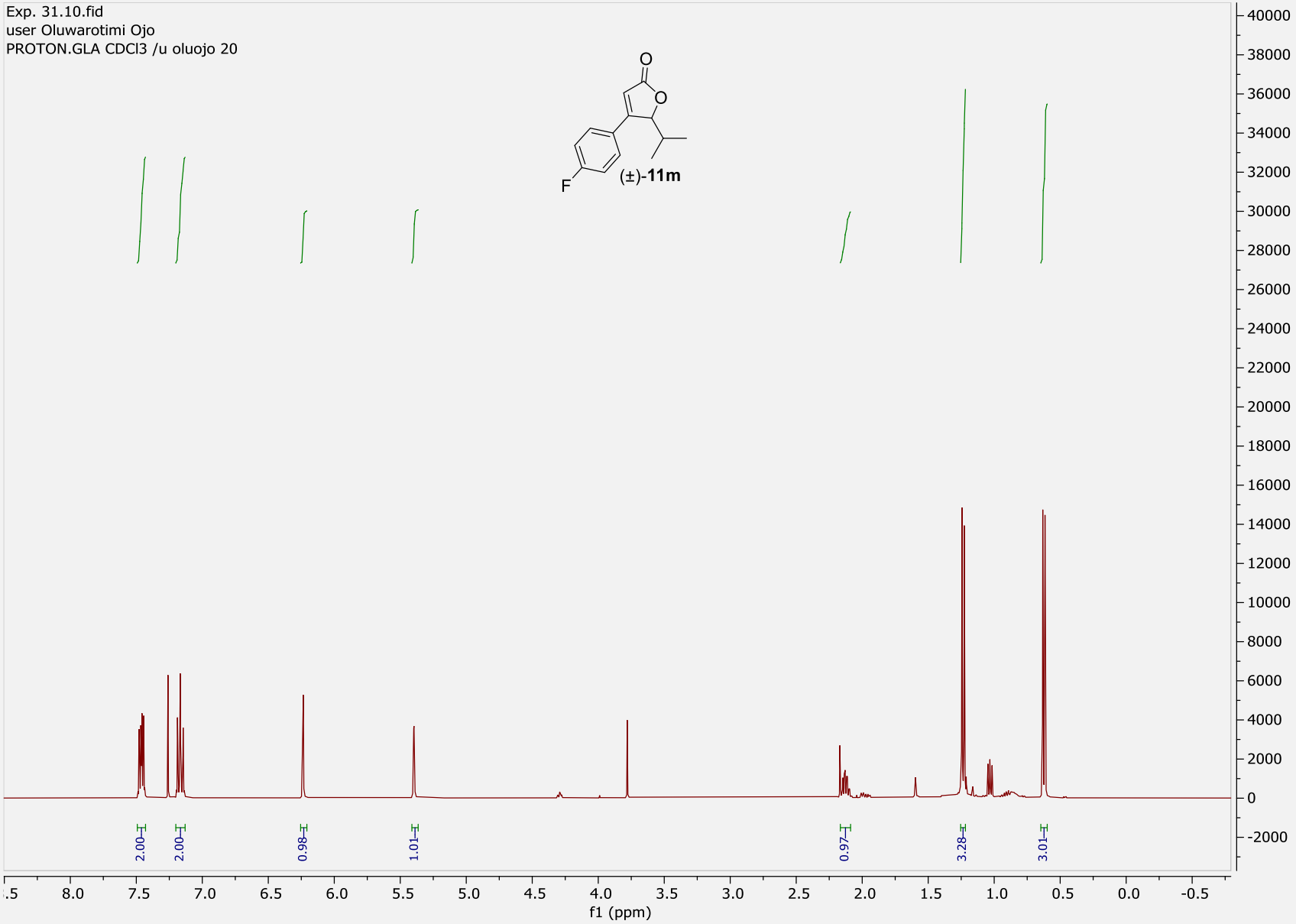

Exp. 30.10.fid
(±)-11k
H2O
4.00
3.06
1.03
1.07
1.25
3.07
3.02
f1 (ppm)

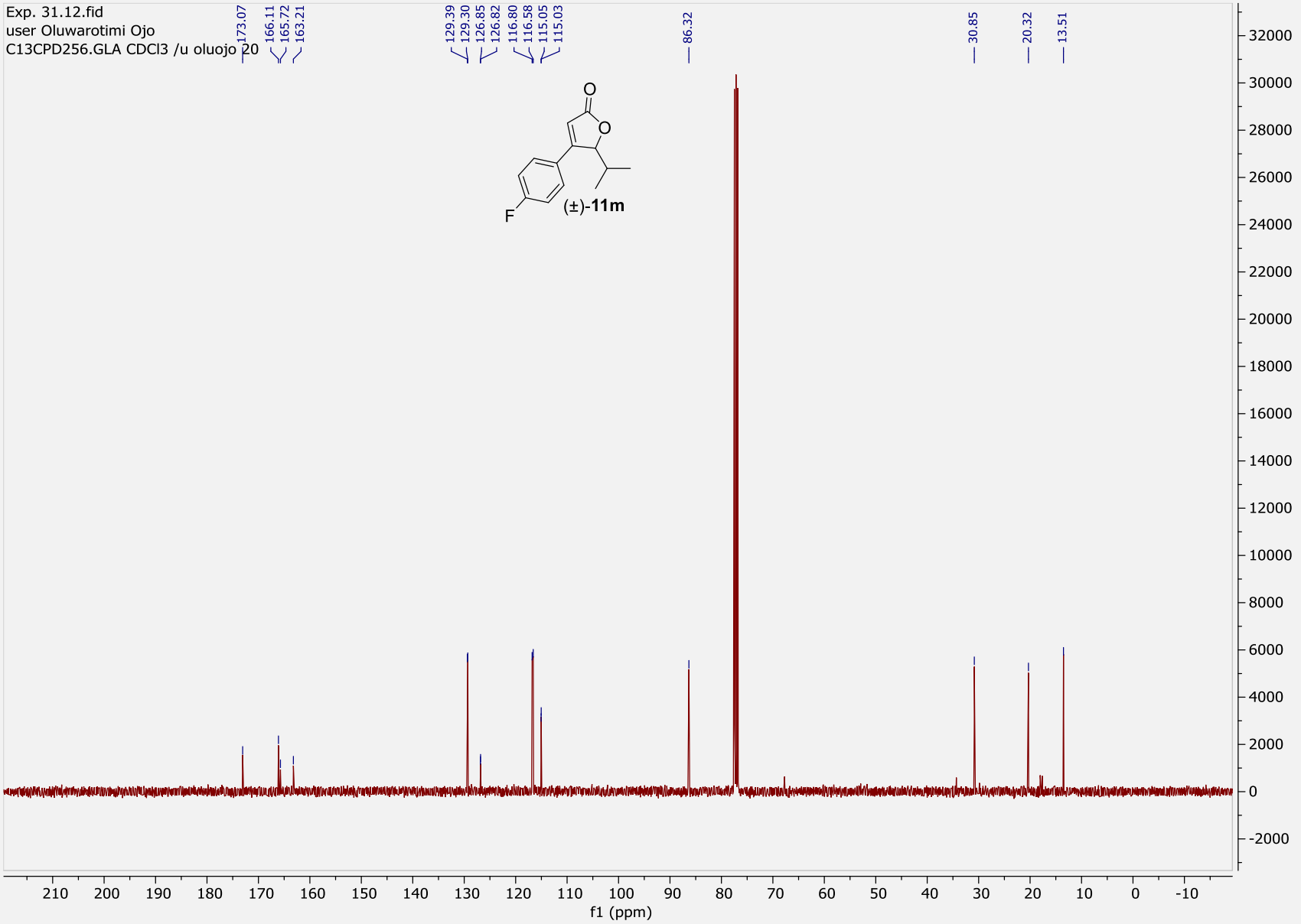

Jan26-2023.11.fid
Exp. 30
173.33
167.28
134.53
133.07
129.37
128.85
128.08
128.04
127.93
127.35
127.30
124.11
115.43
86.52
31.08
20.40
13.62
(±)-11k
f1 (ppm)

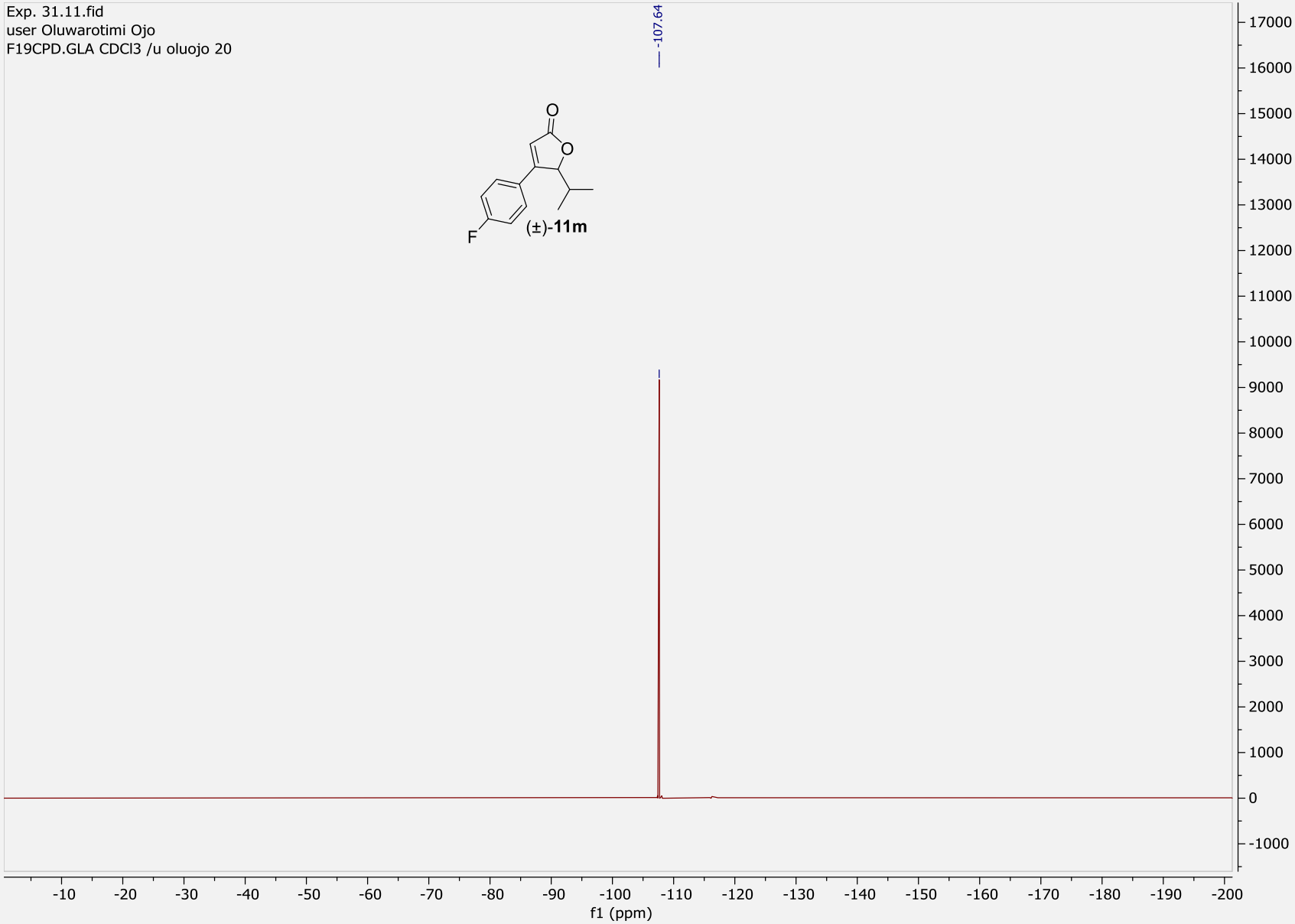

Exp. 37.20.fid
O
O
F
(±)-11l
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
1.08
1.02
2.05
1.00
1.02
1.11
3.19
3.01
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

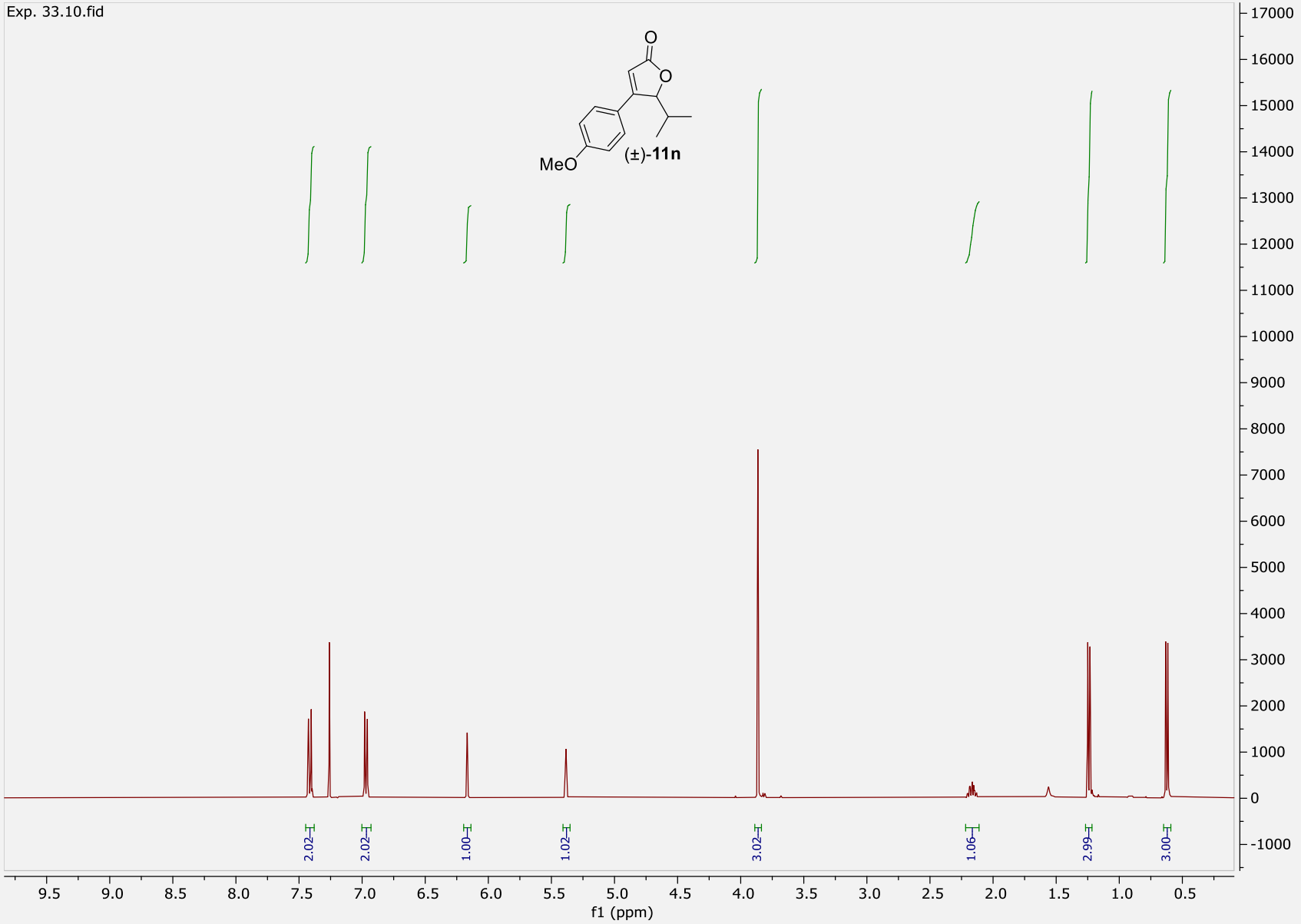

Feb18-2023.21.fid
Exp. 37 3-F
172.77
165.99
165.97
164.33
161.86
132.65
132.58
131.18
131.09
123.01
122.98
118.37
118.16
116.43
114.32
114.09
86.37
30.78
20.28
13.60
O
O
F
(±)-11l
f1 (ppm)

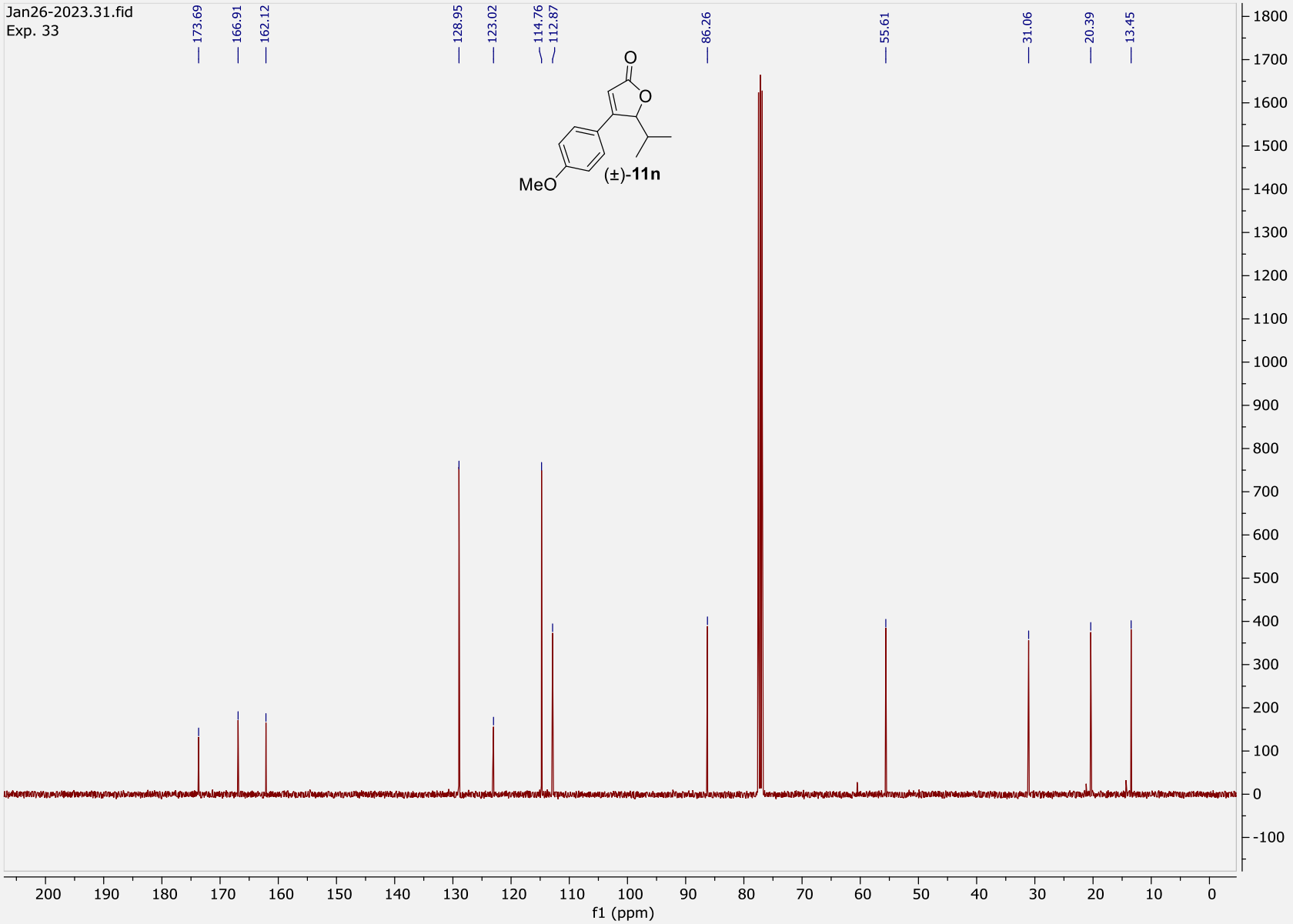

Exp. 37.10.fid
user Oluwarotimi Ojo
F19CPD.GLA CDCl3 /u oluojo 26
-110.94
O
O
F
(±)-11l
4000
3500
3000
2500
2000
1500
1000
500
0
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

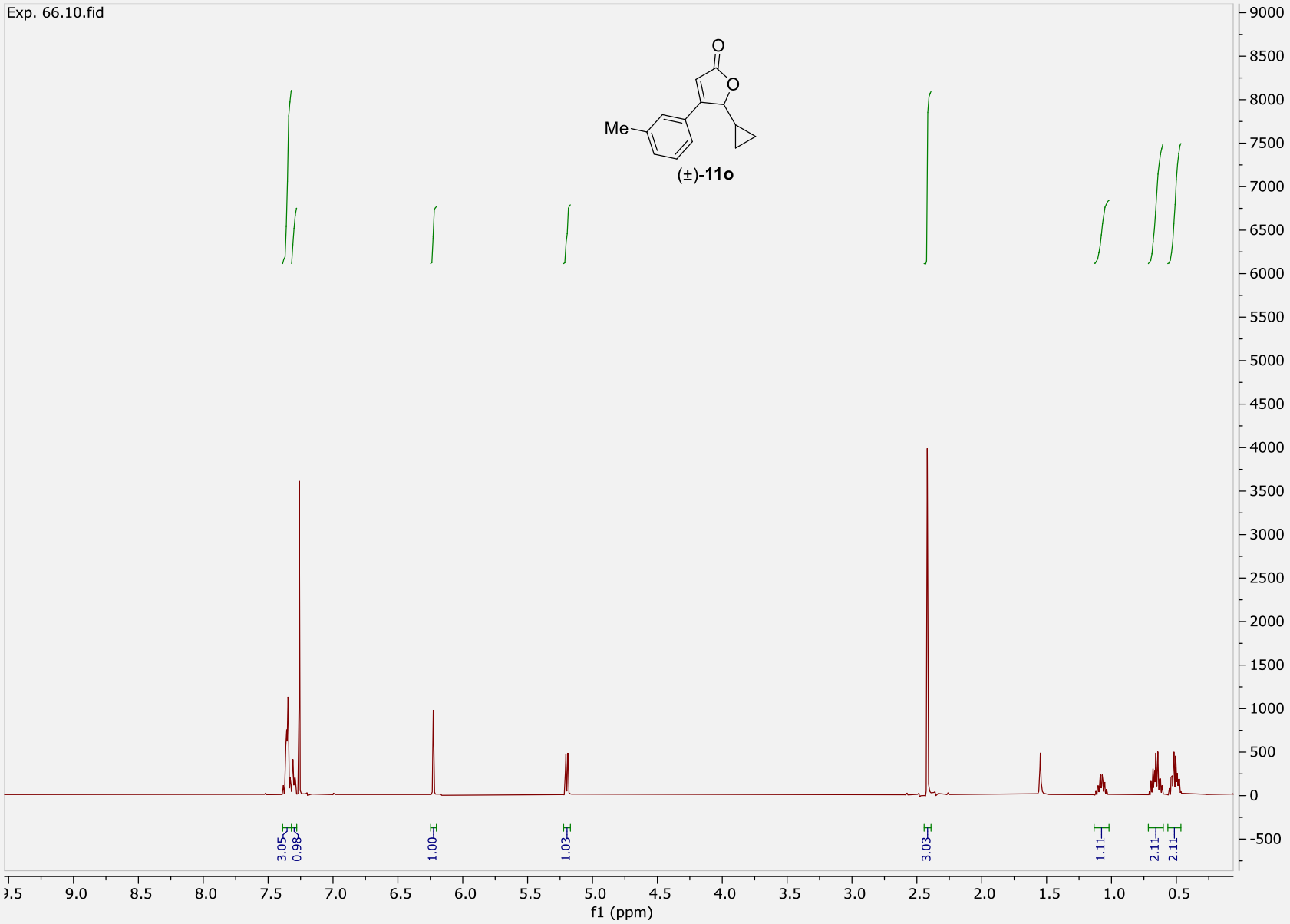

Exp. 31.10.fid
user Oluwarotimi Ojo
PROTON.GLA CDCl3 /u oluojo 20
O
O
F
(±)-11m
2.00
2.00
0.98
1.01
0.97
3.28
3.01
f1 (ppm)

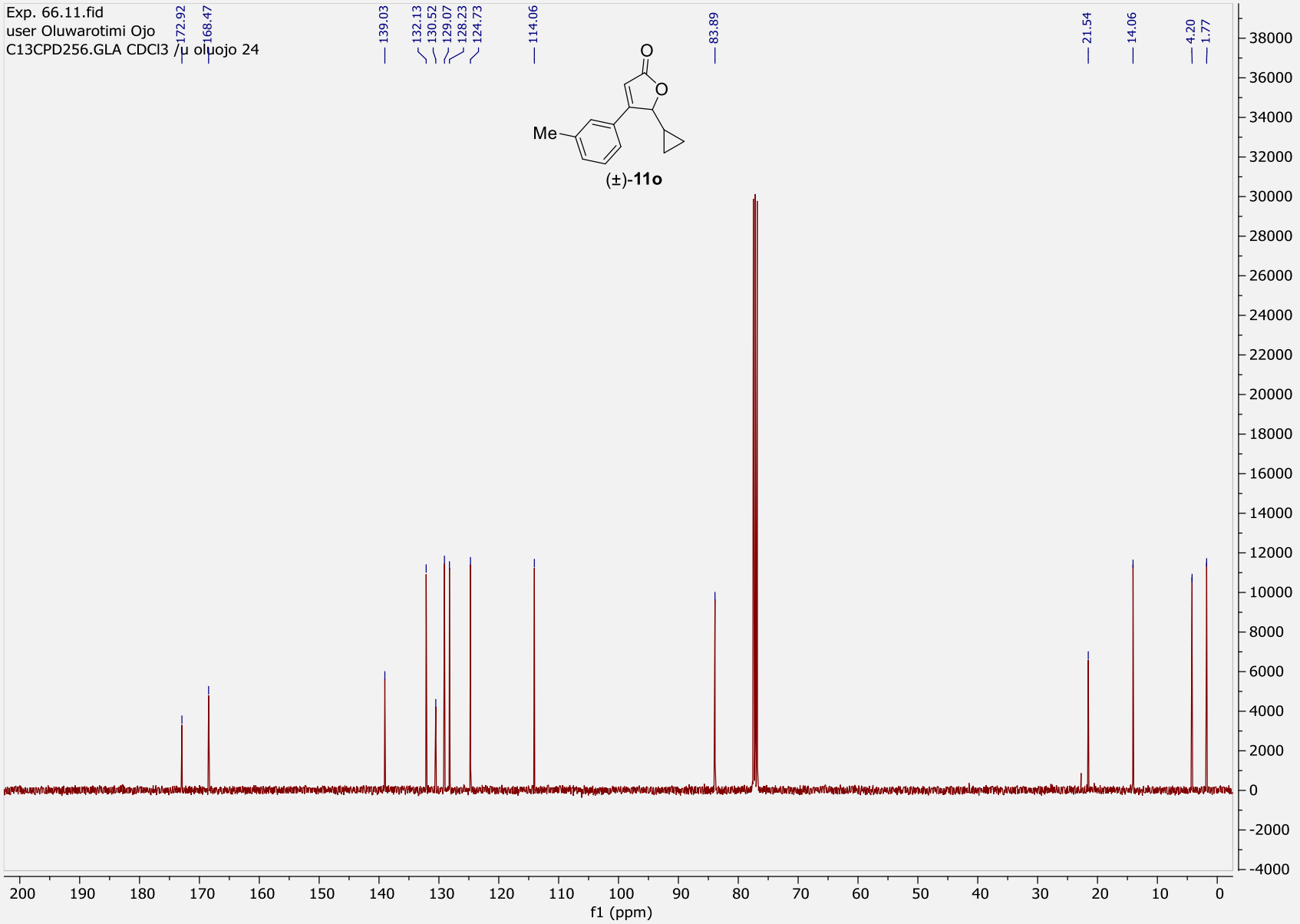

Exp. 31.12.fid
user Oluwarotimi Ojo
C13CPD256.GLA CDCl3 /u oluojo 20
173.07
166.11
165.72
163.21
129.39
129.30
126.85
126.82
116.80
116.58
115.05
115.03
86.32
30.85
20.32
13.51
O
O
F
(±)-11m
f1 (ppm)

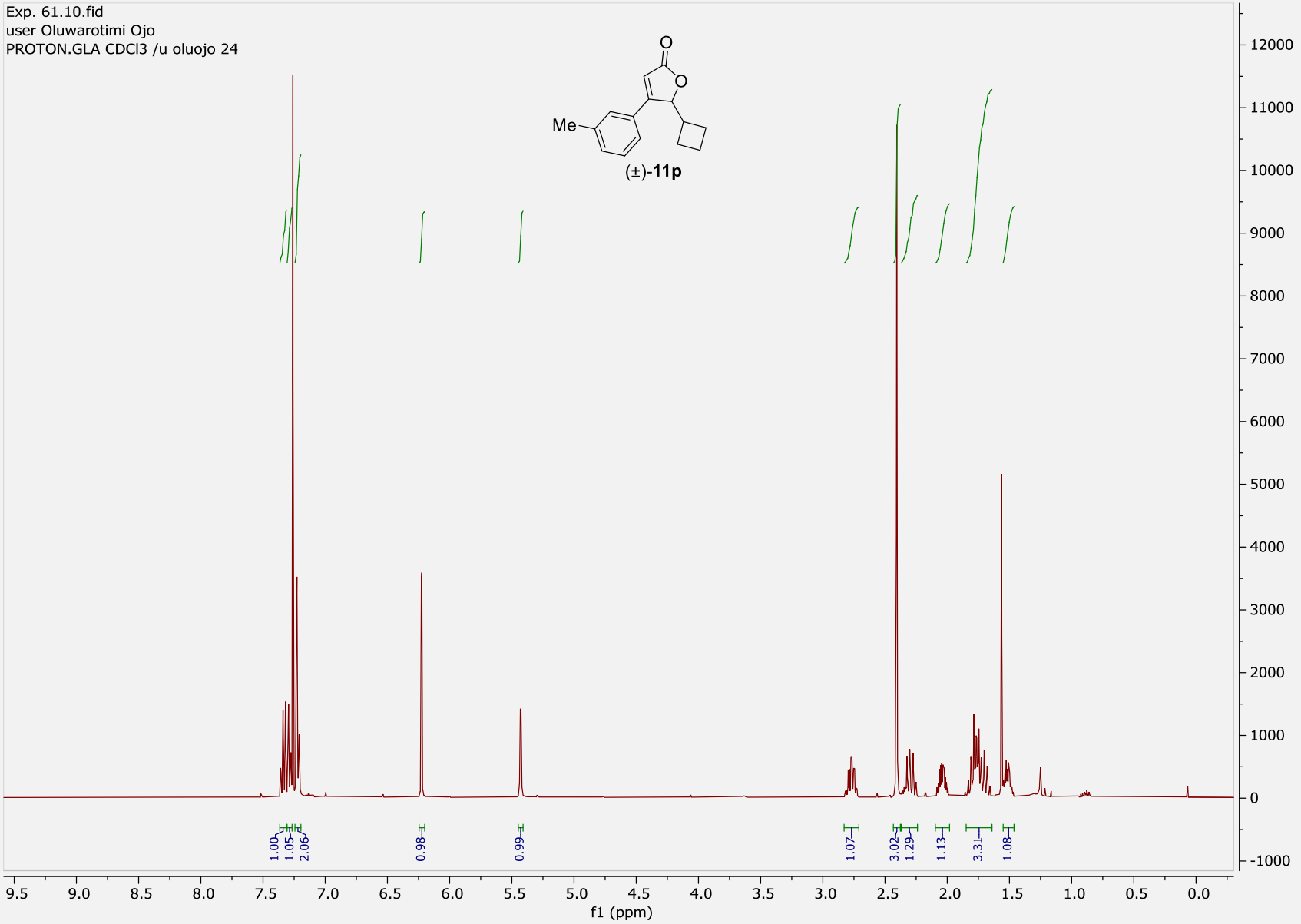

Exp. 31.11.fid
user Oluwarotimi Ojo
F19CPD.GLA CDCl3 /u oluojo 20
-107.64
O
O
F
(±)-11m
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

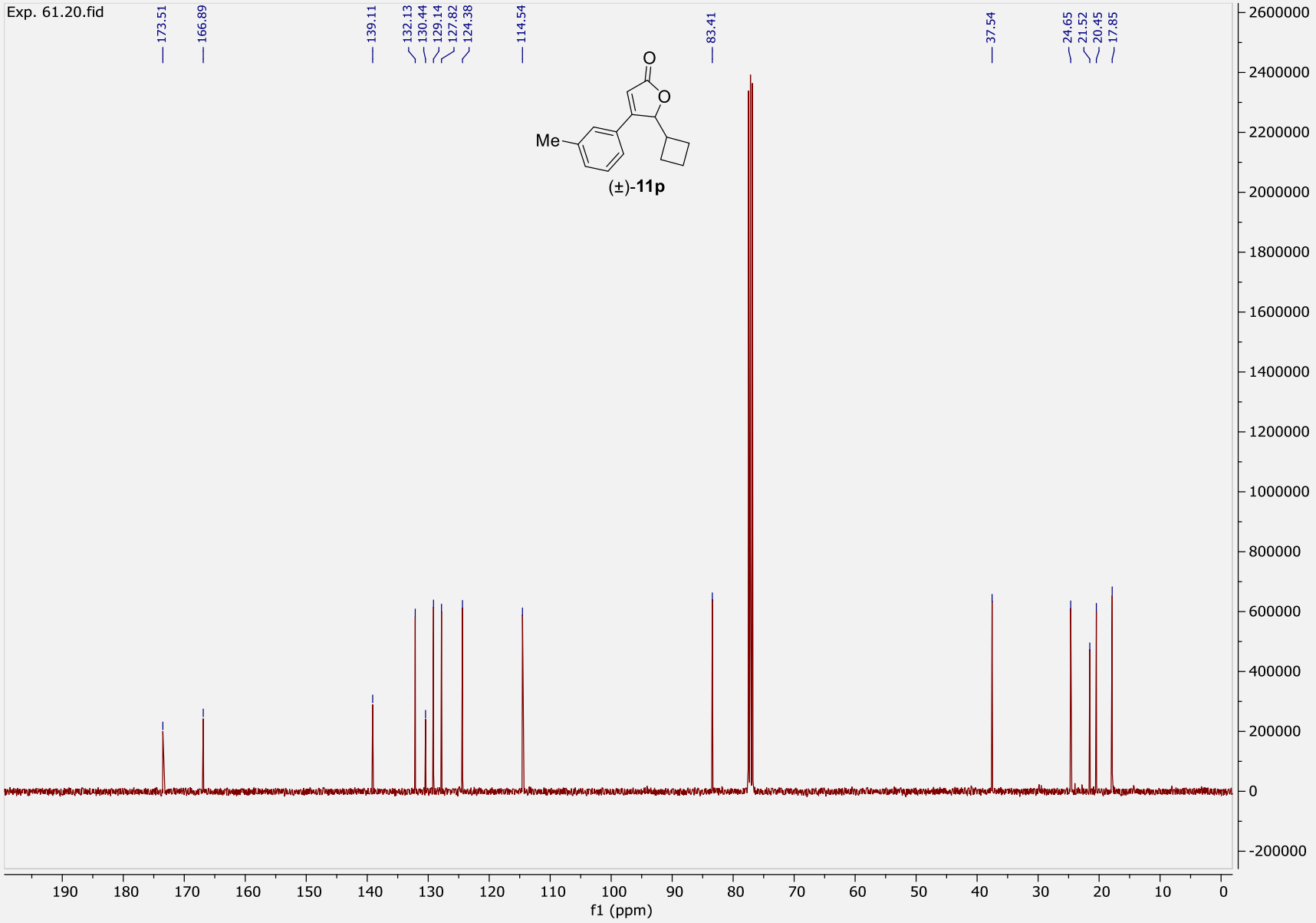

Exp. 33.10.fid
O
O
MeO
(±)-11n
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
2.02
2.02
1.00
1.02
3.02
1.06
2.99
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

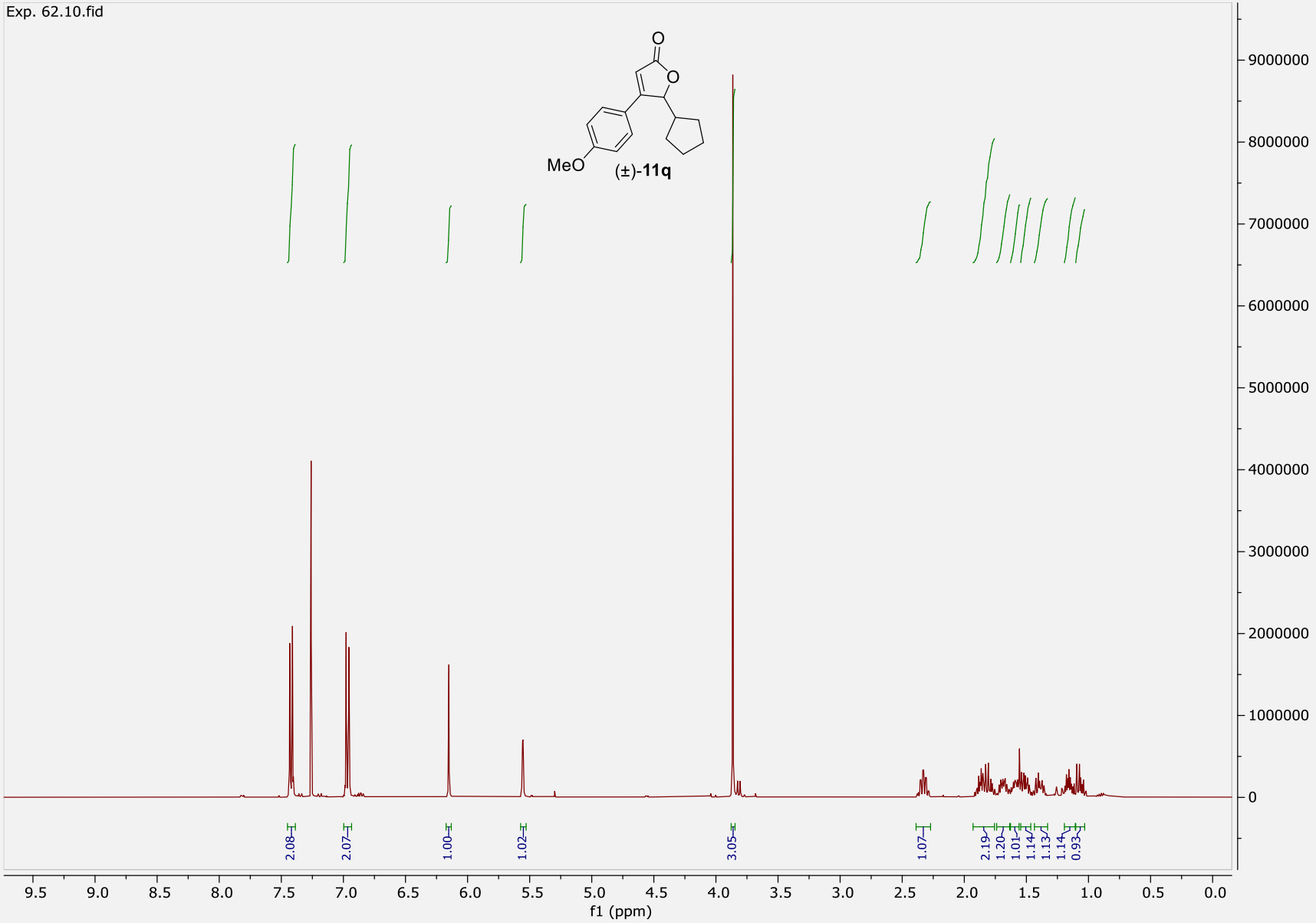

Jan26-2023.31.fid
Exp. 33
173.69
166.91
162.12
128.95
123.02
114.76
112.87
86.26
55.61
31.06
20.39
13.45
MeO
(±)-11n
f1 (ppm)

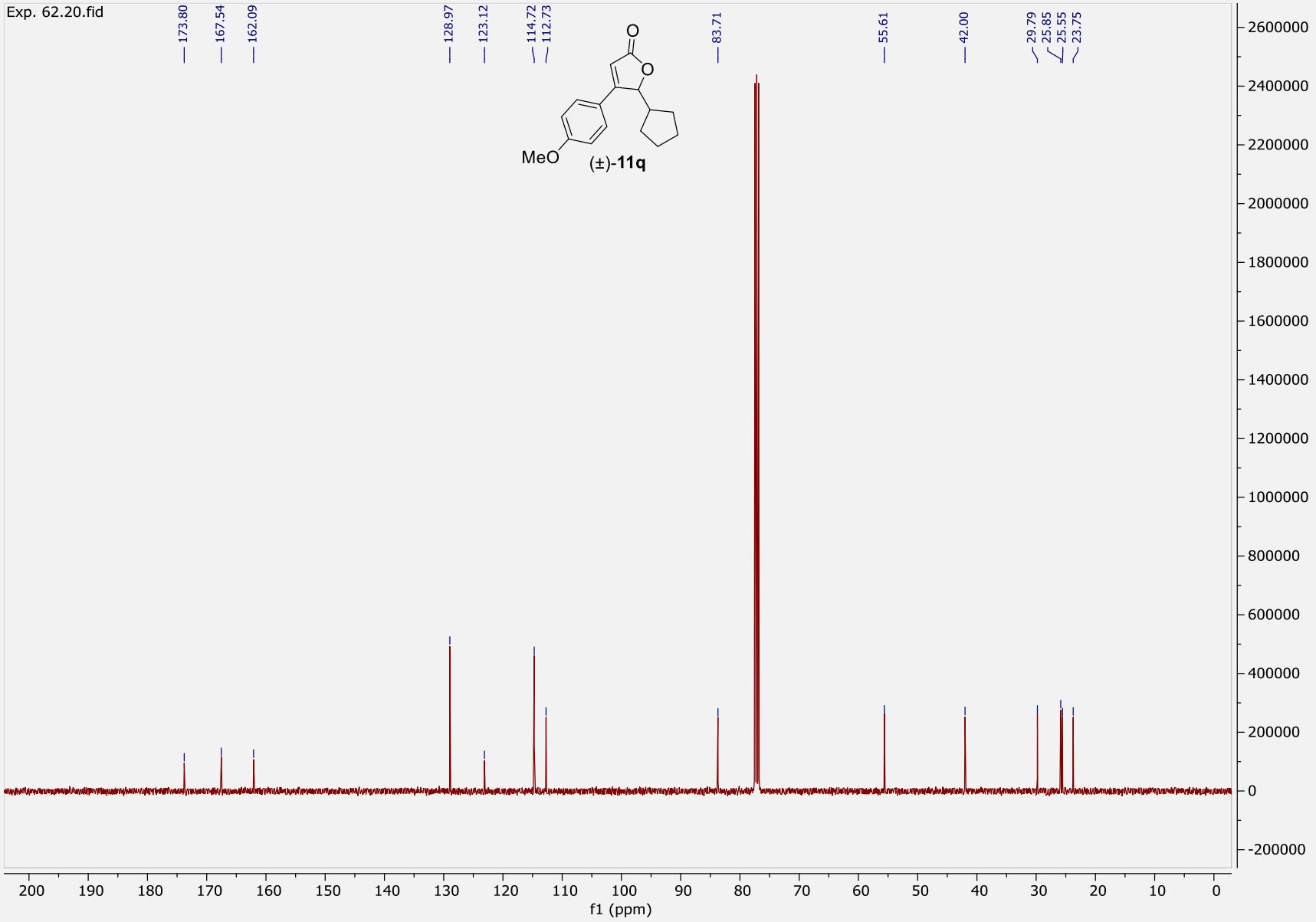

Exp. 66.10.fid
O
O
Me
(±)-11o
3.05
0.98
1.00
1.03
3.03
1.11
2.11
2.11
f1 (ppm)

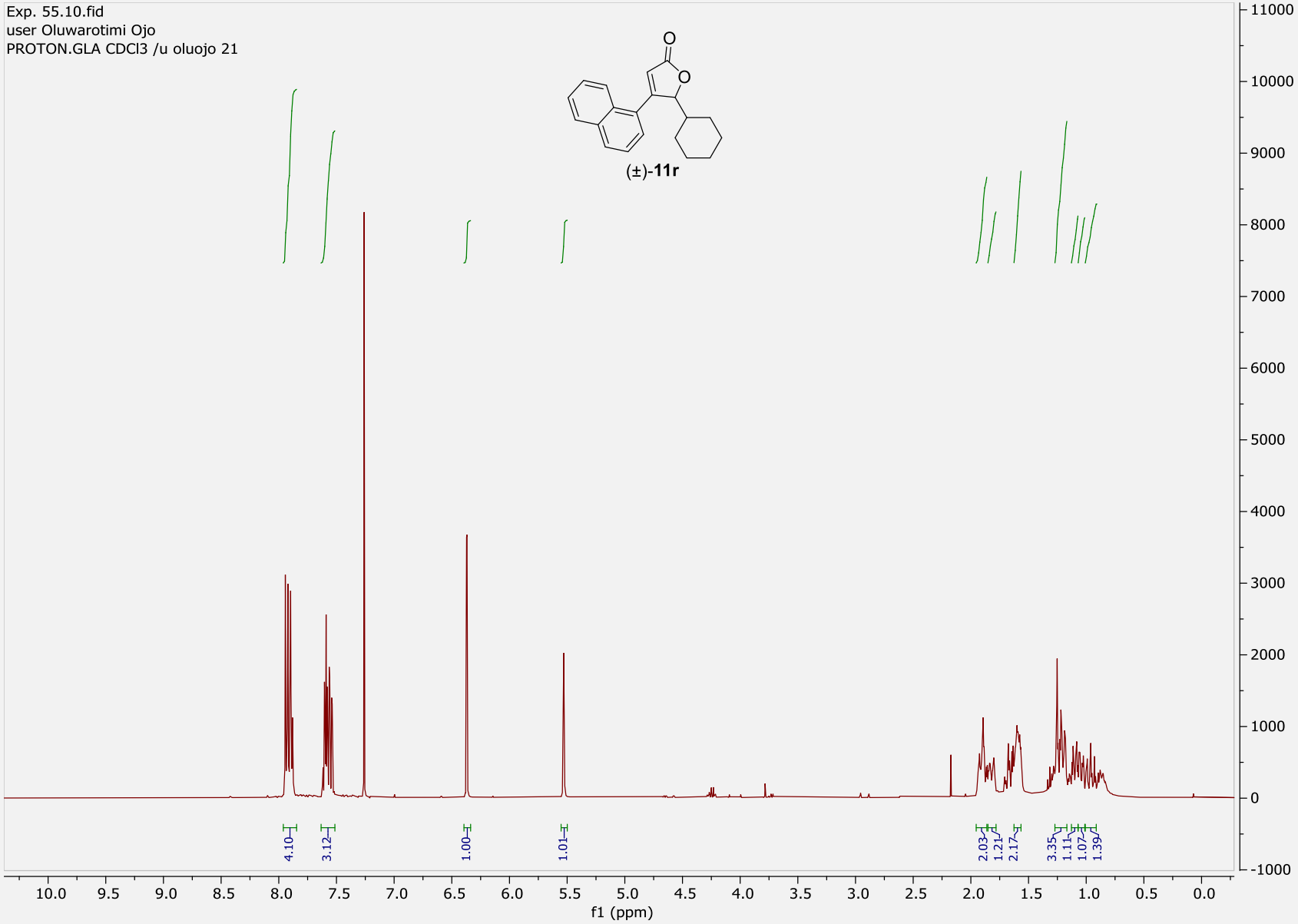

Exp. 66.11.fid
user Oluwarotimi Ojo
C13CPD256.GLA CDCl3 /u oluojo 24
172.92
168.47
139.03
132.13
130.52
129.07
128.23
124.73
114.06
83.89
21.54
14.06
4.20
1.77
O
O
Me
(±)-11o
f1 (ppm)

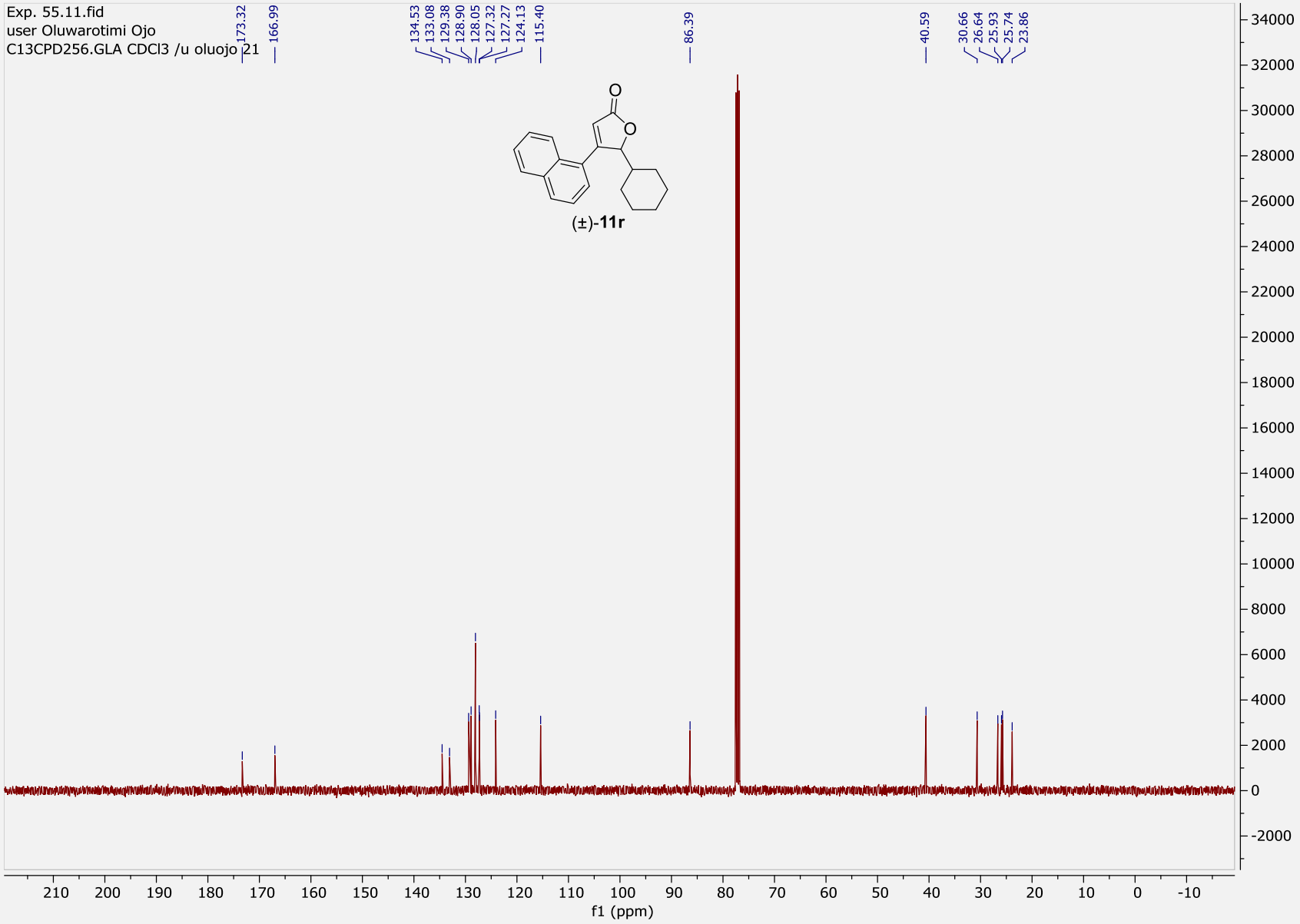

Exp. 61.10.fid
user Oluwarotimi Ojo
PROTON.GLA CDCl3 /u oluojo 24
O
O
Me
(±)-11p
1.00
1.05
2.06
0.98
0.99
1.07
3.02
1.29
1.13
3.31
1.08
f1 (ppm)

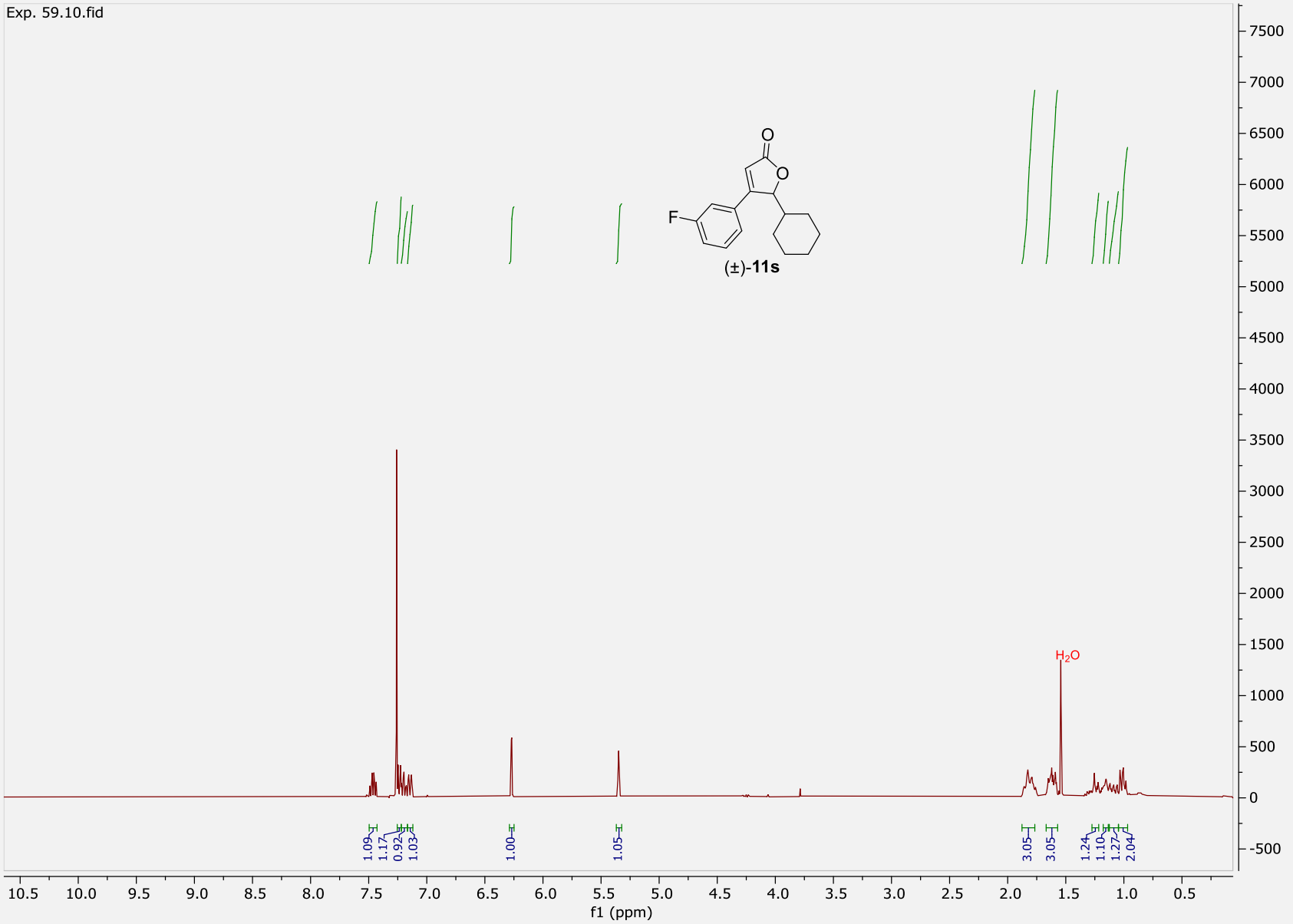

Exp. 61.20.fid
173.51
166.89
139.11
132.13
130.44
129.14
127.82
124.38
114.54
83.41
37.54
24.65
21.52
20.45
17.85
O
O
Me
(±)-11p
2600000
2400000
2200000
2000000
1800000
1600000
1400000
1200000
1000000
800000
600000
400000
200000
0
-200000
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

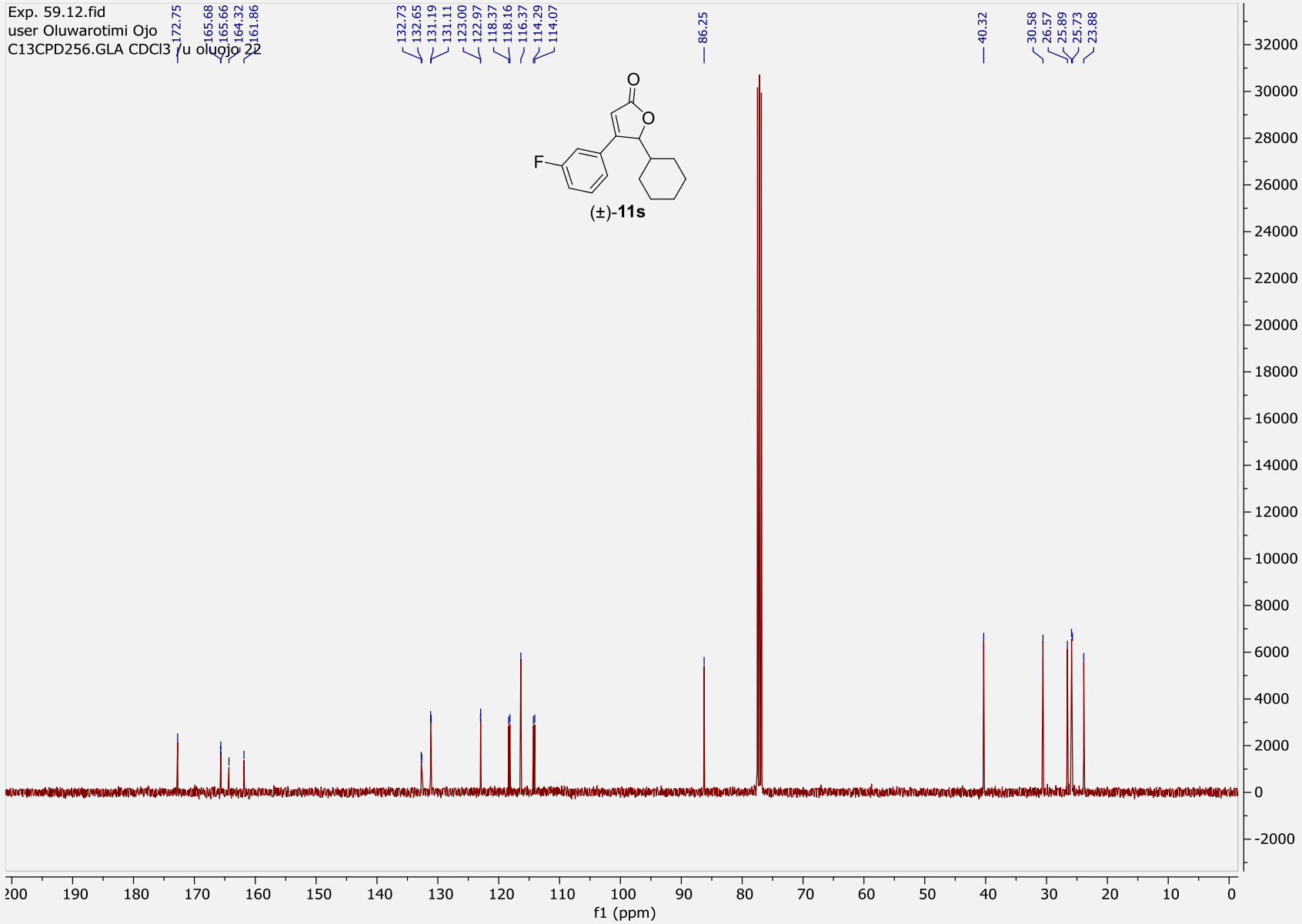

Exp. 62.10.fid
O
O
MeO
(±)-11q
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
2.08
2.07
1.00
1.02
3.05
1.07
2.19
1.20
1.01
1.14
1.13
1.14
0.93
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

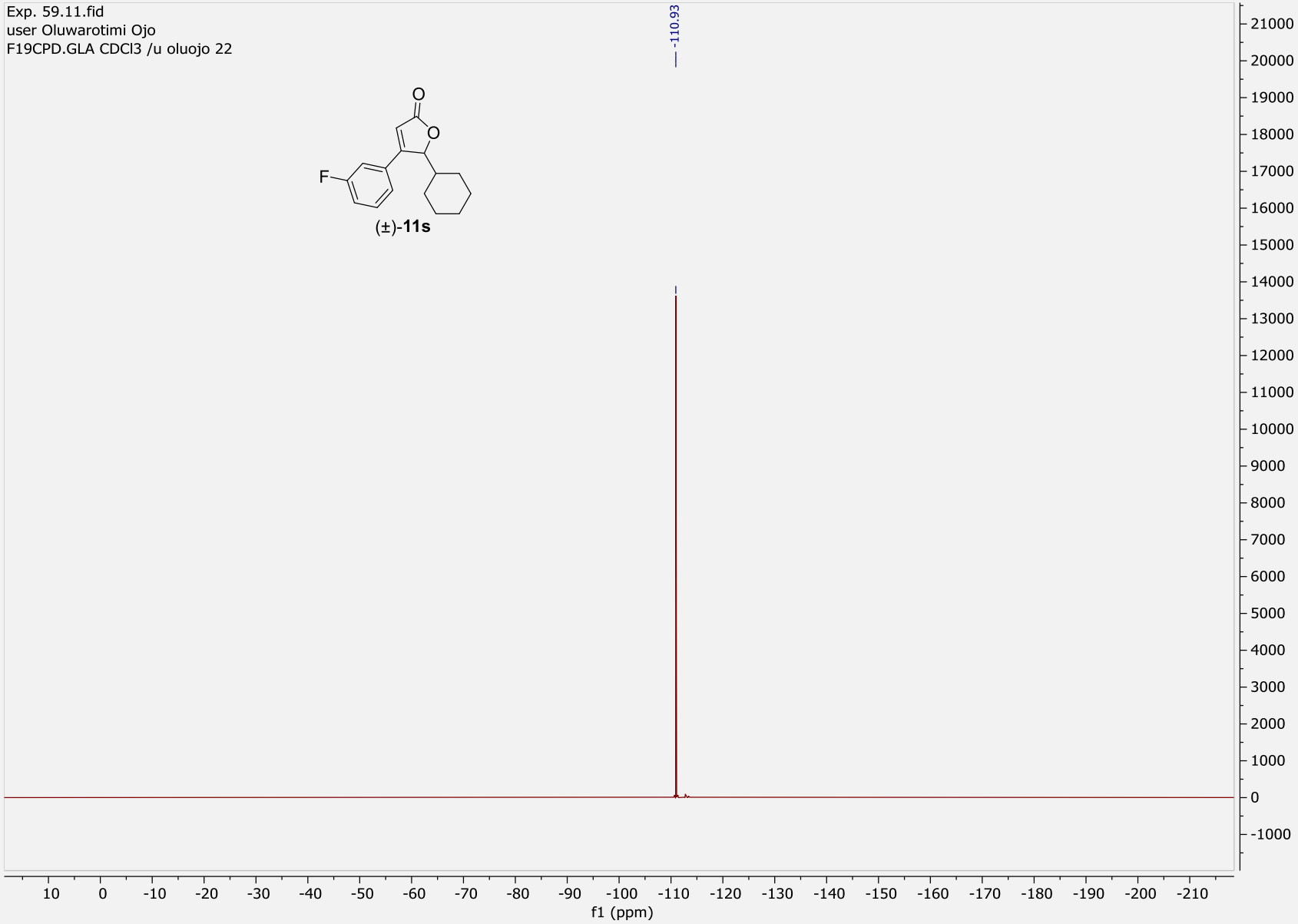

Exp. 62.20.fid
173.80
167.54
162.09
128.97
123.12
114.72
112.73
83.71
55.61
42.00
29.79
25.85
25.55
23.75
MeO
(±)-11q
2600000
2400000
2200000
2000000
1800000
1600000
1400000
1200000
1000000
800000
600000
400000
200000
0
-200000
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

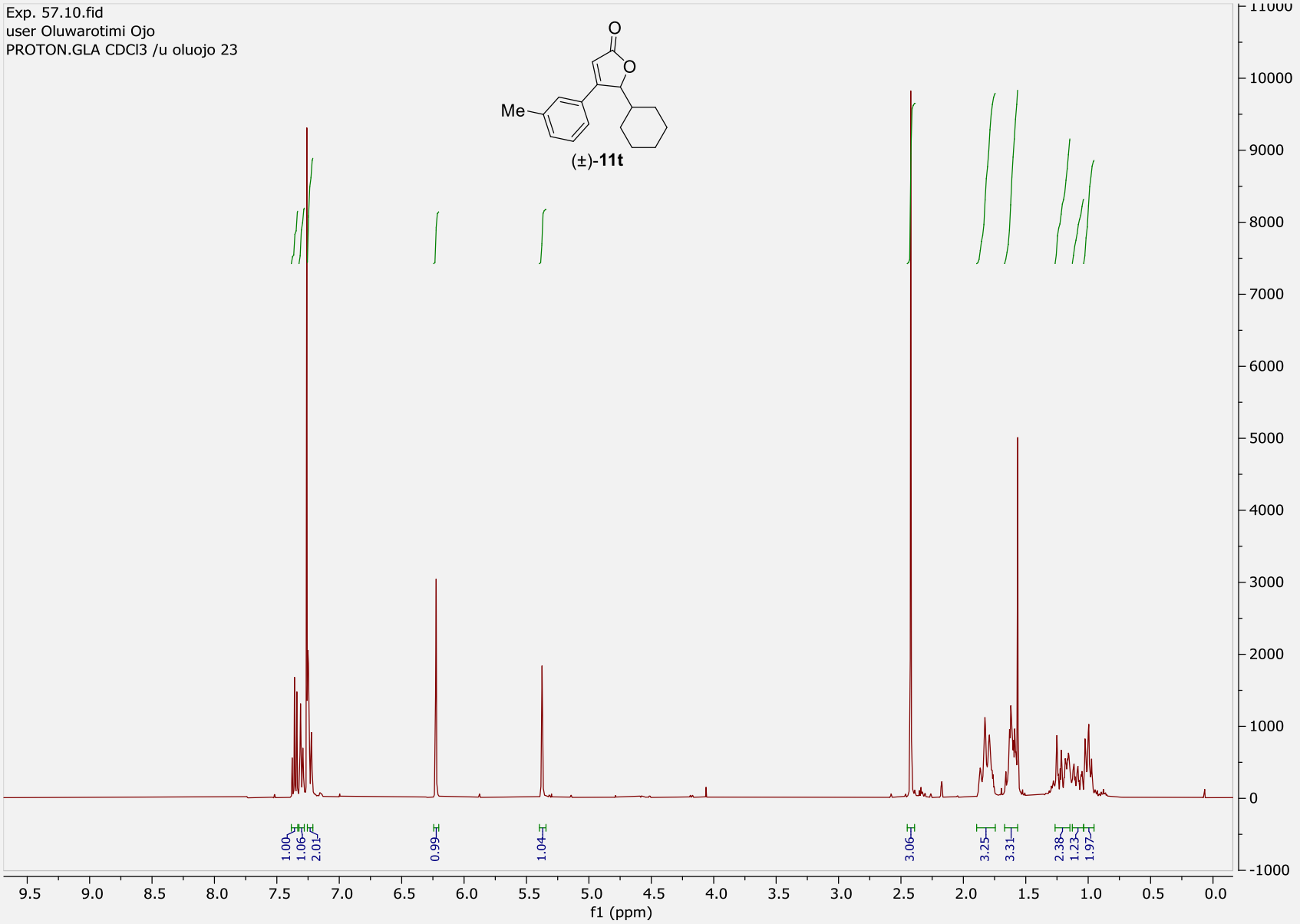

Exp. 55.10.fid
user Oluwarotimi Ojo
PROTON.GLA CDCl3 /u oluojo 21
O
O
(±)-11r
4.10
3.12
1.00
1.01
2.03
1.21
2.17
3.35
1.11
1.07
1.39
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

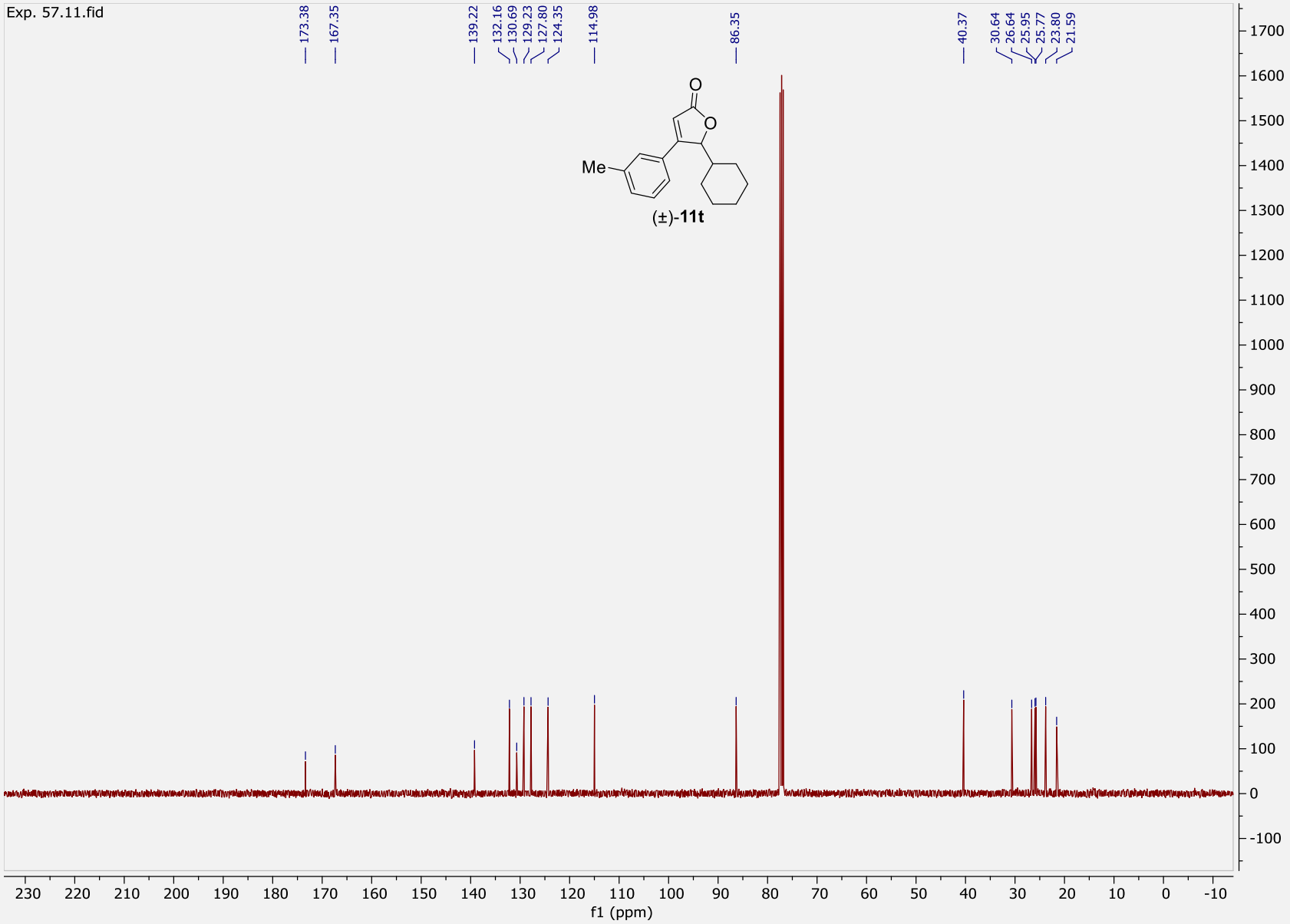

Exp. 55.11.fid
user Oluwarotimi Ojo
C13CPD256.GLA CDCl3 /u oluojo 21
173.32
166.99
134.53
133.08
129.38
128.90
128.05
127.32
127.27
124.13
115.40
86.39
40.59
30.66
26.64
25.93
25.74
23.86
O
O
(±)-11r
f1 (ppm)

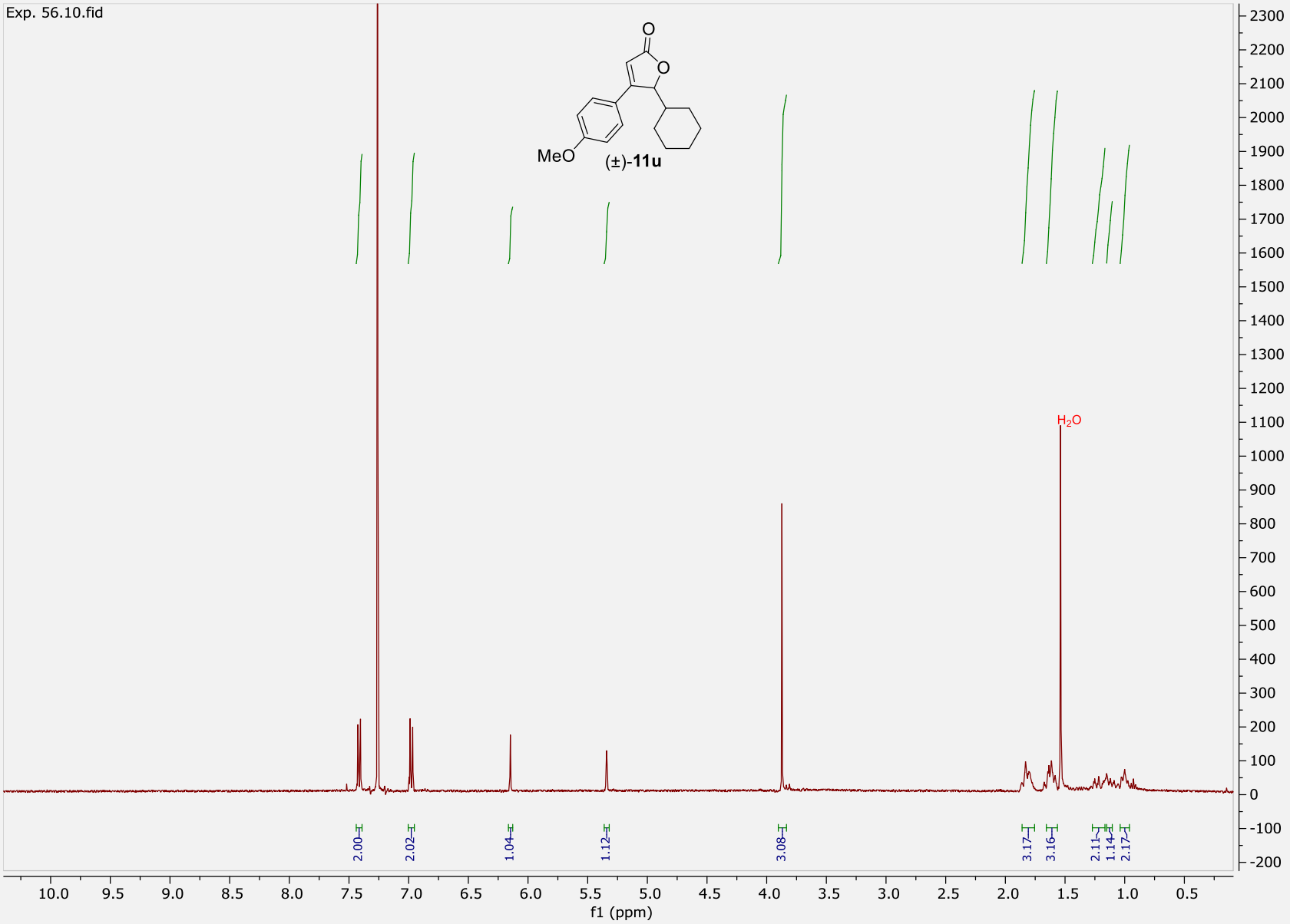

Exp. 59.10.fid
O
O
F
(±)-11s
H2O
1.09
1.17
0.92
1.03
1.00
1.05
3.05
3.05
1.24
1.10
1.27
2.04
f1 (ppm)

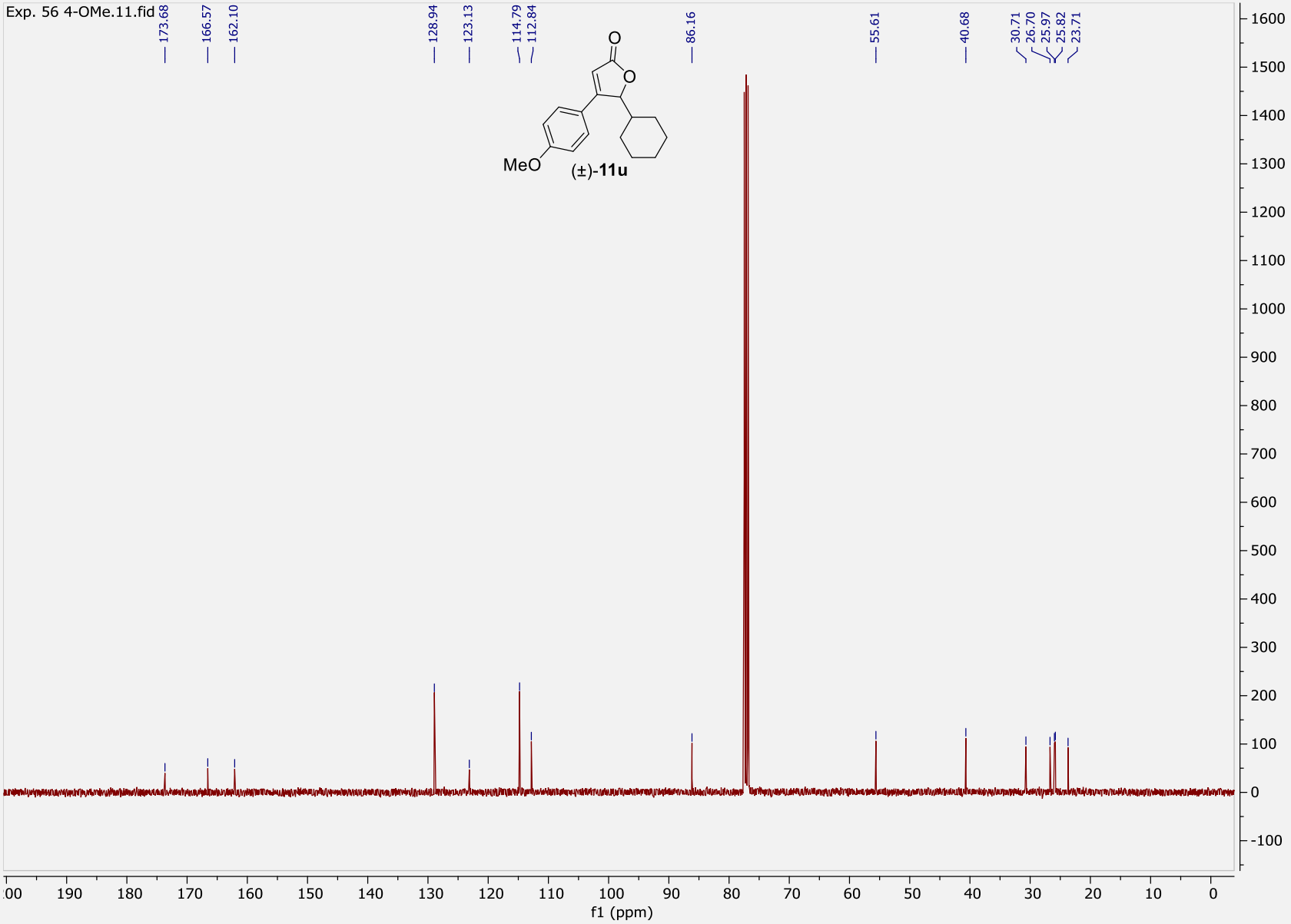

Exp. 59.12.fid
user Oluwarotimi Ojo
C13CPD256.GLA CDCl3 /u oluojo 22
172.75
165.68
165.66
164.32
161.86
132.73
132.65
131.19
131.11
123.00
122.97
118.37
118.16
116.37
114.29
114.07
86.25
40.32
30.58
26.57
25.89
25.73
23.88
O
O
F
(±)-11s
f1 (ppm)

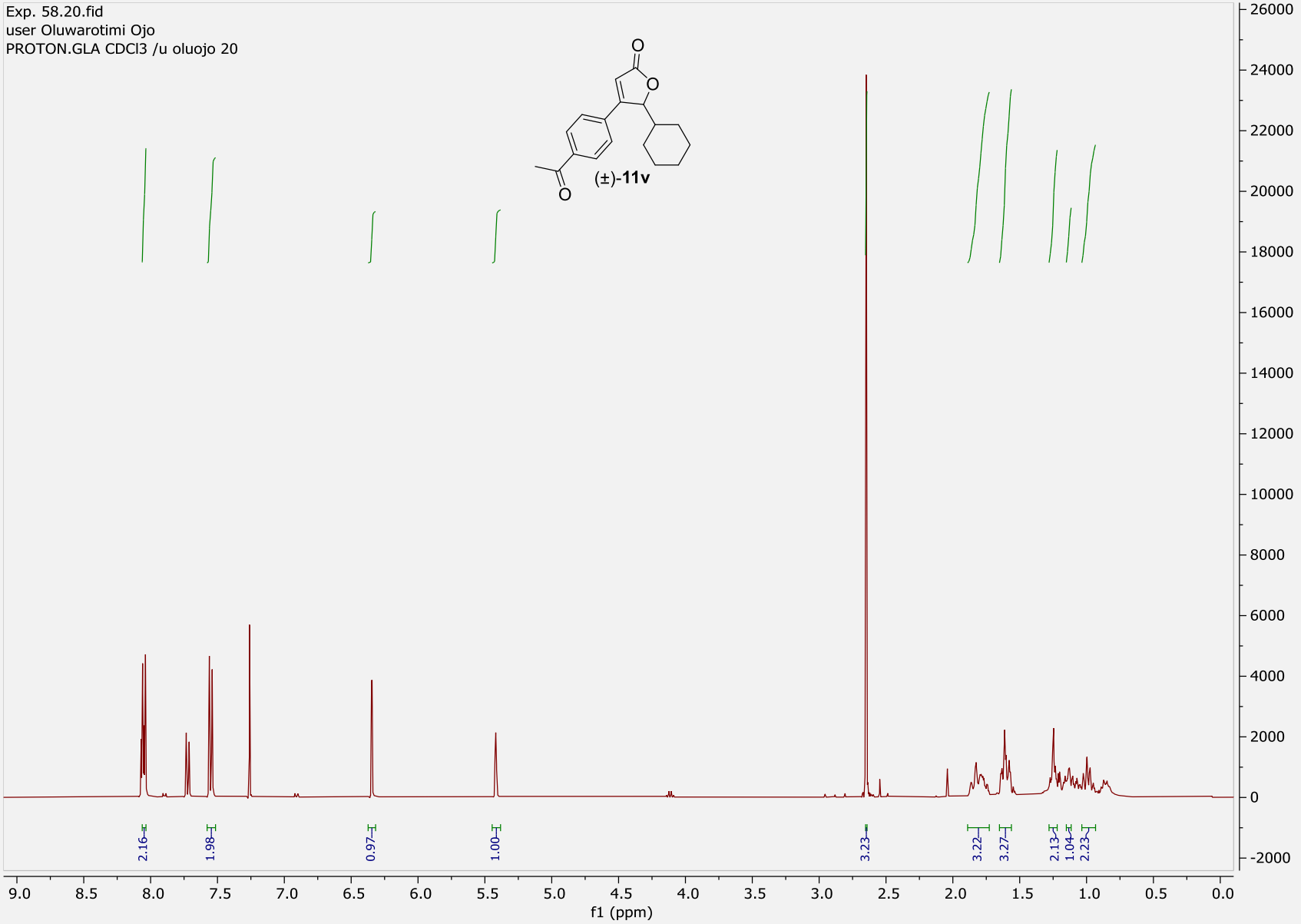

Exp. 59.11.fid
user Oluwarotimi Ojo
F19CPD.GLA CDCl3 /u oluojo 22
-110.93
O
O
F
(±)-11s
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

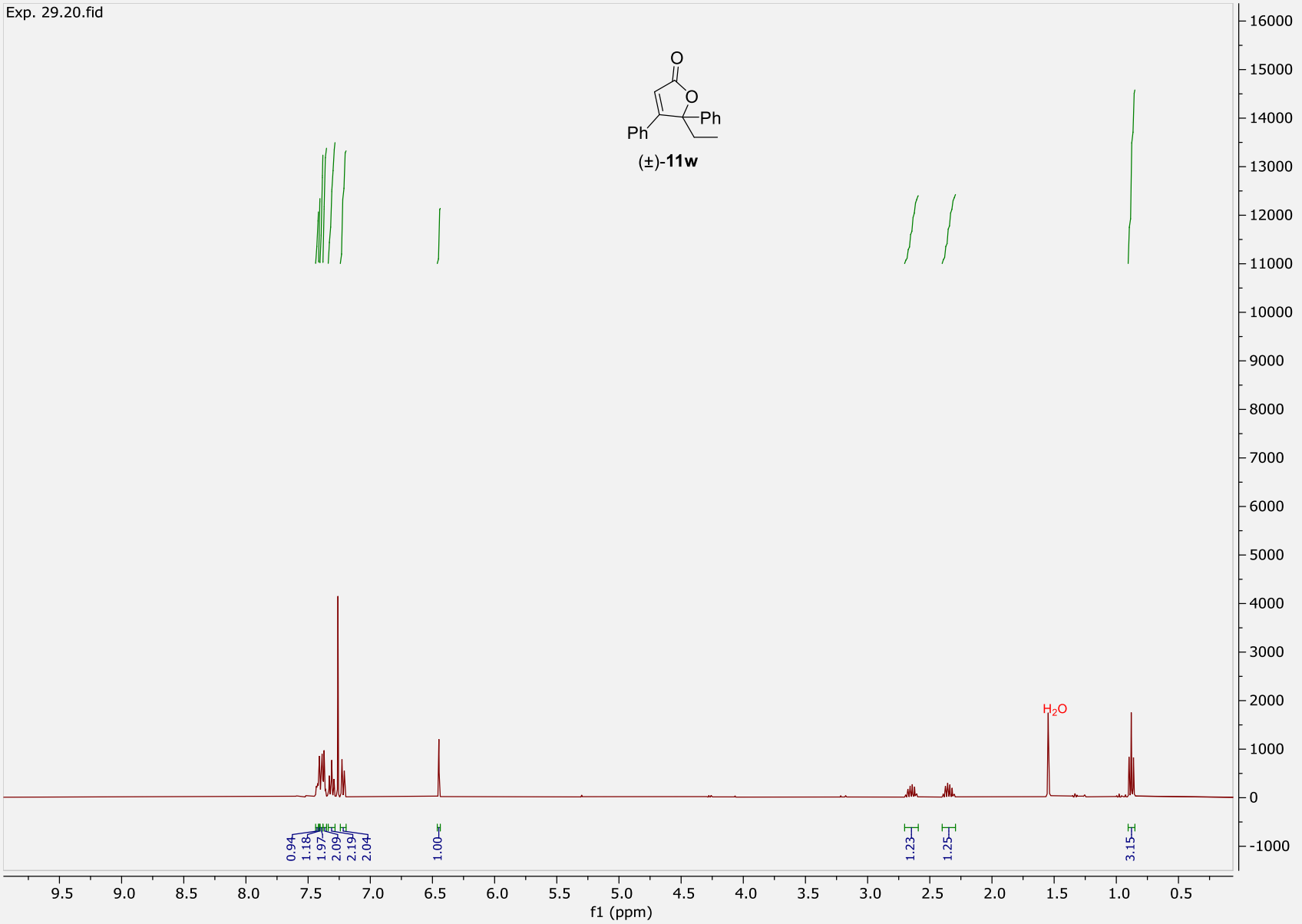

Exp. 57.10.fid
user Oluwarotimi Ojo
PROTON.GLA CDCl3 /u oluojo 23
O
O
Me
(±)-11t
1.00
1.06
2.01
0.99
1.04
3.06
3.25
3.31
2.38
1.23
1.97
f1 (ppm)

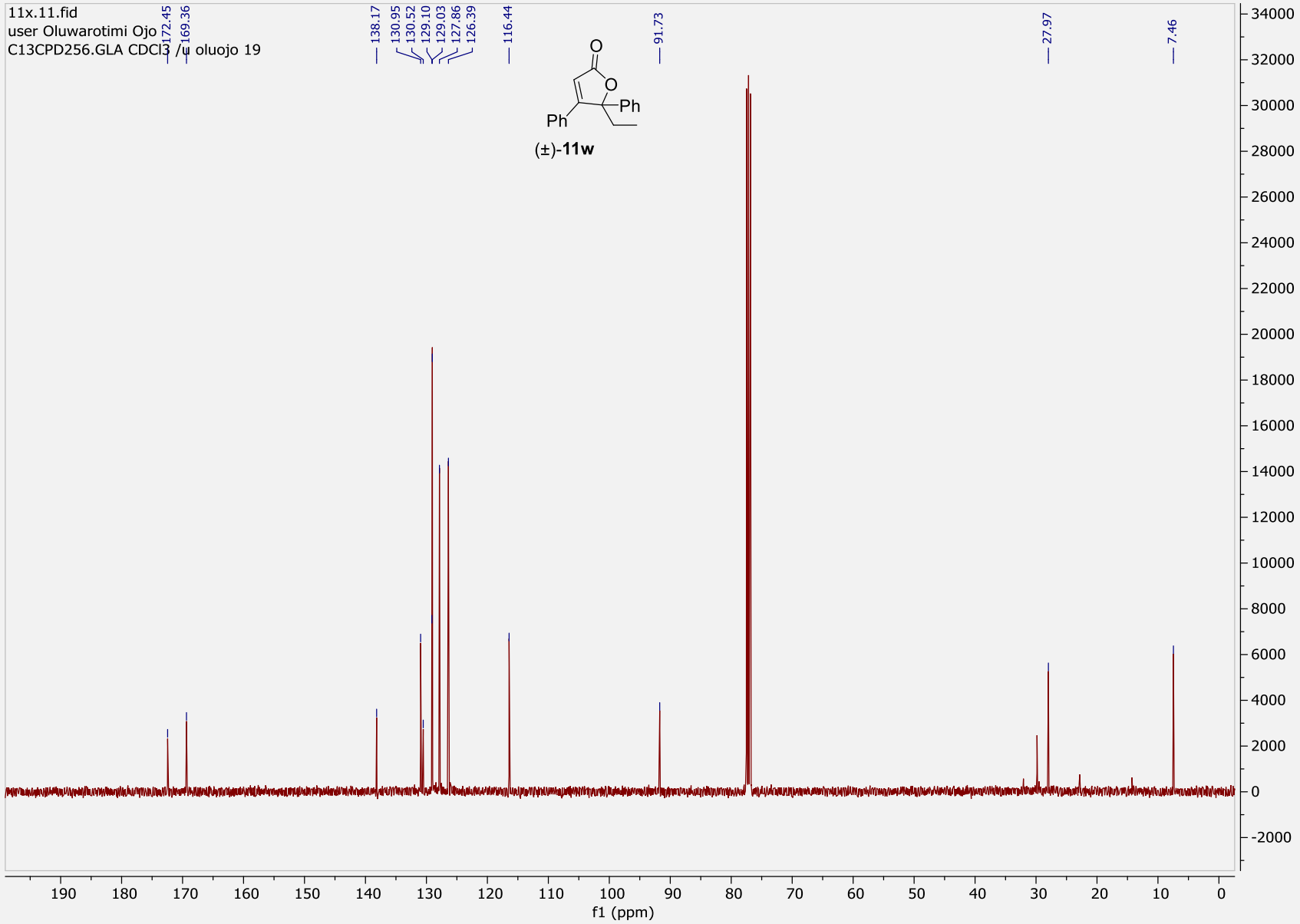

Exp. 57.11.fid
173.38
167.35
139.22
132.16
130.69
129.23
127.80
124.35
114.98
86.35
40.37
30.64
26.64
25.95
25.77
23.80
21.59
O
O
Me
(±)-11t
f1 (ppm)


Exp. 56.10.fid
O
O
MeO
(±)-11u
H2O
2.00
2.02
1.04
1.12
3.08
3.17
3.16
2.11
1.14
2.17
f1 (ppm)


Exp. 56 4-OMe.11.fid
173.68
166.57
162.10
128.94
123.13
114.79
112.84
86.16
55.61
40.68
30.71
26.70
25.97
25.82
23.71
MeO
(±)-11u
f1 (ppm)


Exp. 58.20.fid
user Oluwarotimi Ojo
PROTON.GLA CDCl3 /u oluojo 20
(±)-11v
2.16
1.98
0.97
1.00
3.23
3.22
3.27
2.13
1.04
2.23
f1 (ppm)

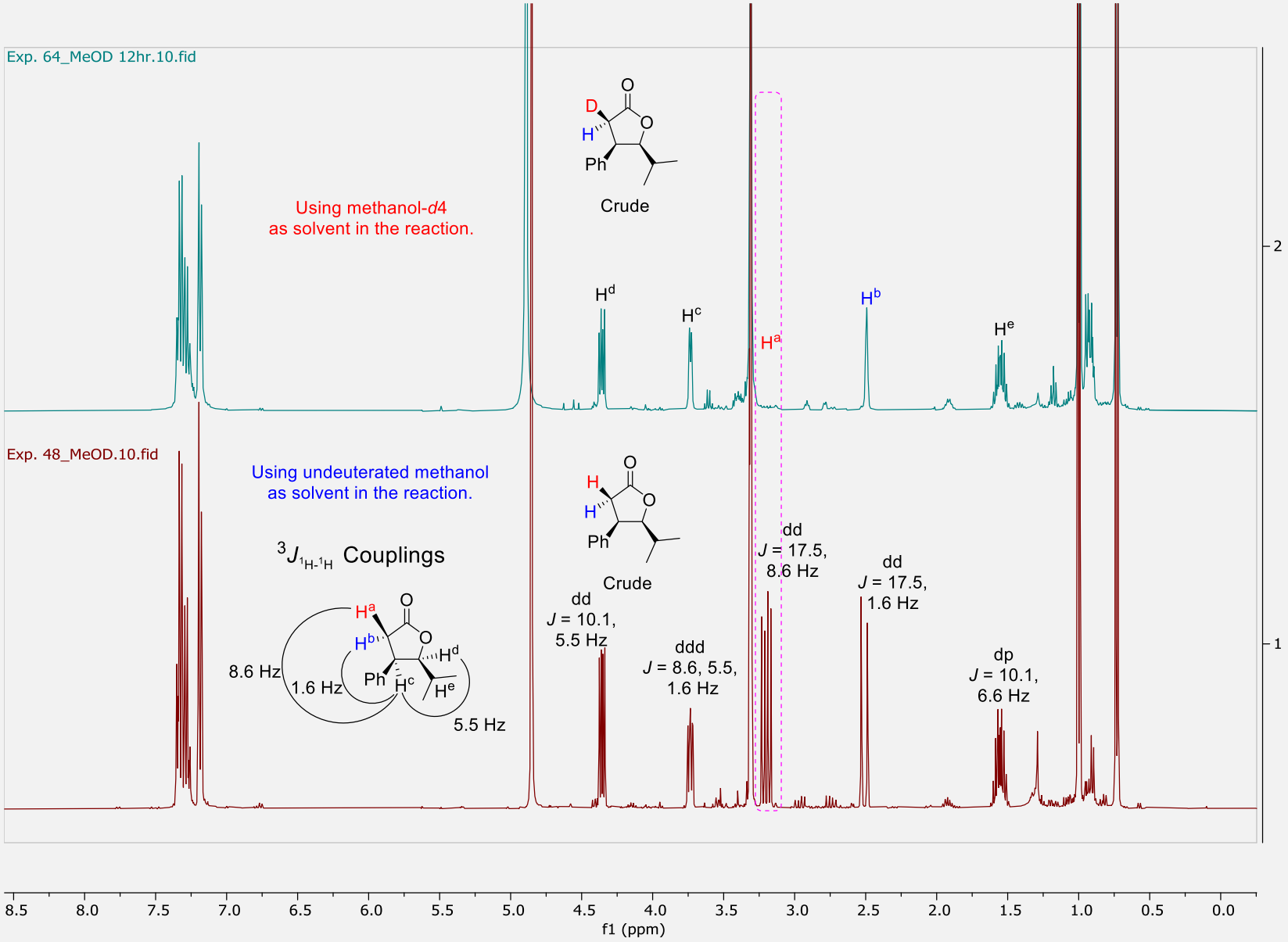

Exp. 29.20.fid
O
O
Ph
Ph
(±)-11w
H2O
0.94
1.18
1.97
2.09
2.19
2.04
1.00
1.23
1.25
3.15
f1 (ppm)

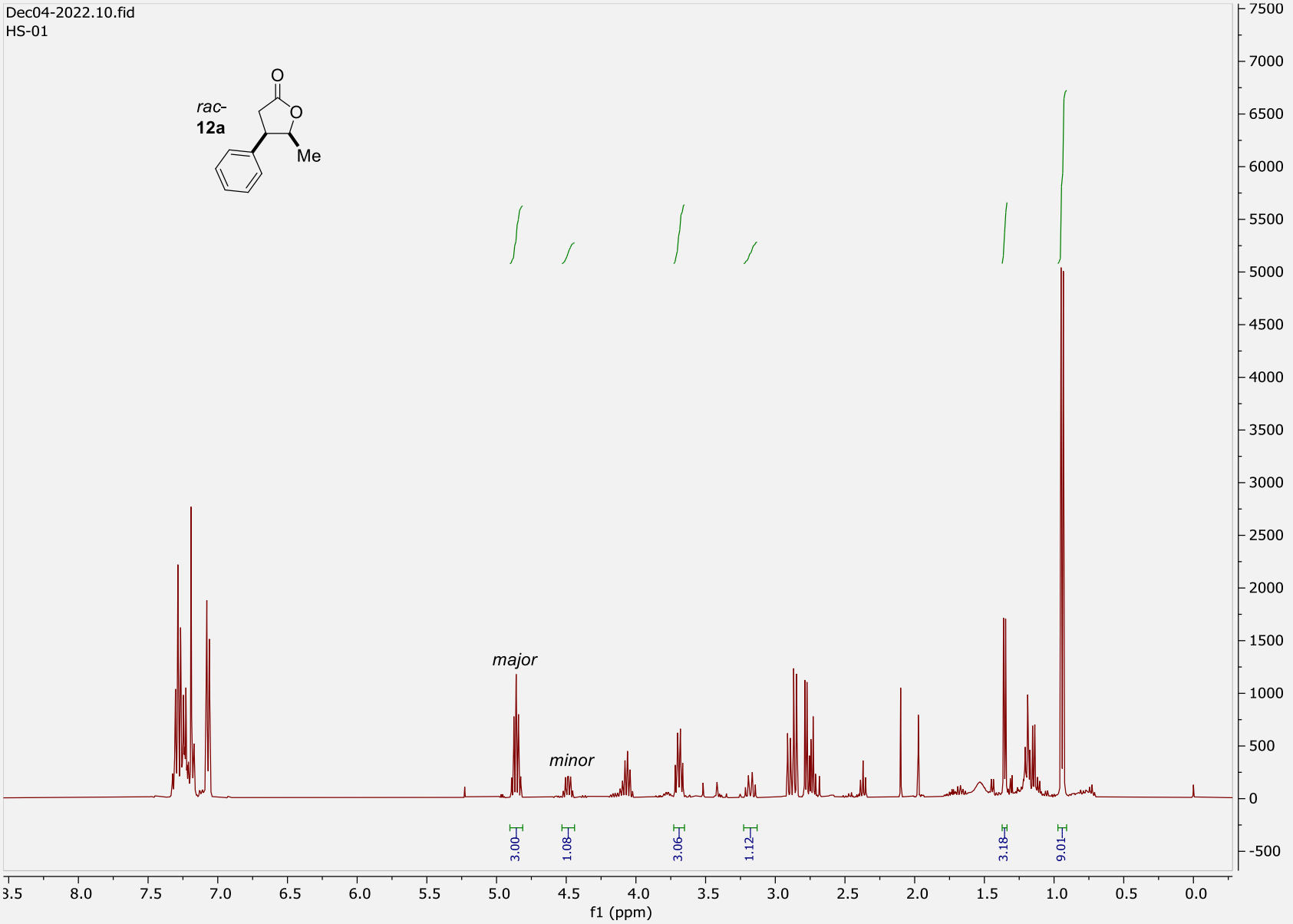

11x.11.fid
user Oluwarotimi Ojo
C13CPD256.GLA CDCl3 /u oluojo 19
172.45
169.36
138.17
130.95
130.52
129.10
129.03
127.86
126.39
116.44
91.73
27.97
7.46
O
O
Ph
Ph
(±)-11w
f1 (ppm)

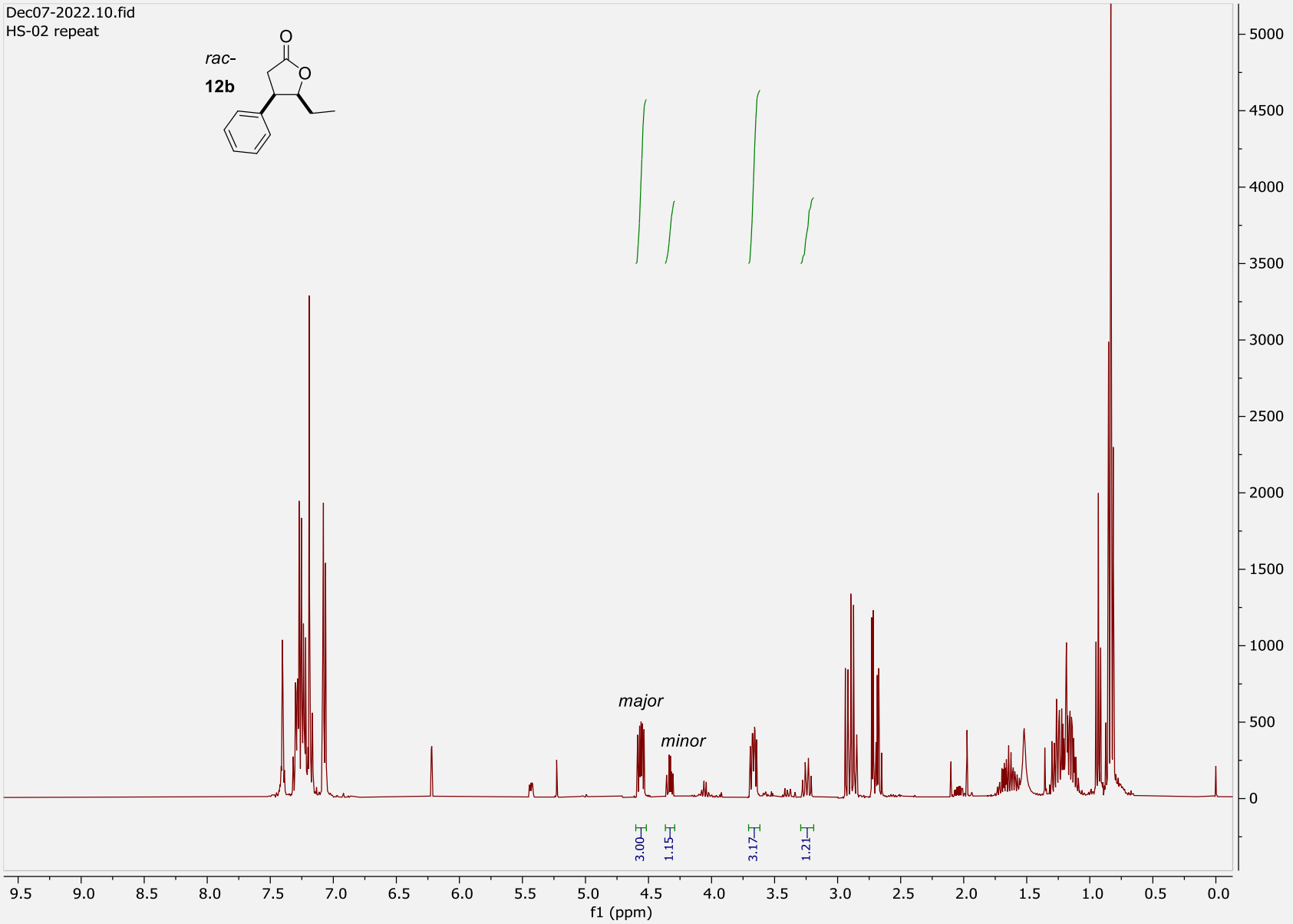
^{1}H NMR Spectra

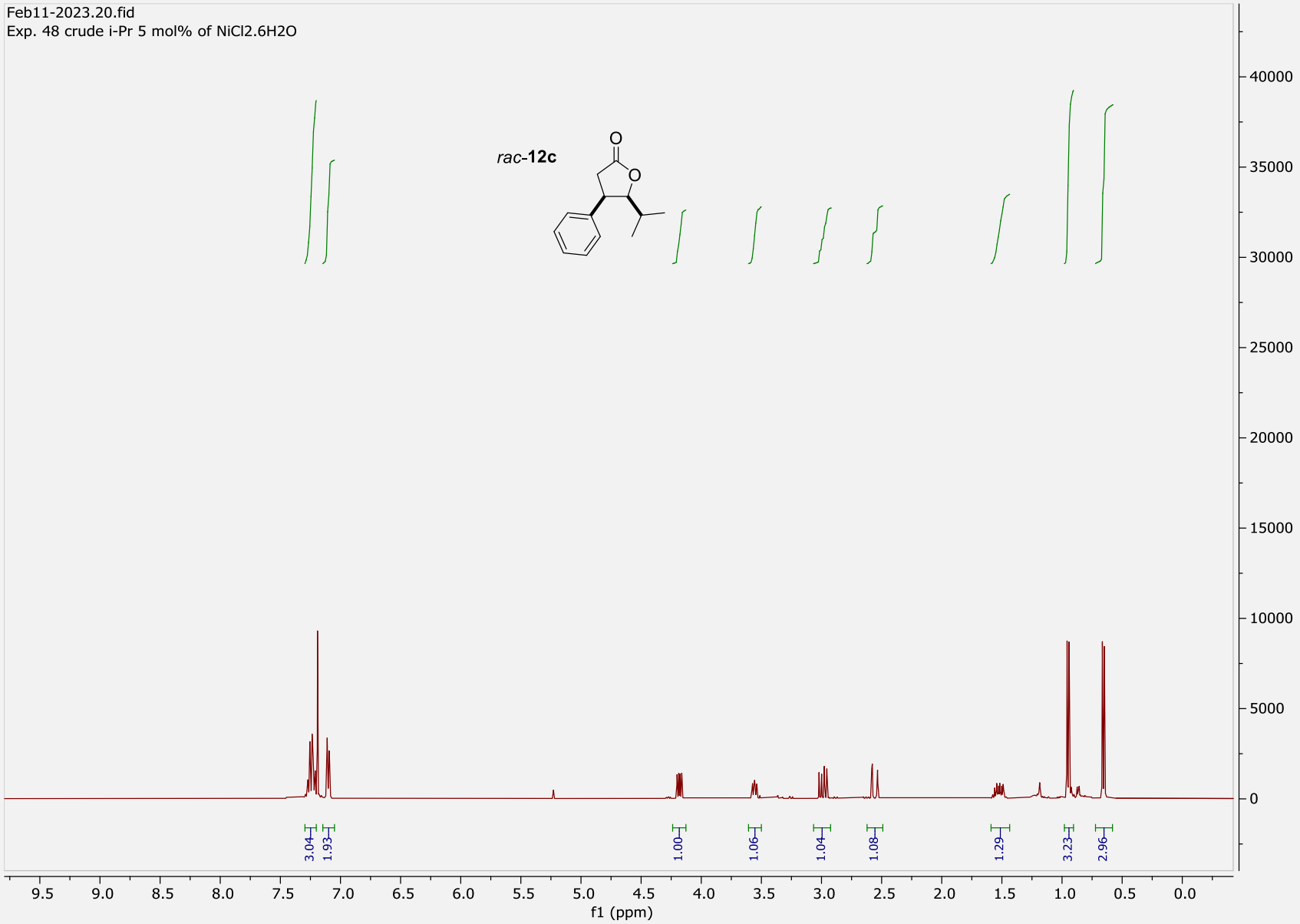
of

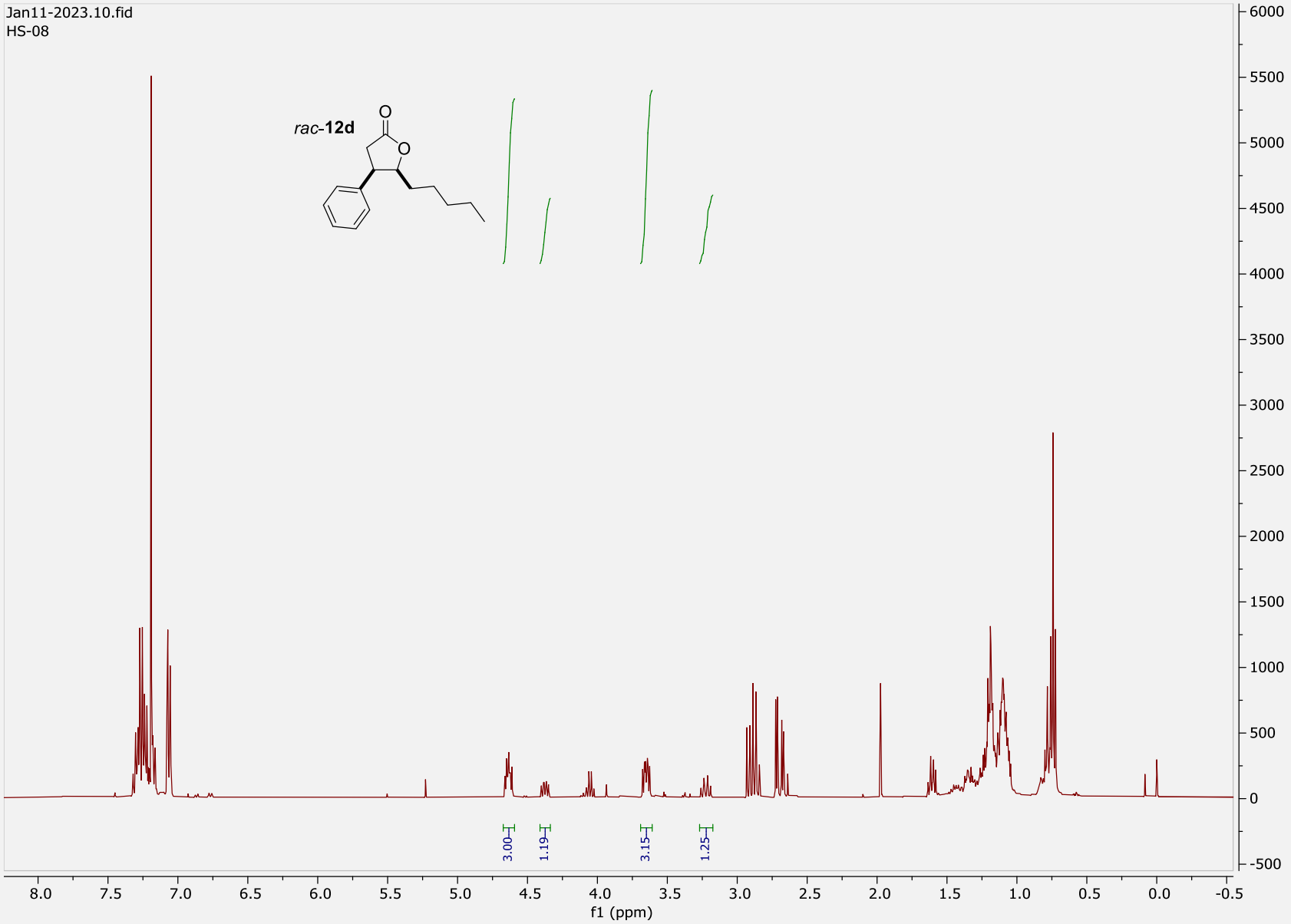
Crude γ-Butyrolactones

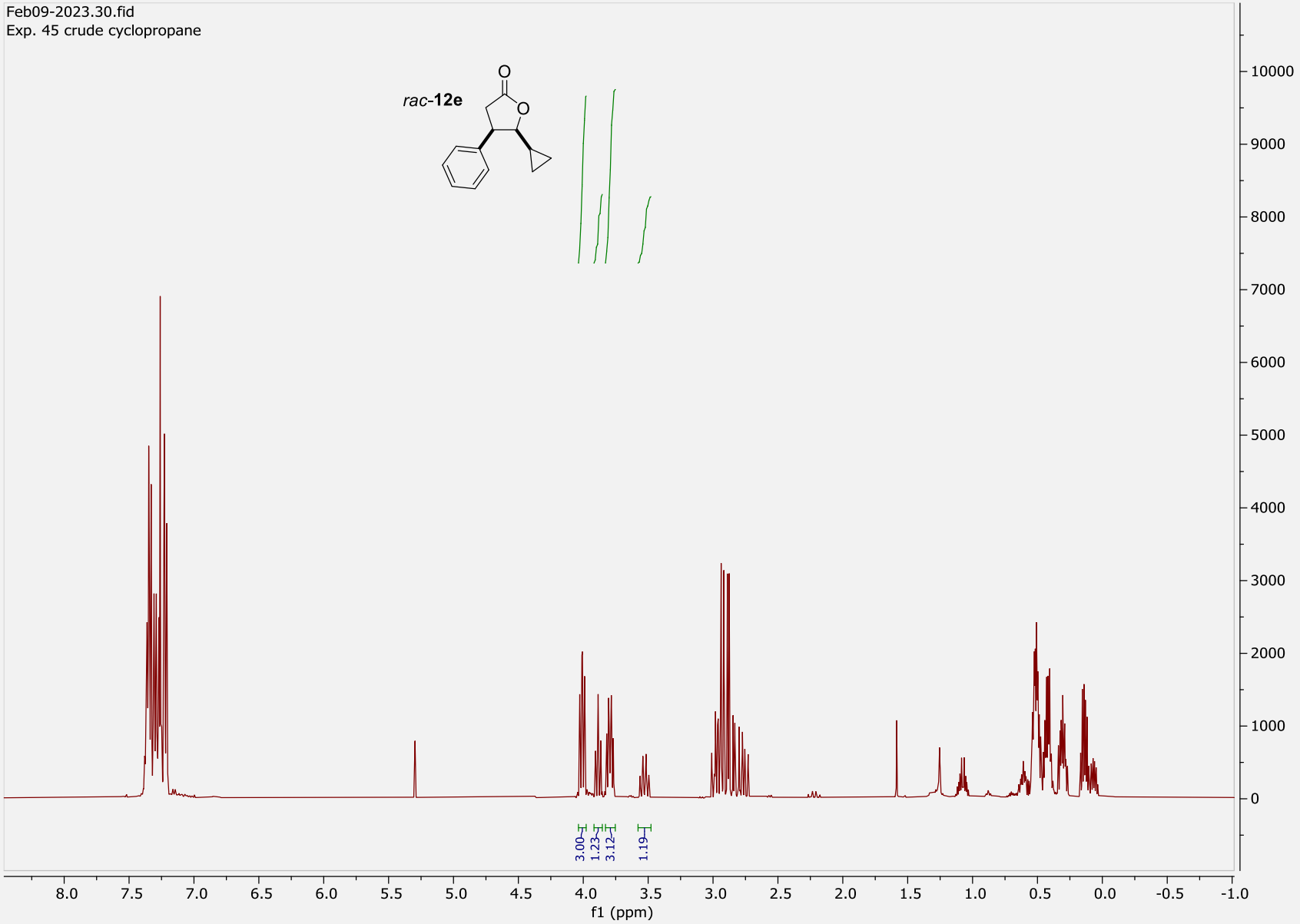

Exp. 64_MeOD 12hr.10.fid
Using methanol-d4
as solvent in the reaction.
Crude
Hd
Hc
Ha
Hb
He
Exp. 48_MeOD.10.fid
Using undeuterated methanol
as solvent in the reaction.
3J1H-1H Couplings
8.6 Hz
1.6 Hz
5.5 Hz
Crude
dd
J = 10.1,
5.5 Hz
ddd
J = 8.6, 5.5,
1.6 Hz
dd
J = 17.5,
8.6 Hz
dd
J = 17.5,
1.6 Hz
dp
J = 10.1,
6.6 Hz
f1 (ppm)

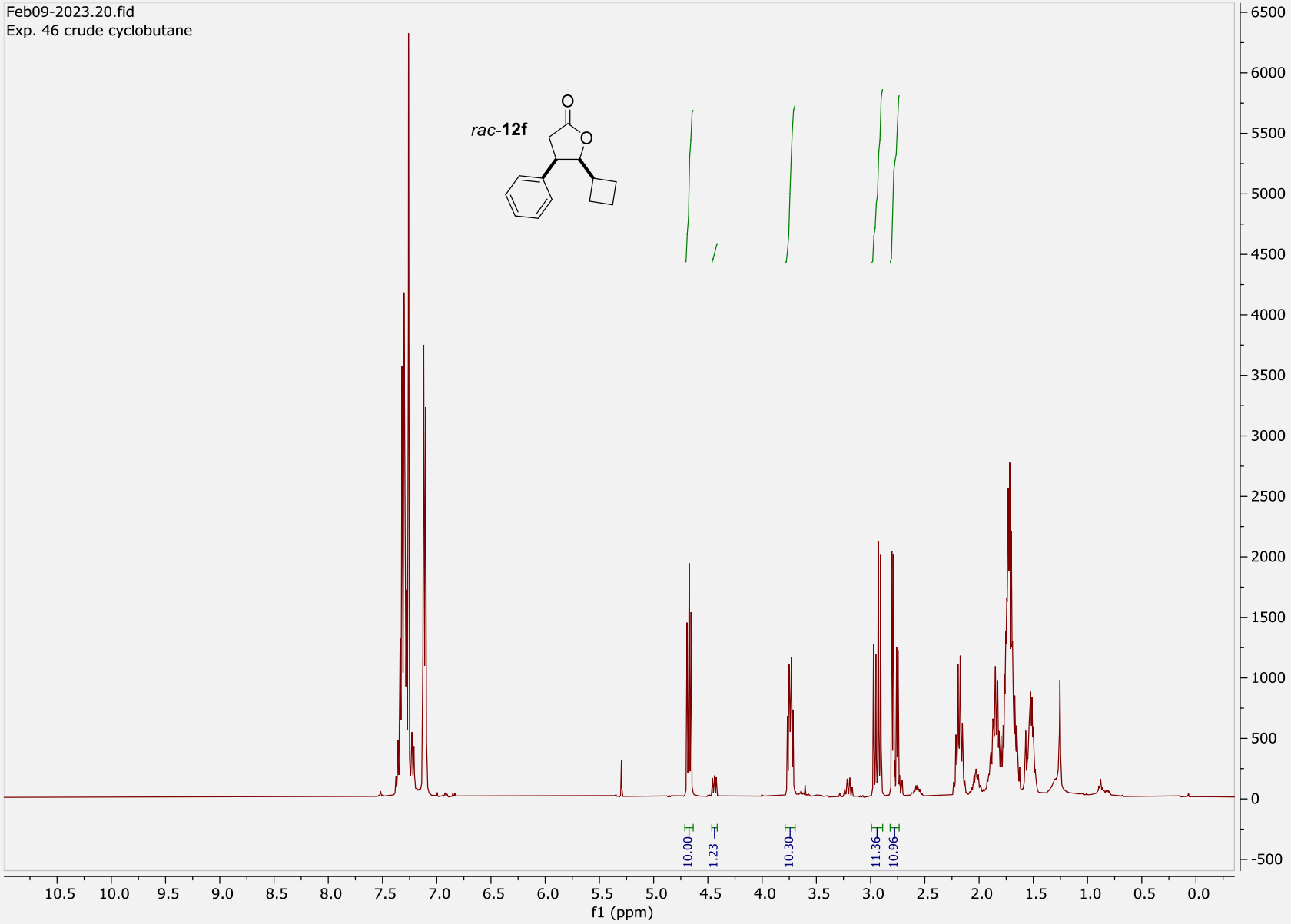

Dec04-2022.10.fid
HS-01
rac-
12a
O
O
Me
major
minor
3.00
1.08
3.06
1.12
3.18
9.01
f1 (ppm)

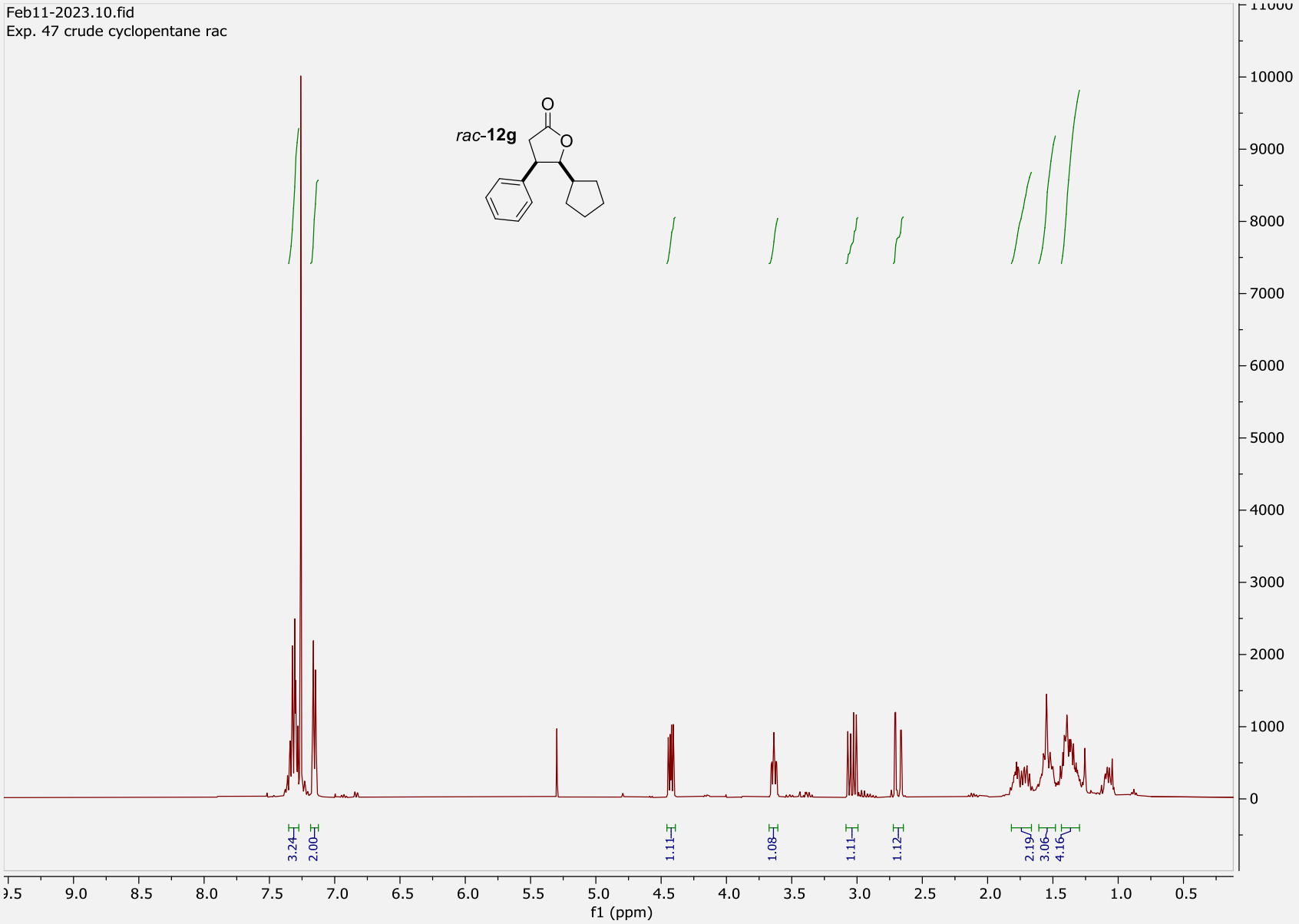

Dec07-2022.10.fid
HS-02 repeat
rac-
12b
major
minor
3.00
1.15
3.17
1.21
f1 (ppm)

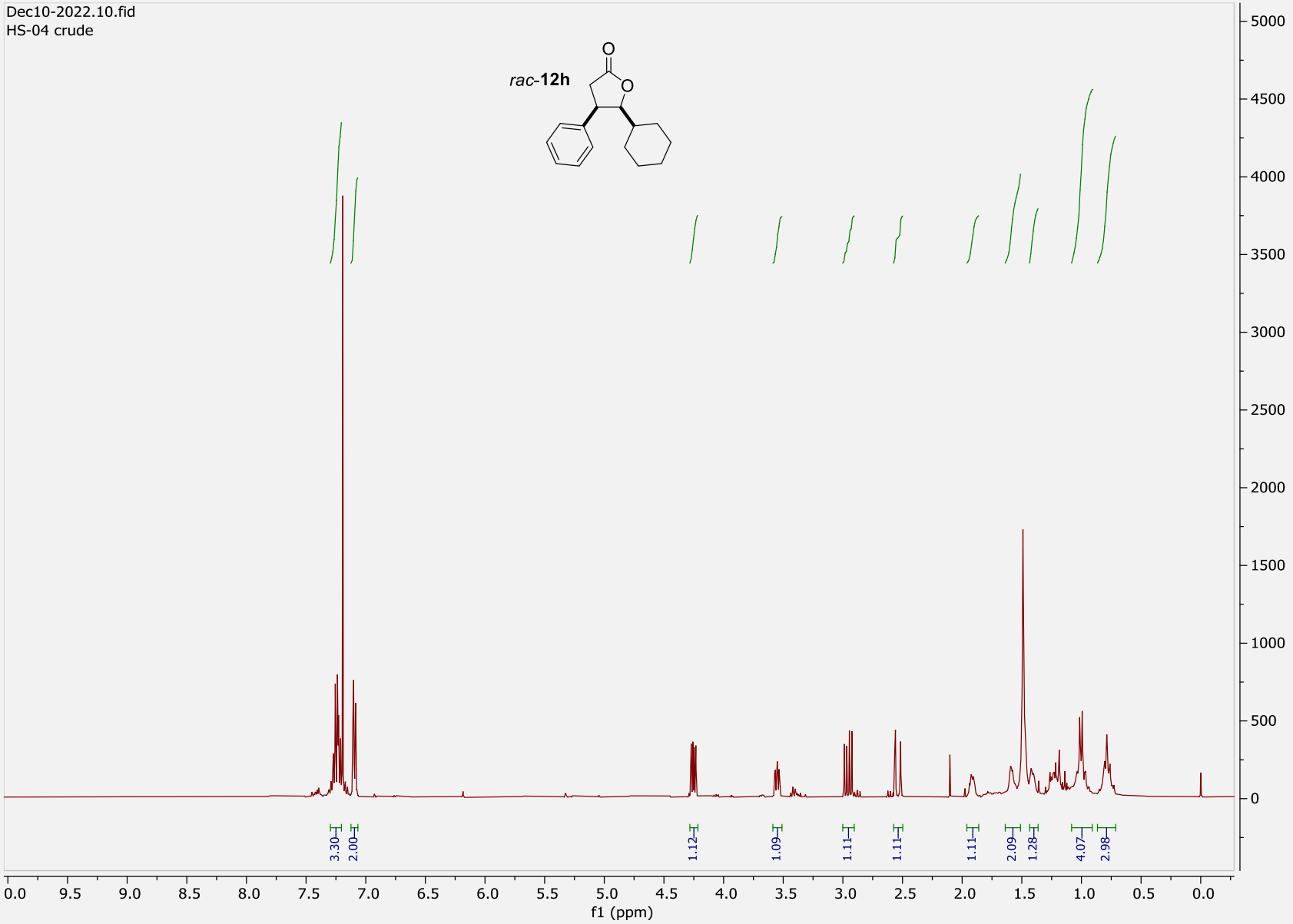

Feb11-2023.20.fid
Exp. 48 crude i-Pr 5 mol% of NiCl2.6H2O
rac-12c
O
O
40000
35000
30000
25000
20000
15000
10000
5000
0
3.04
1.93
1.00
1.06
1.04
1.08
1.29
3.23
2.96
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

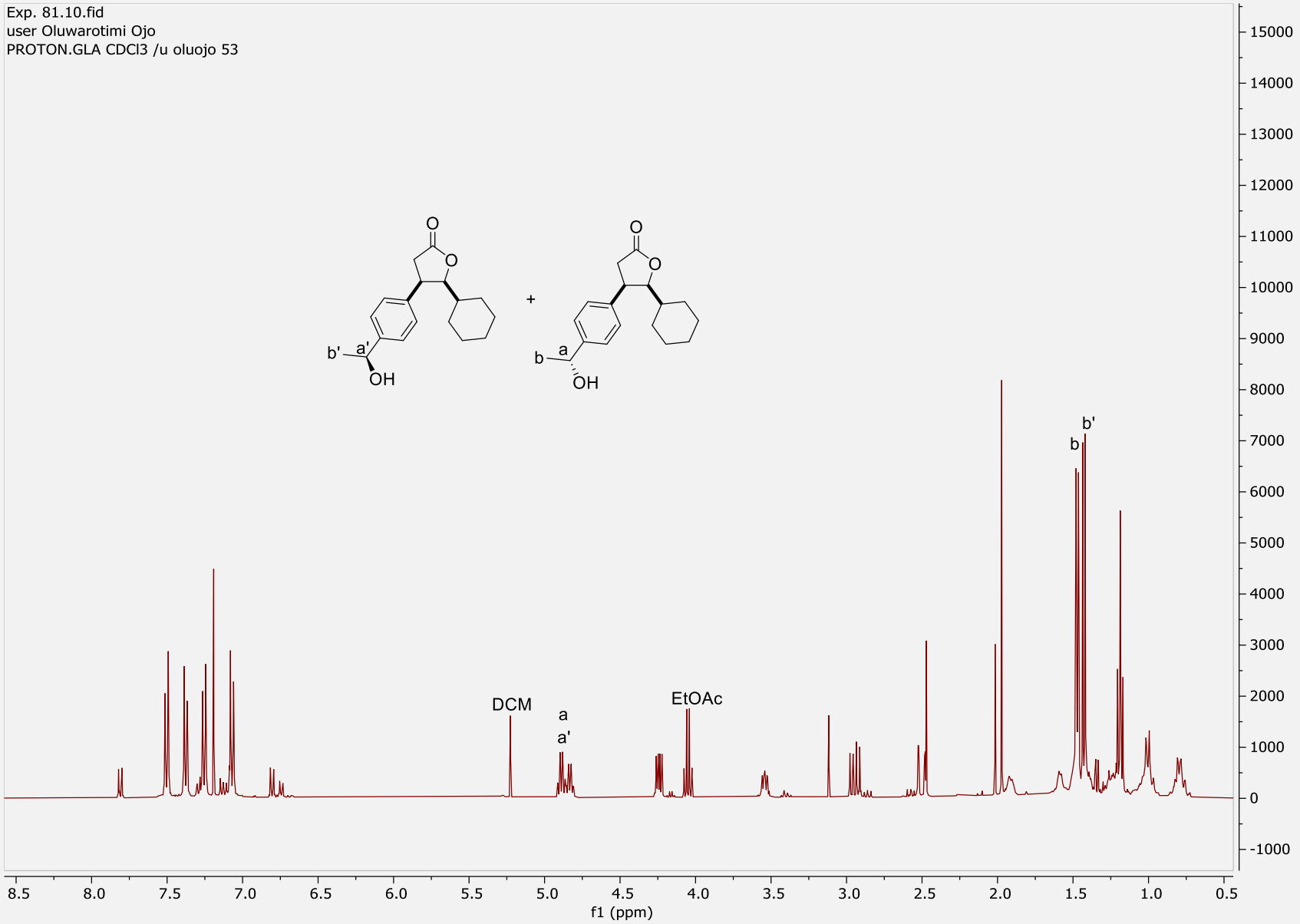

Jan11-2023.10.fid
HS-08
rac-12d
O
O
3.00
1.19
3.15
1.25
f1 (ppm)


Feb09-2023.30.fid
Exp. 45 crude cyclopropane
rac-12e
O
O
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
3.00
1.23
3.12
1.19
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


Feb09-2023.20.fid
Exp. 46 crude cyclobutane
rac-12f
O
O
10.00
1.23
10.30
11.36
10.96
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


Feb11-2023.10.fid
Exp. 47 crude cyclopentane rac
rac-12g
O
O
3.24
2.00
1.11
1.08
1.11
1.12
2.19
3.06
4.16
f1 (ppm)


Dec10-2022.10.fid
HS-04 crude
rac-12h
O
O
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
3.30
2.00
1.12
1.09
1.11
1.11
1.11
2.09
1.28
4.07
2.98
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

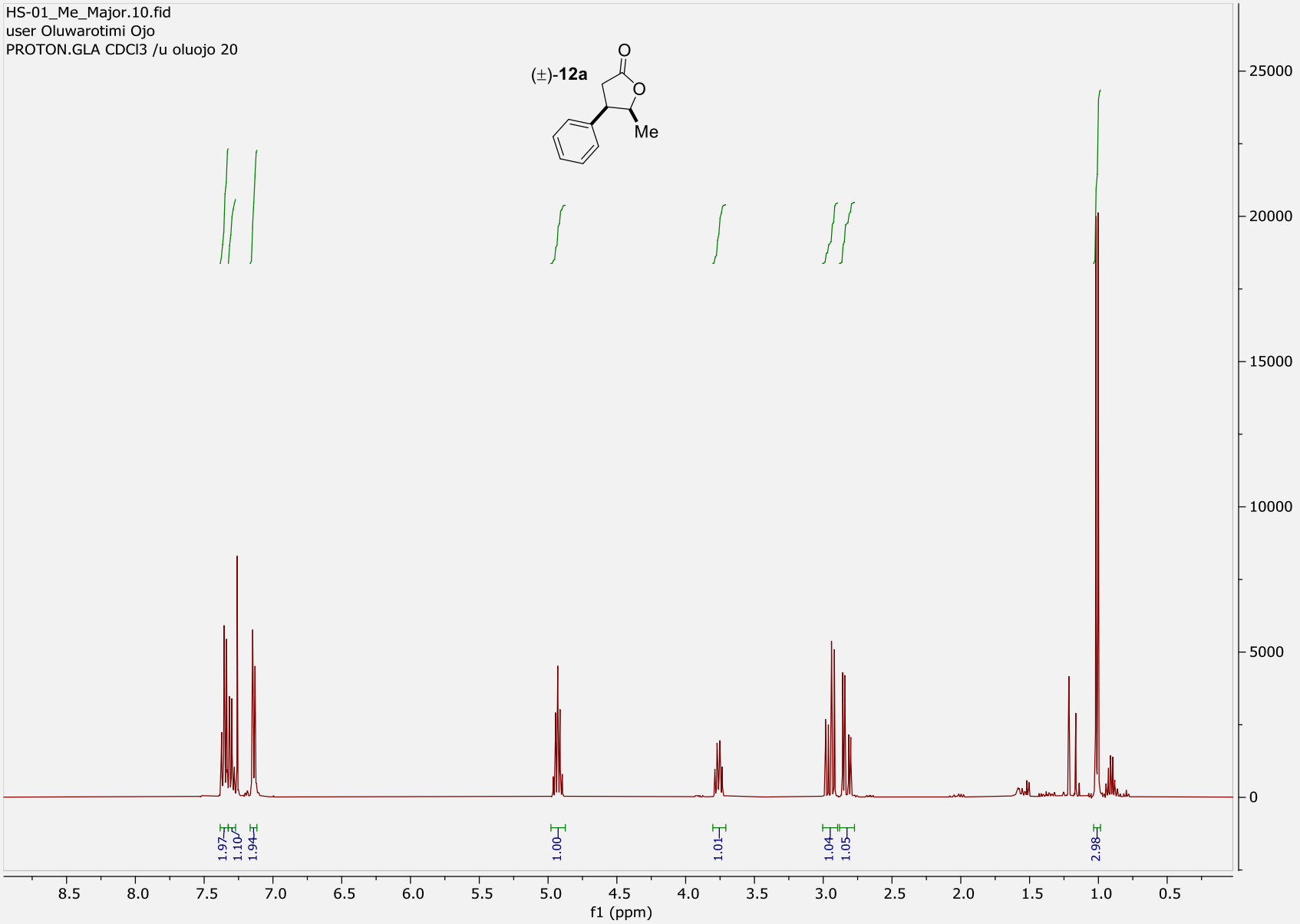

Exp. 81.10.fid
user Oluwarotimi Ojo
PROTON.GLA CDCl3 /u oluojo 53
O
O
b'
a'
OH
+
O
O
b
a
OH
DCM
a
a'
EtOAc
b'
b
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

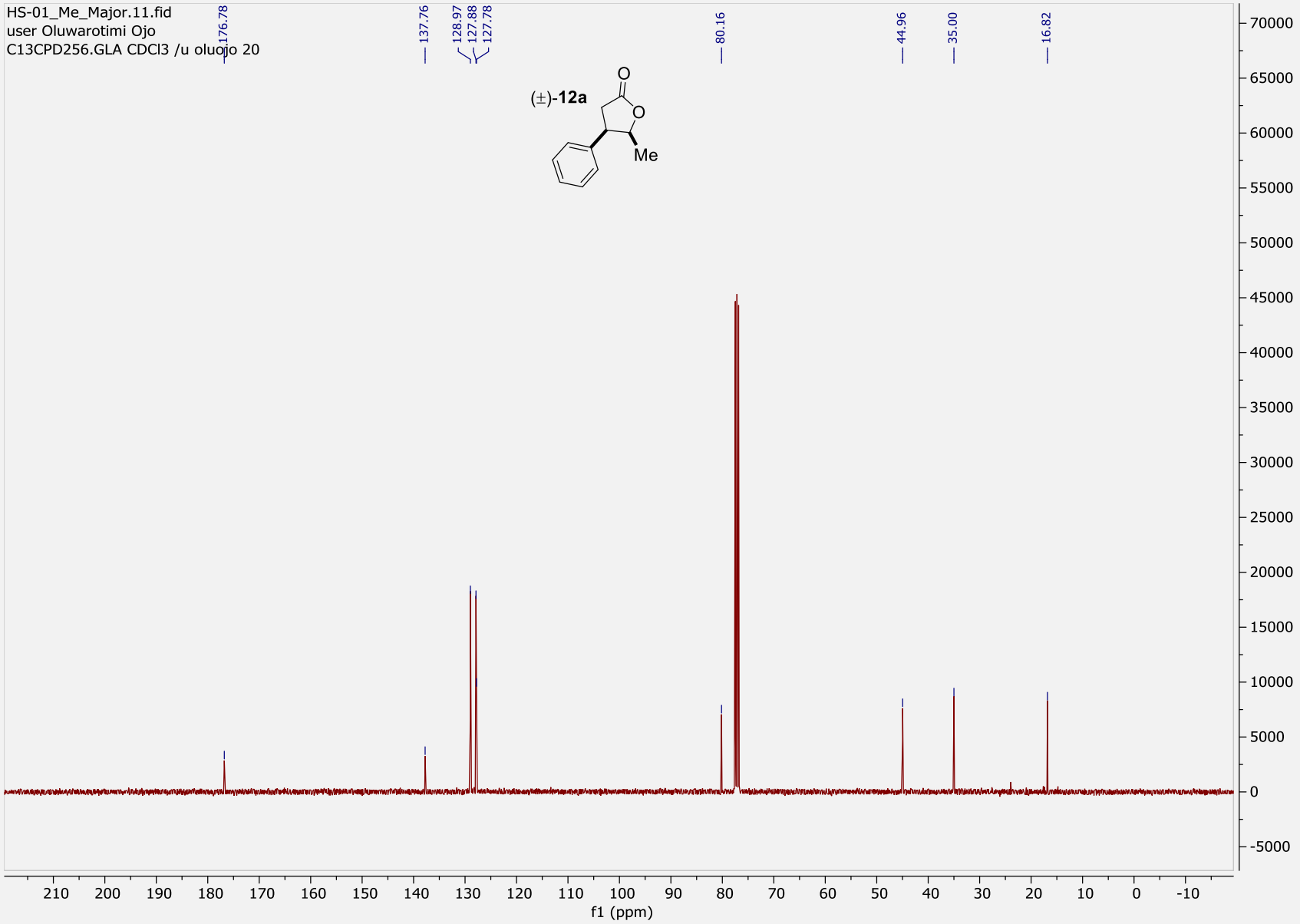
^{1}H and ^{13}C

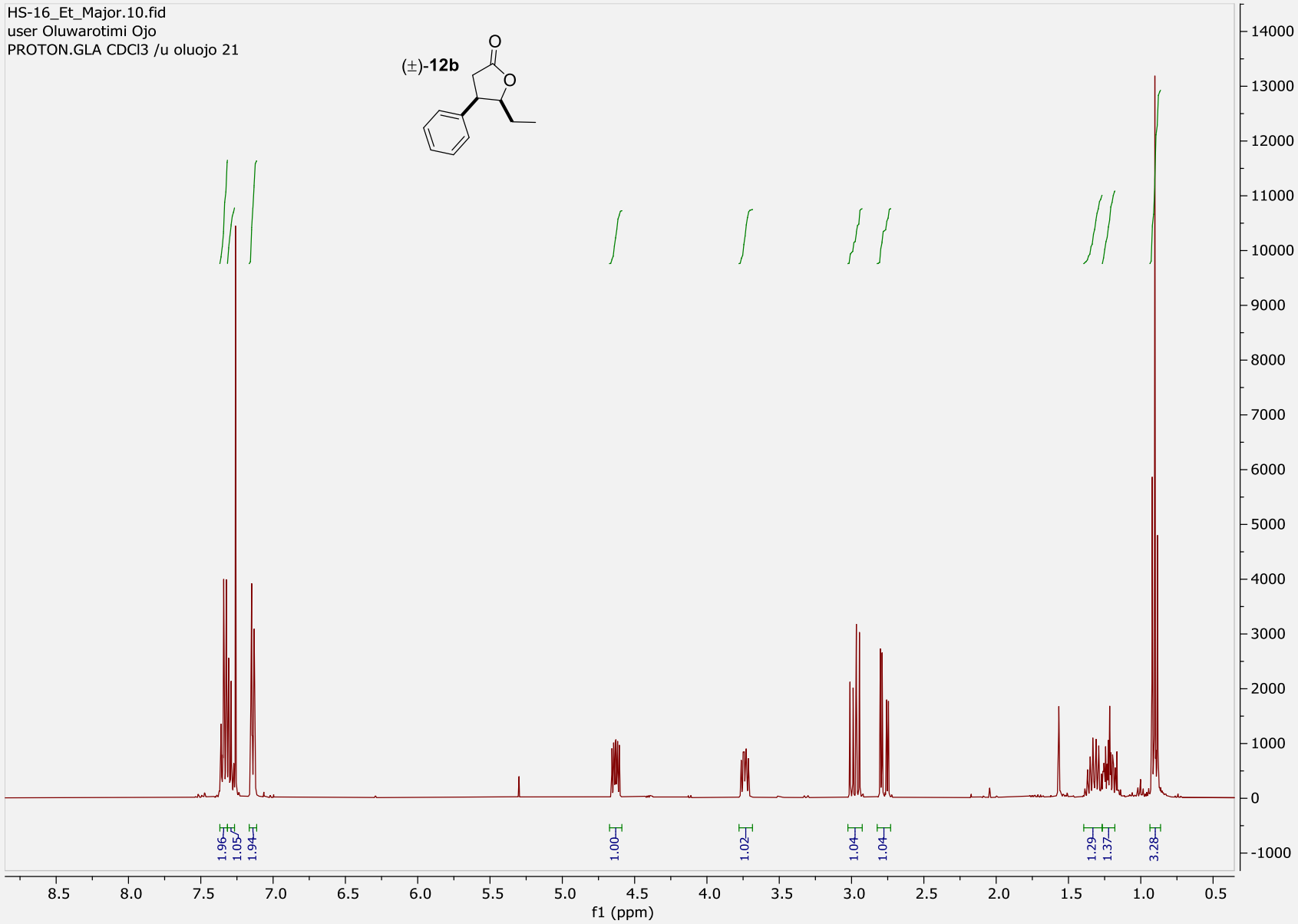
NMR Spectra

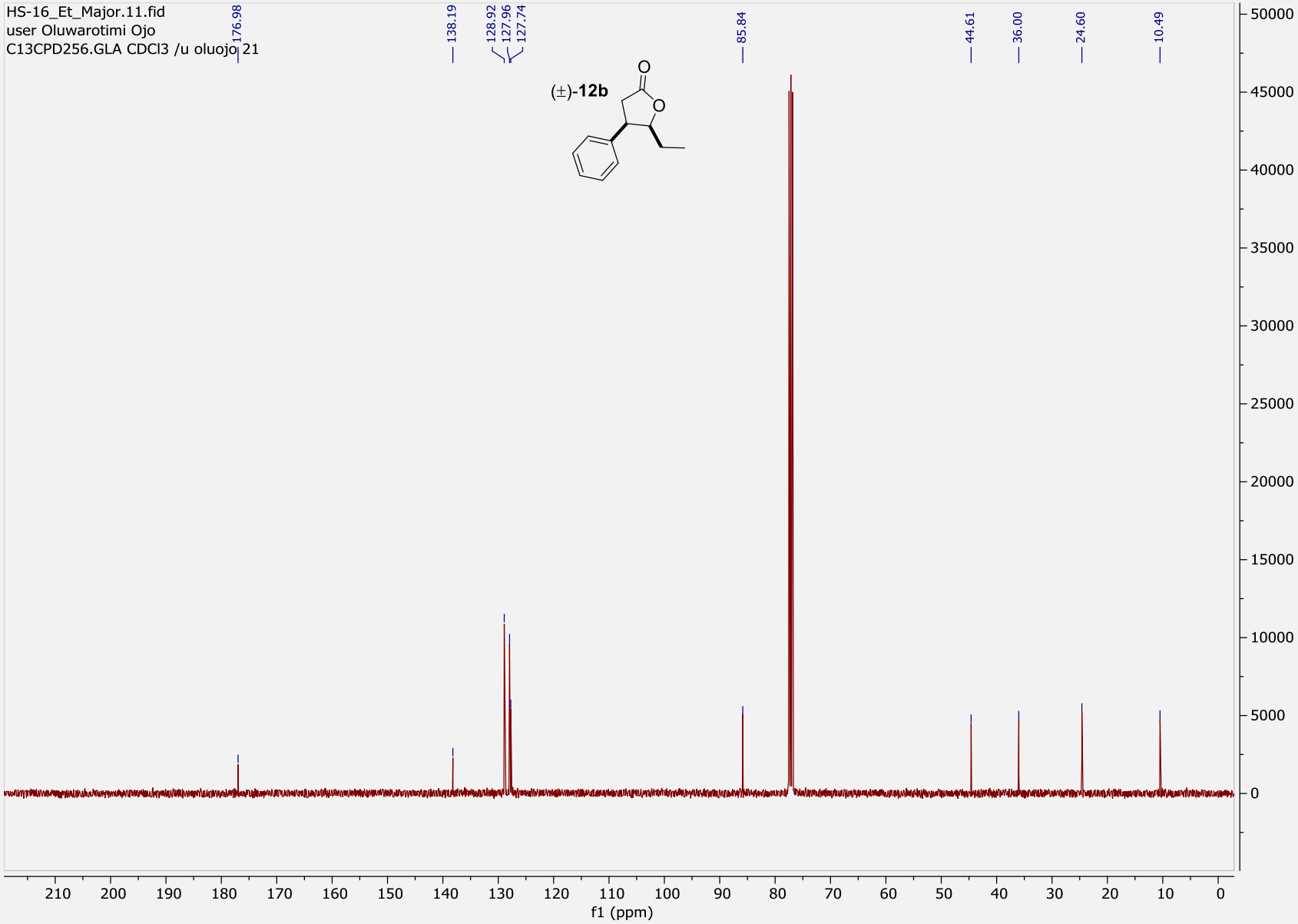
of

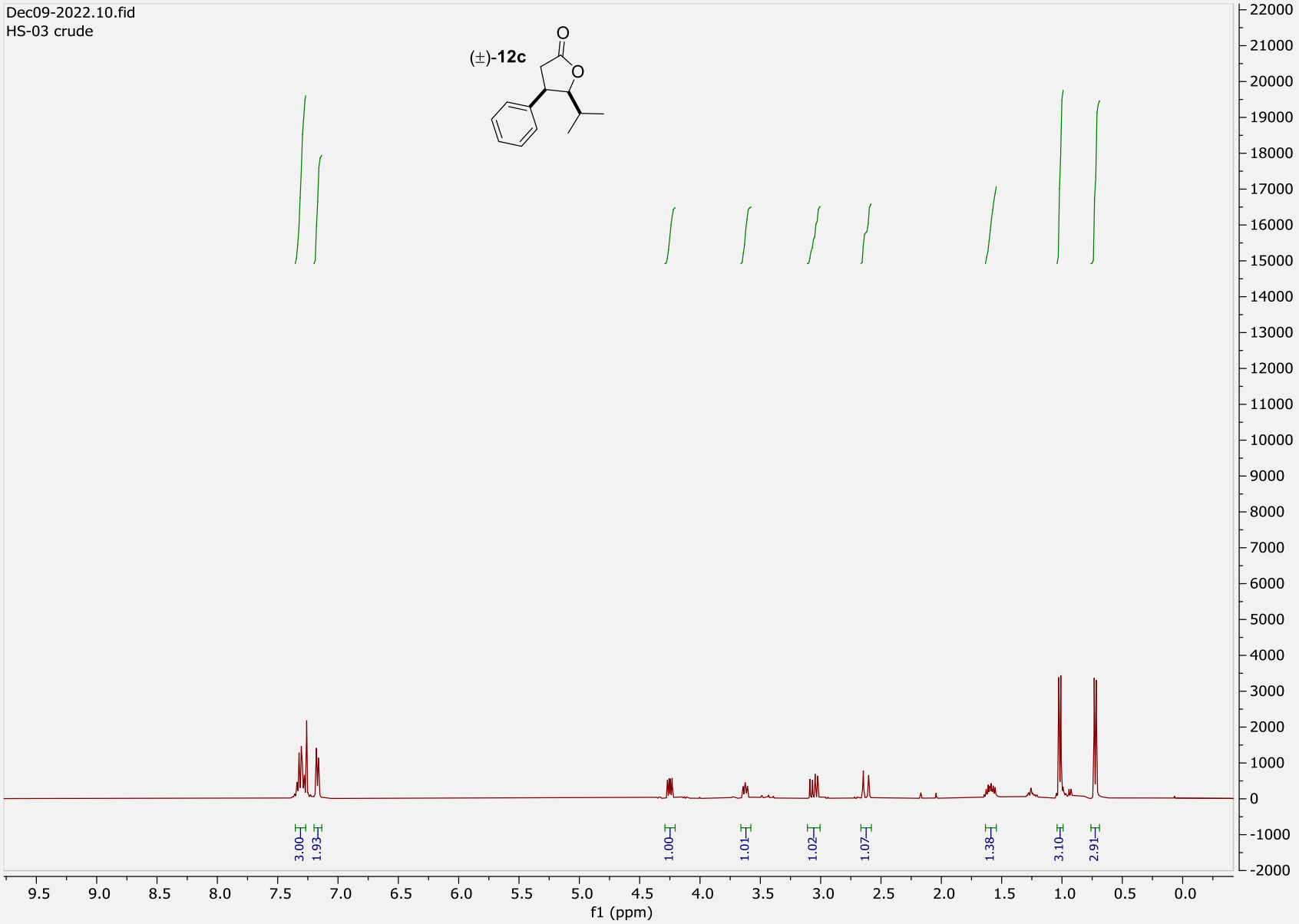
Purified γ-Butyrolactones

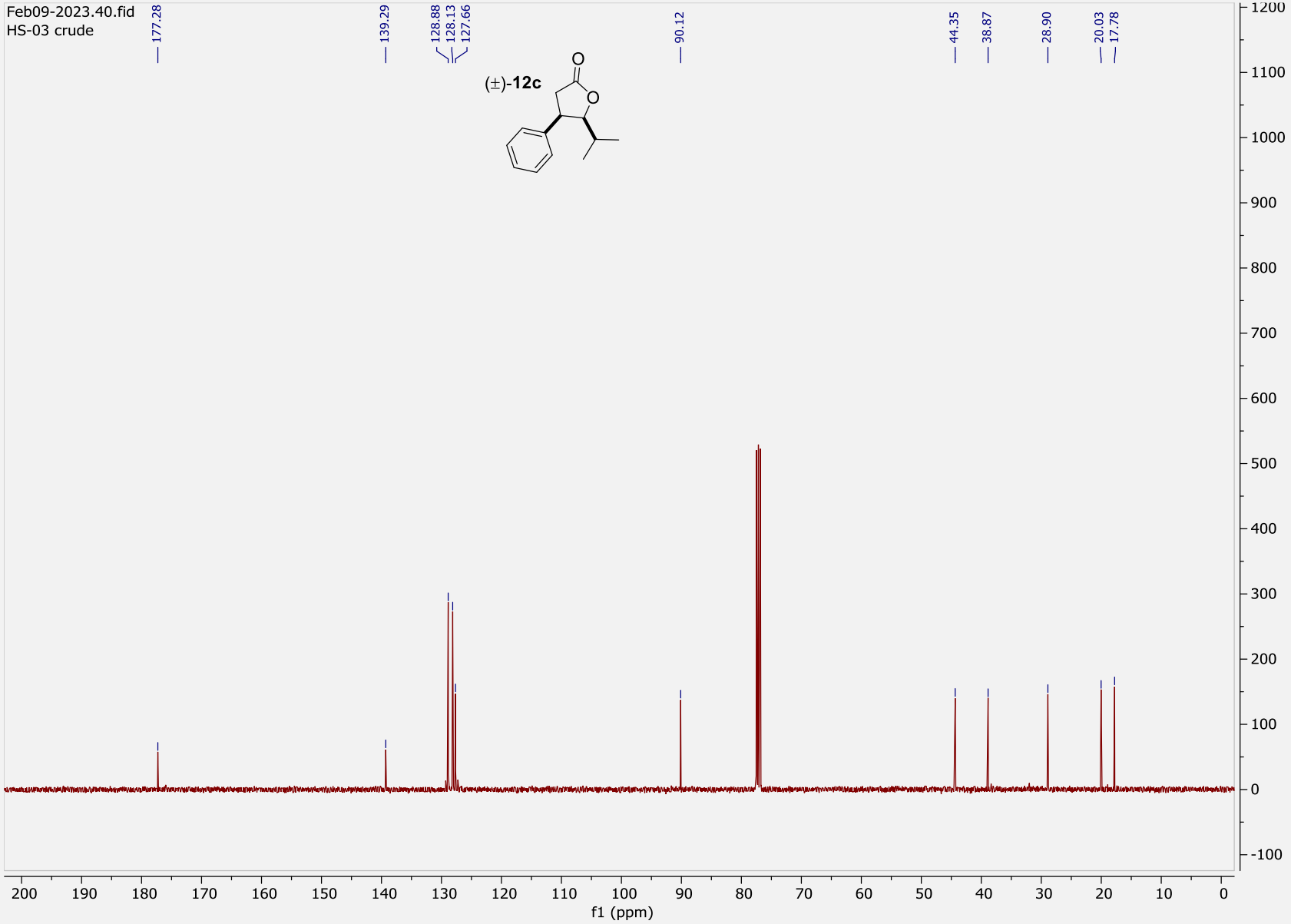

HS-01_Me_Major.10.fid
user Oluwarotimi Ojo
PROTON.GLA CDCl3 /u oluojo 20
(±)-12a
O
O
Me
25000
20000
15000
10000
5000
0
1.97
1.10
1.94
1.00
1.01
1.04
1.05
2.98
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

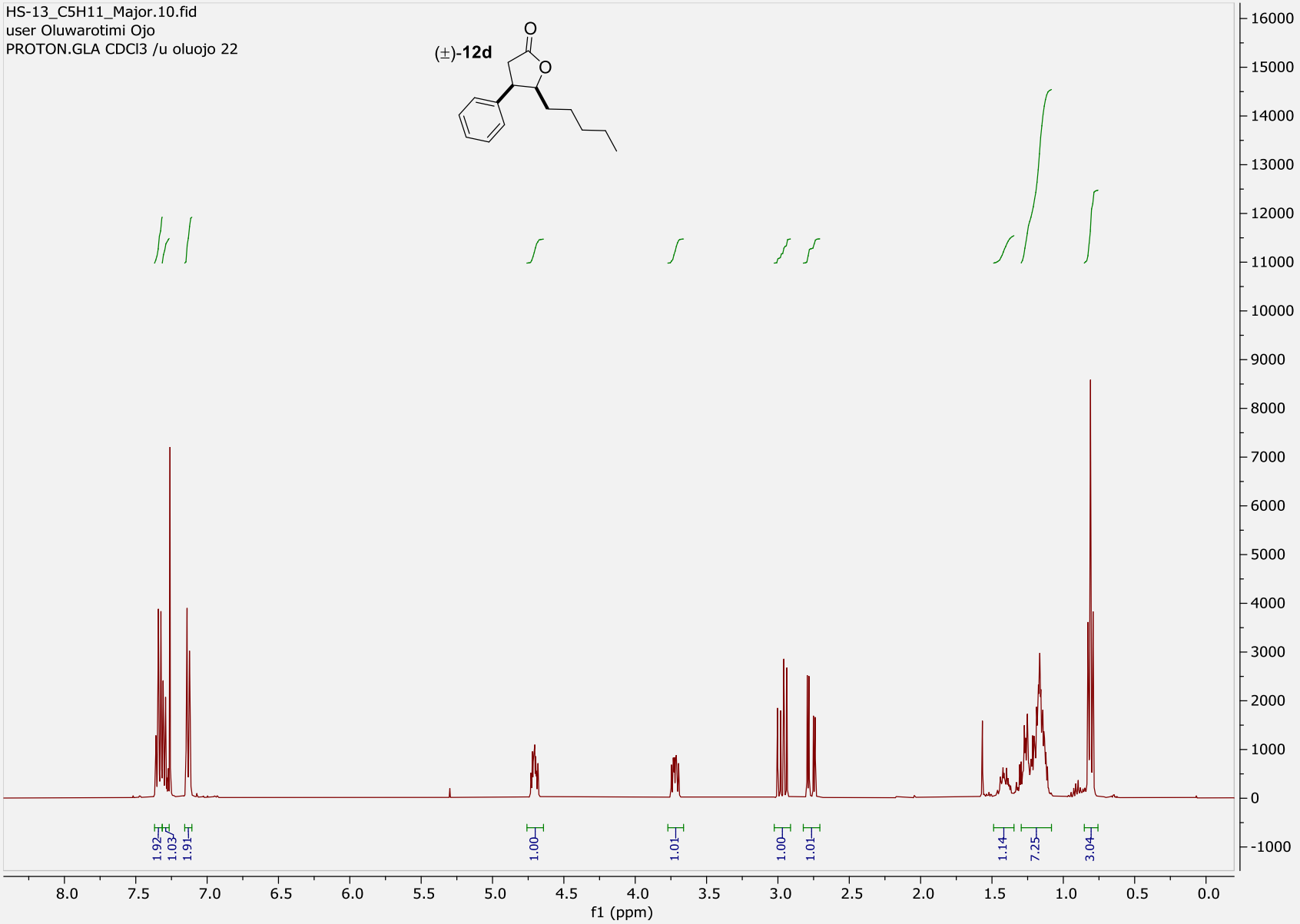

HS-01_Me_Major.11.fid
user Oluwarotimi Ojo
C13CPD256.GLA CDCl3 /u oluojo 20
176.78
137.76
128.97
127.88
127.78
80.16
44.96
35.00
16.82
(±)-12a
O
O
Me
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

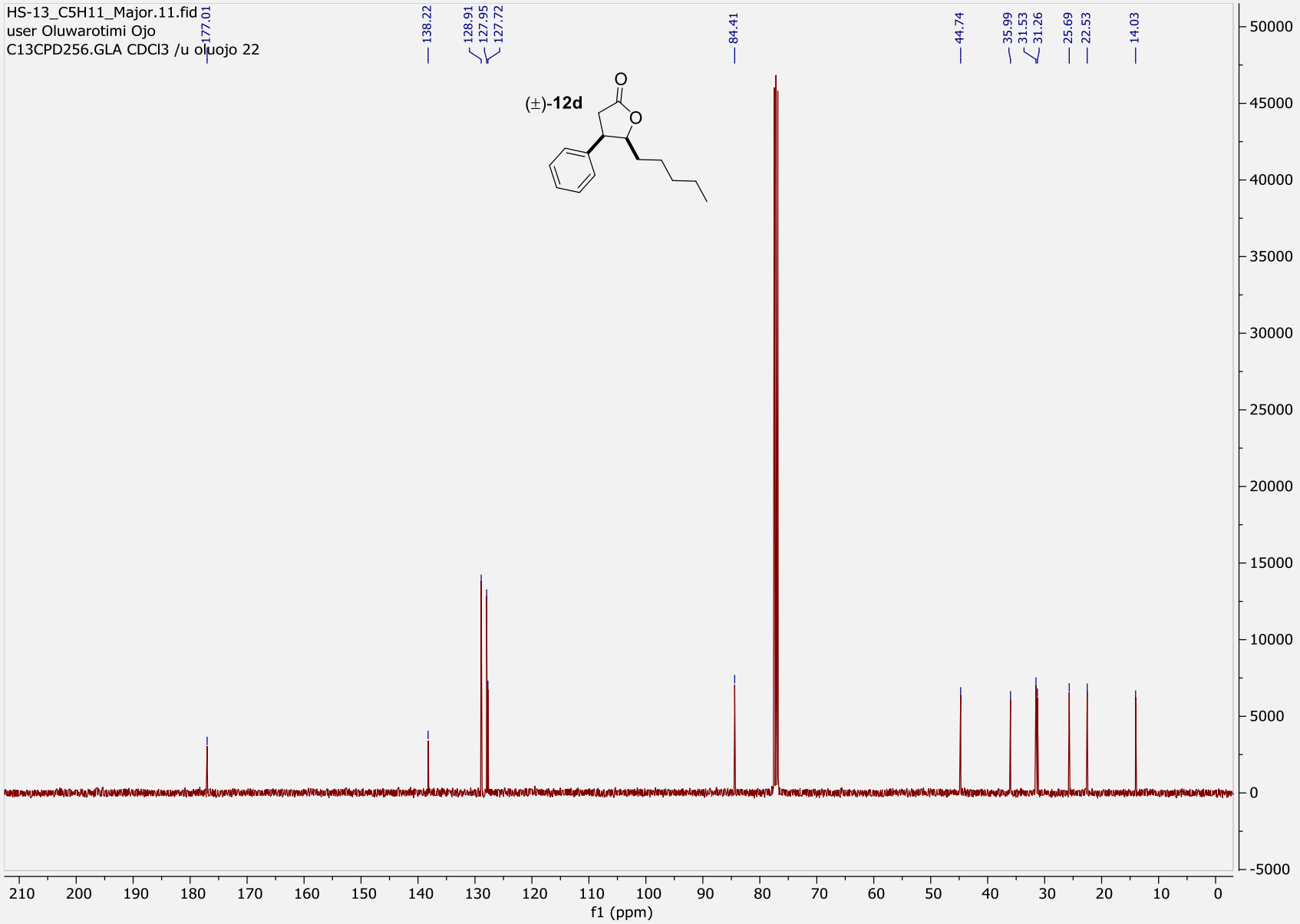

HS-16_Et_Major.10.fid
user Oluwarotimi Ojo
PROTON.GLA CDCl3 /u oluojo 21
(±)-12b
O
O
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
1.96
1.05
1.94
1.00
1.02
1.04
1.04
1.29
1.37
3.28
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

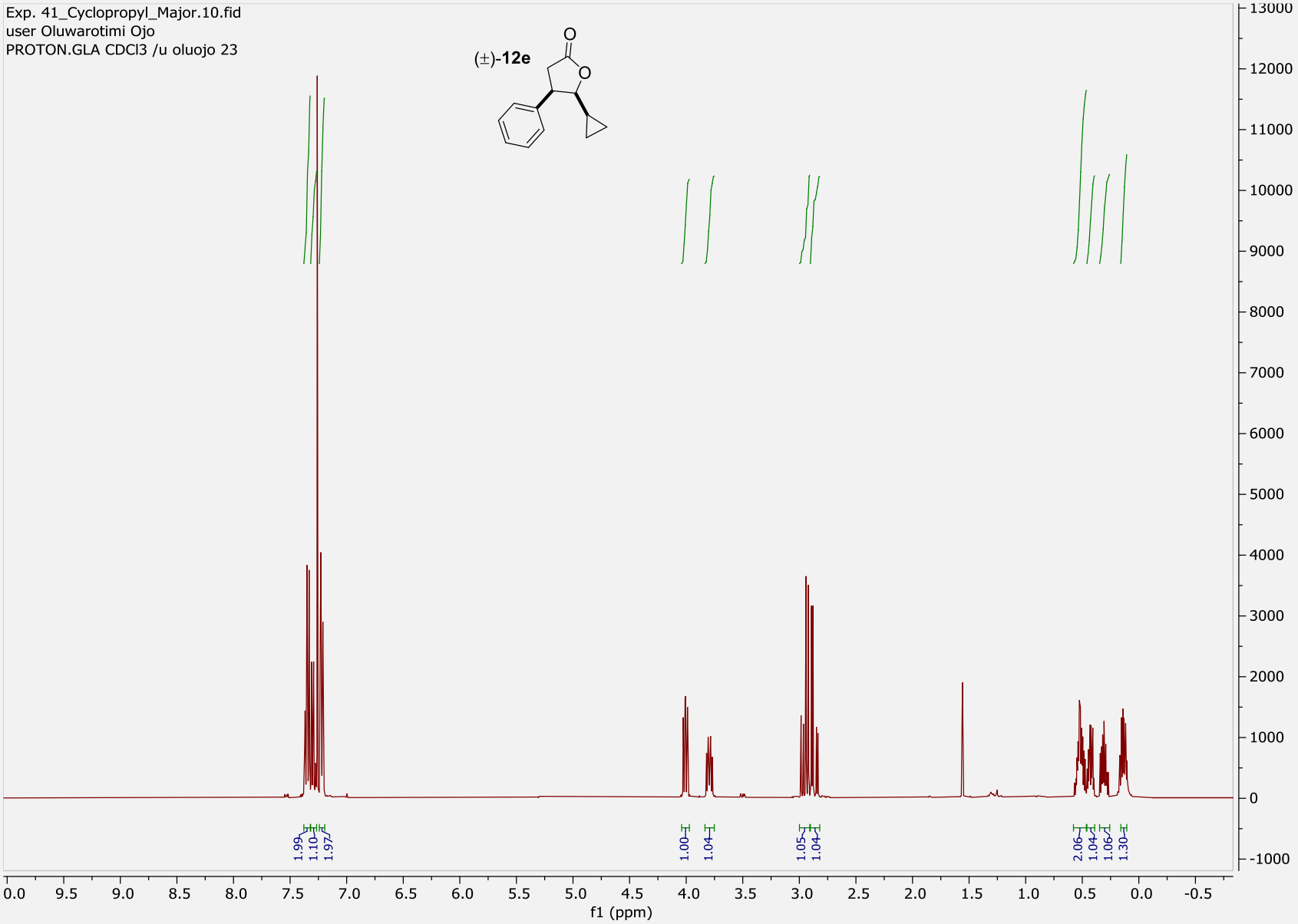

HS-16_Et_Major.11.fid
user Oluwarotimi Ojo
C13CPD256.GLA CDCl3 /u oluojo 21
176.98
138.19
128.92
127.96
127.74
85.84
44.61
36.00
24.60
10.49
(±)-12b
O
O
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

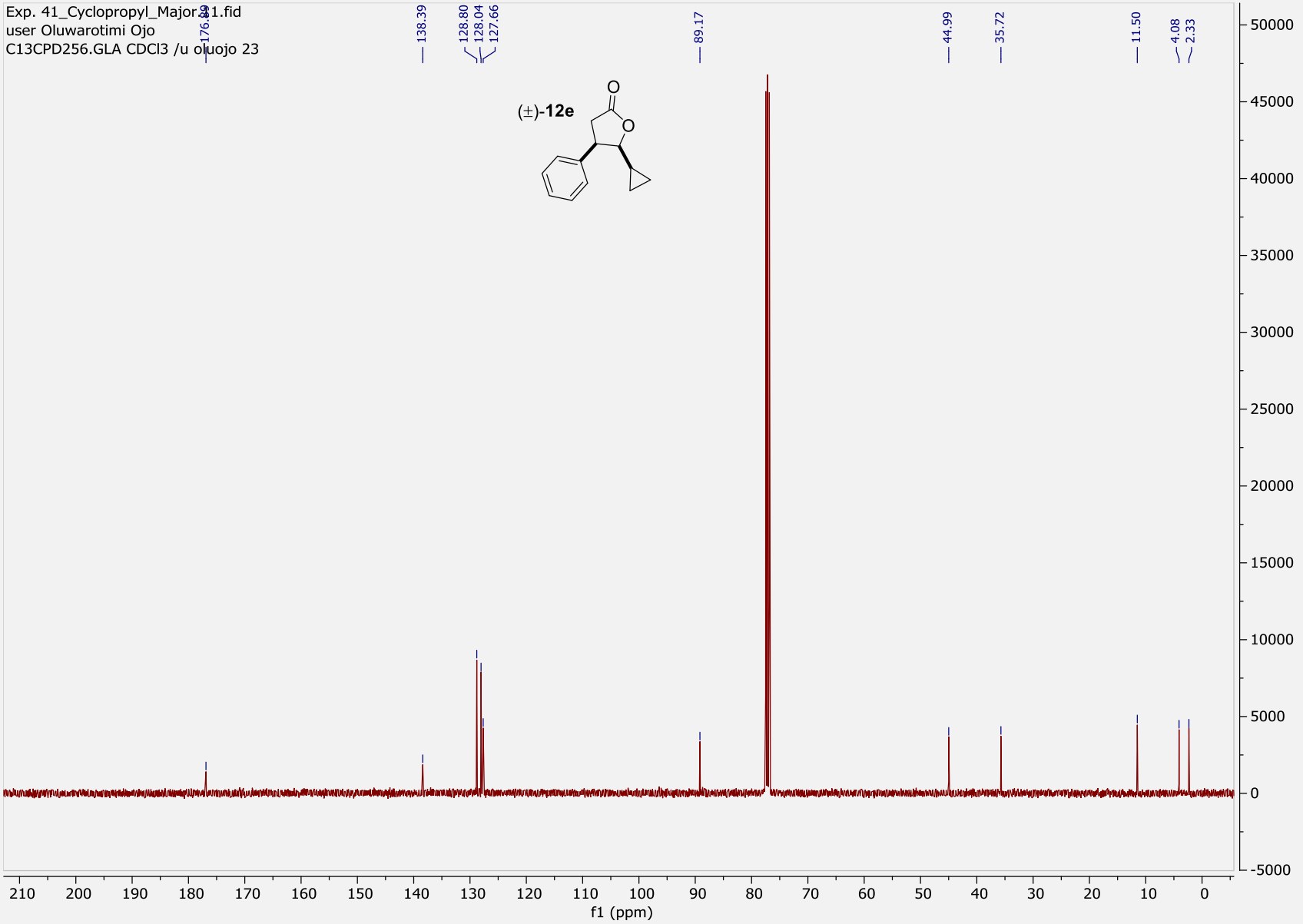

Dec09-2022.10.fid
HS-03 crude
(±)-12c
O
O
3.00
1.93
1.00
1.01
1.02
1.07
1.38
3.10
2.91
f1 (ppm)

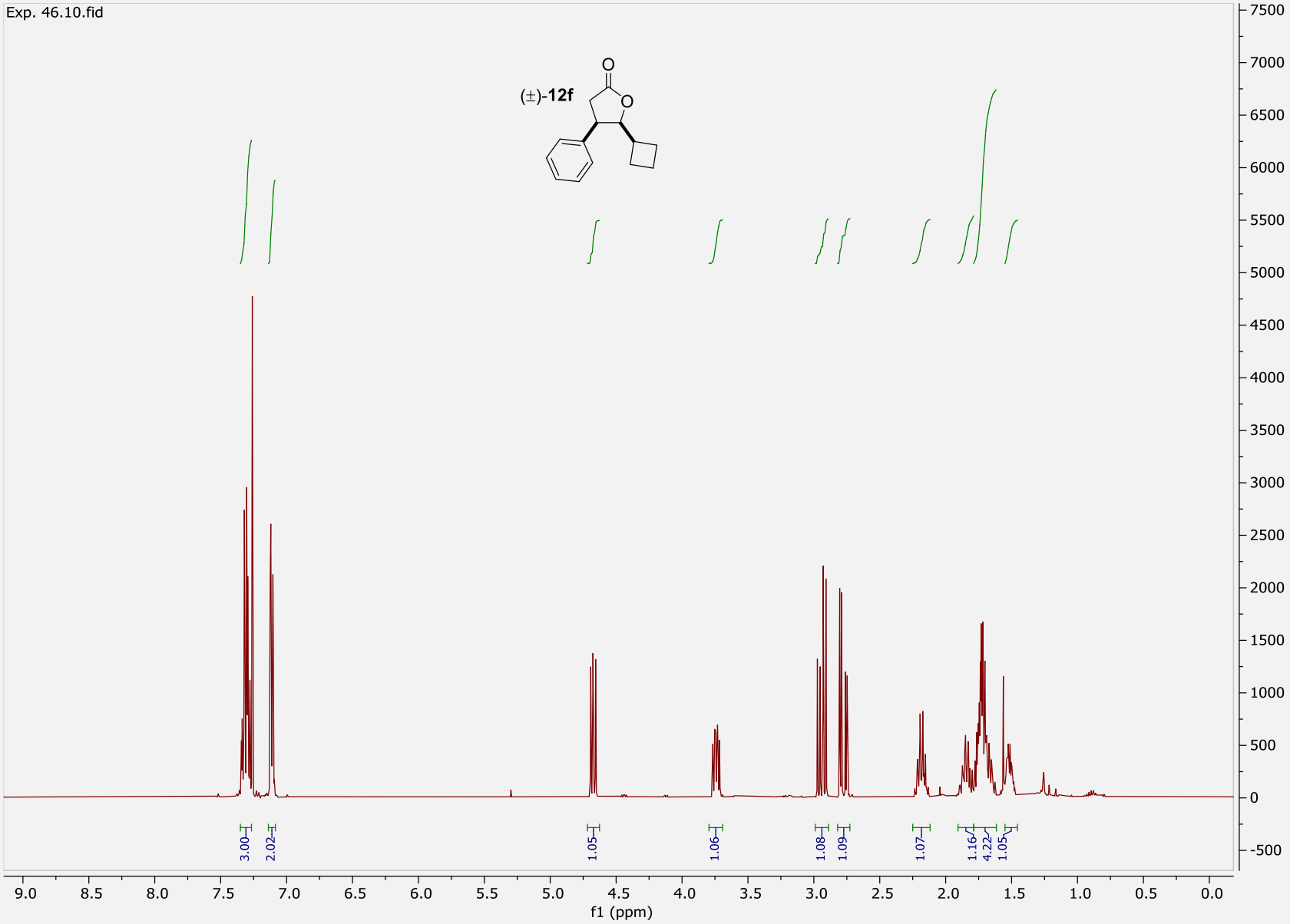

Feb09-2023.40.fid
HS-03 crude
177.28
139.29
128.88
128.13
127.66
90.12
44.35
38.87
28.90
20.03
17.78
(±)-12c
O
O
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

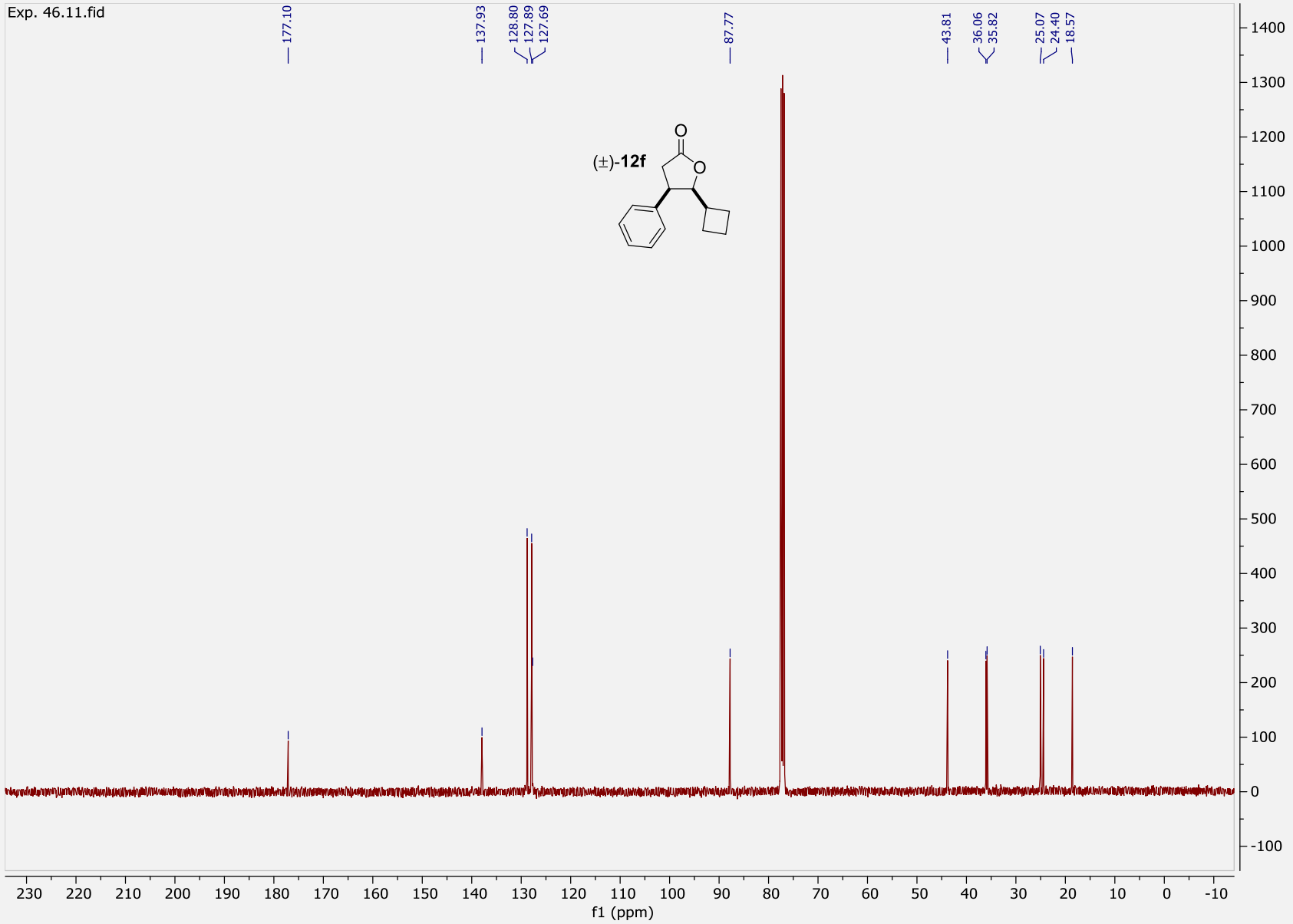

HS-13_C5H11_Major.10.fid
user Oluwarotimi Ojo
PROTON.GLA CDCl3 /u oluojo 22
(±)-12d
O
O
1.92
1.03
1.91
1.00
1.01
1.00
1.01
1.14
7.25
3.04
f1 (ppm)

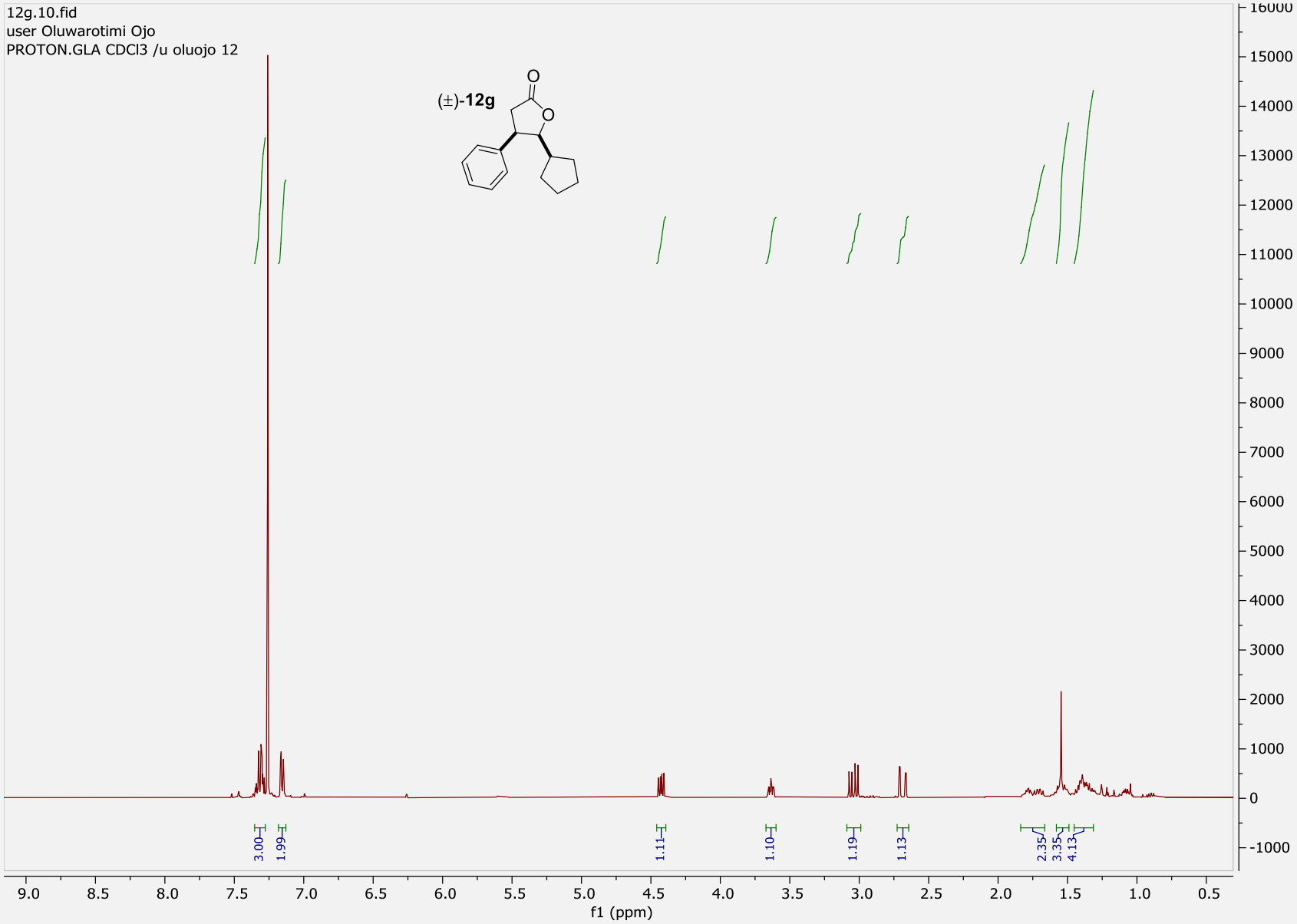

HS-13_C5H11_Major.11.fid
user Oluwarotimi Ojo
C13CPD256.GLA CDCl3 /u oluojo 22
177.01
138.22
128.91
127.95
127.72
84.41
44.74
35.99
31.53
31.26
25.69
22.53
14.03
(±)-12d
O
O
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

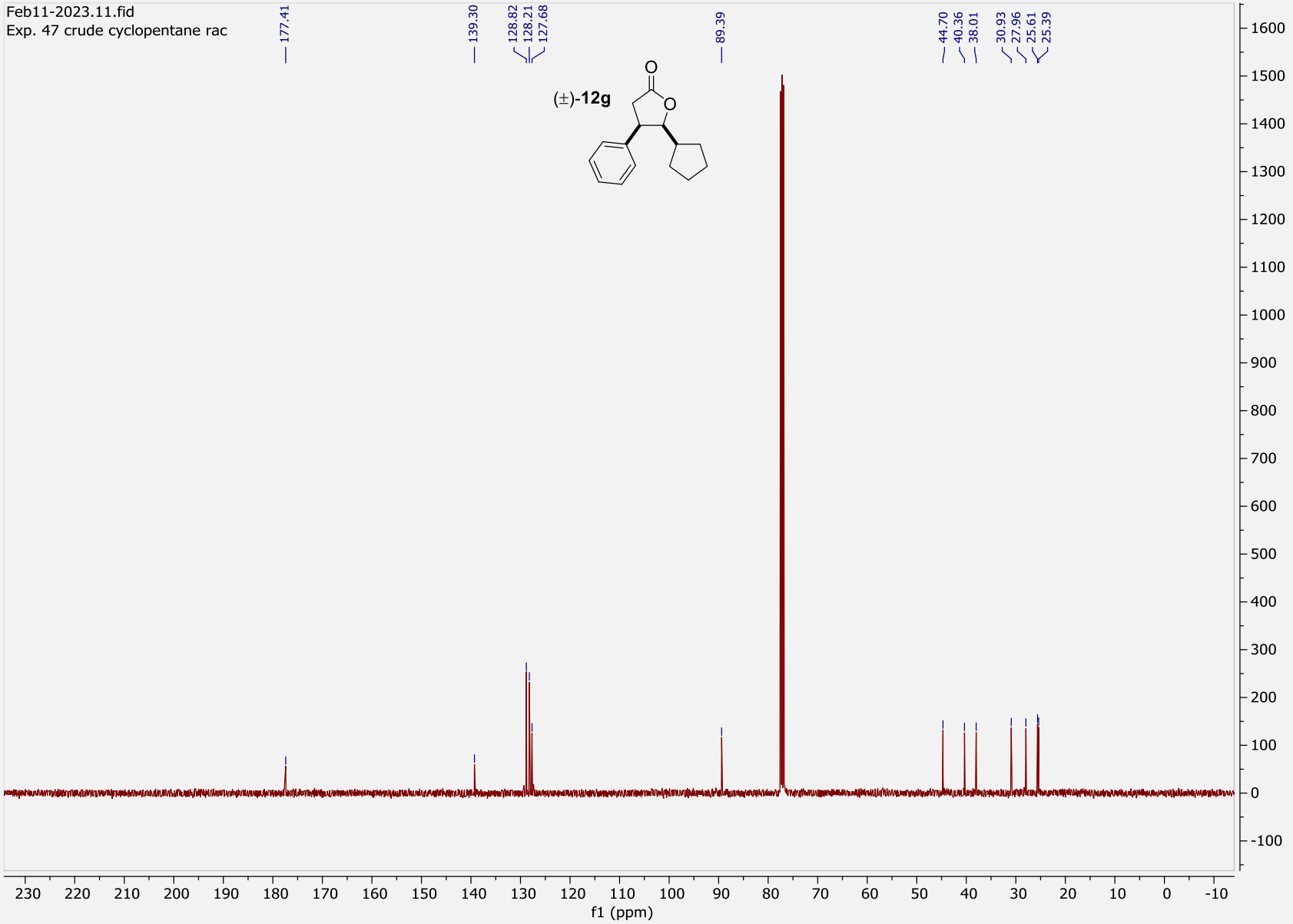

Exp. 41_Cyclopropyl_Major.10.fid
user Oluwarotimi Ojo
PROTON.GLA CDCl3 /u oluojo 23
(±)-12e
O
O
1.99
1.10
1.97
1.00
1.04
1.05
1.04
2.06
1.04
1.06
1.30
f1 (ppm)

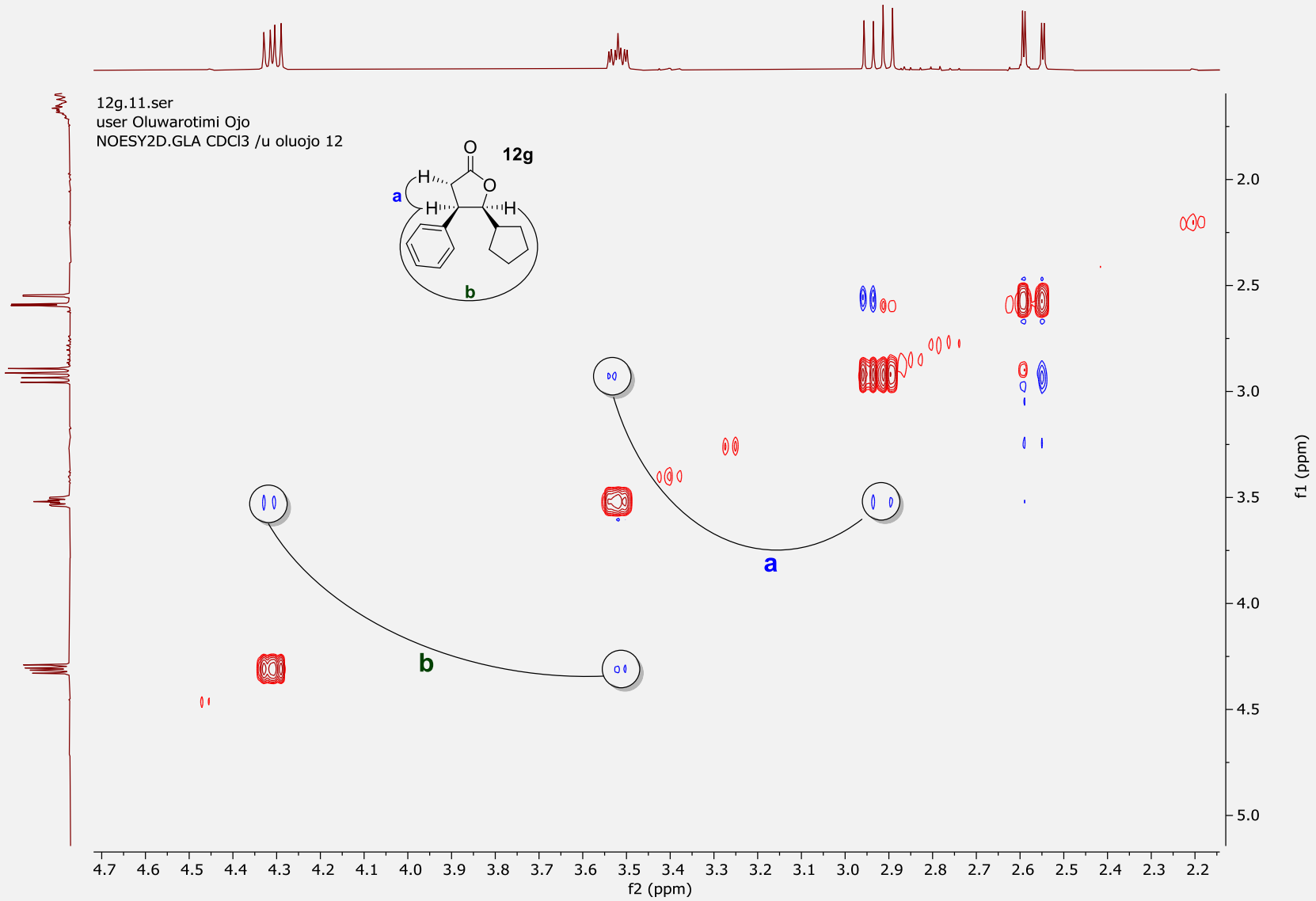

Exp. 41_Cyclopropyl_Major.11.fid
user Oluwarotimi Ojo
C13CPD256.GLA CDCl3 /u oluojo 23
176.49
138.39
128.80
128.04
127.66
89.17
44.99
35.72
11.50
4.08
2.33
(±)-12e
O
O
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

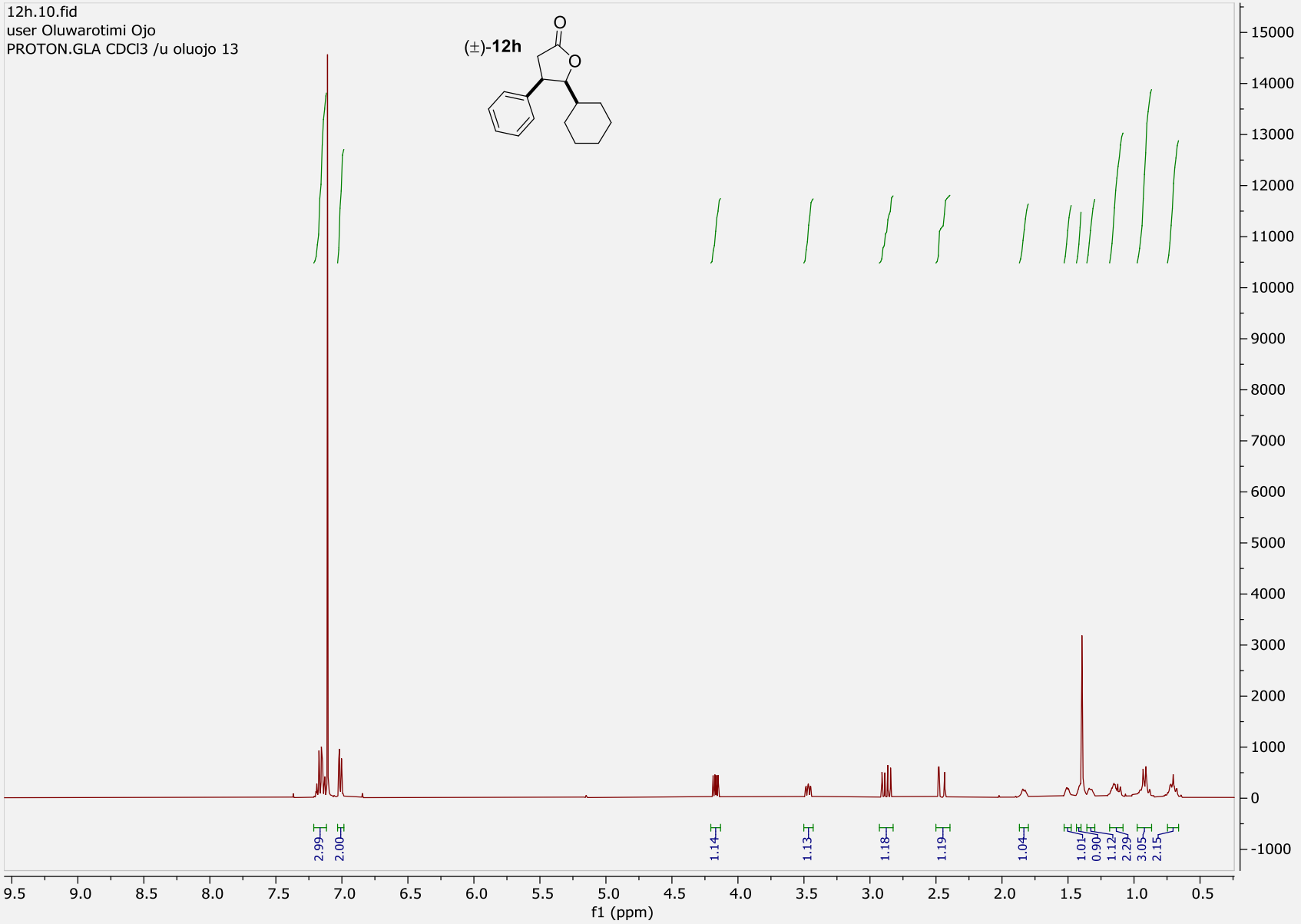

Exp. 46.10.fid
(±)-12f
O
O
3.00
2.02
1.05
1.06
1.08
1.09
1.07
1.16
4.22
1.05
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500

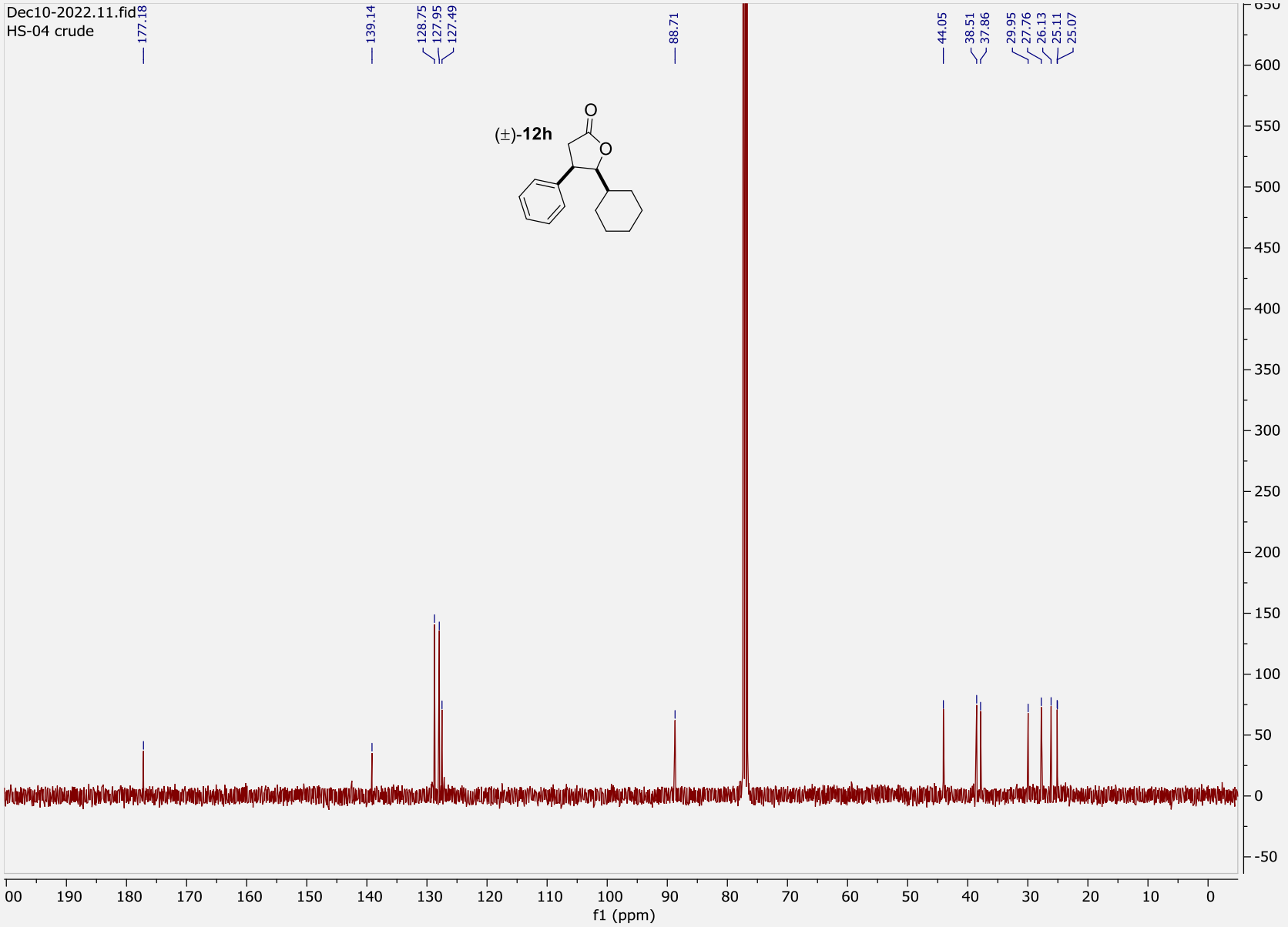

Exp. 46.11.fid
177.10
137.93
128.80
127.89
127.69
87.77
43.81
36.06
35.82
25.07
24.40
18.57
(±)-12f
O
O
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

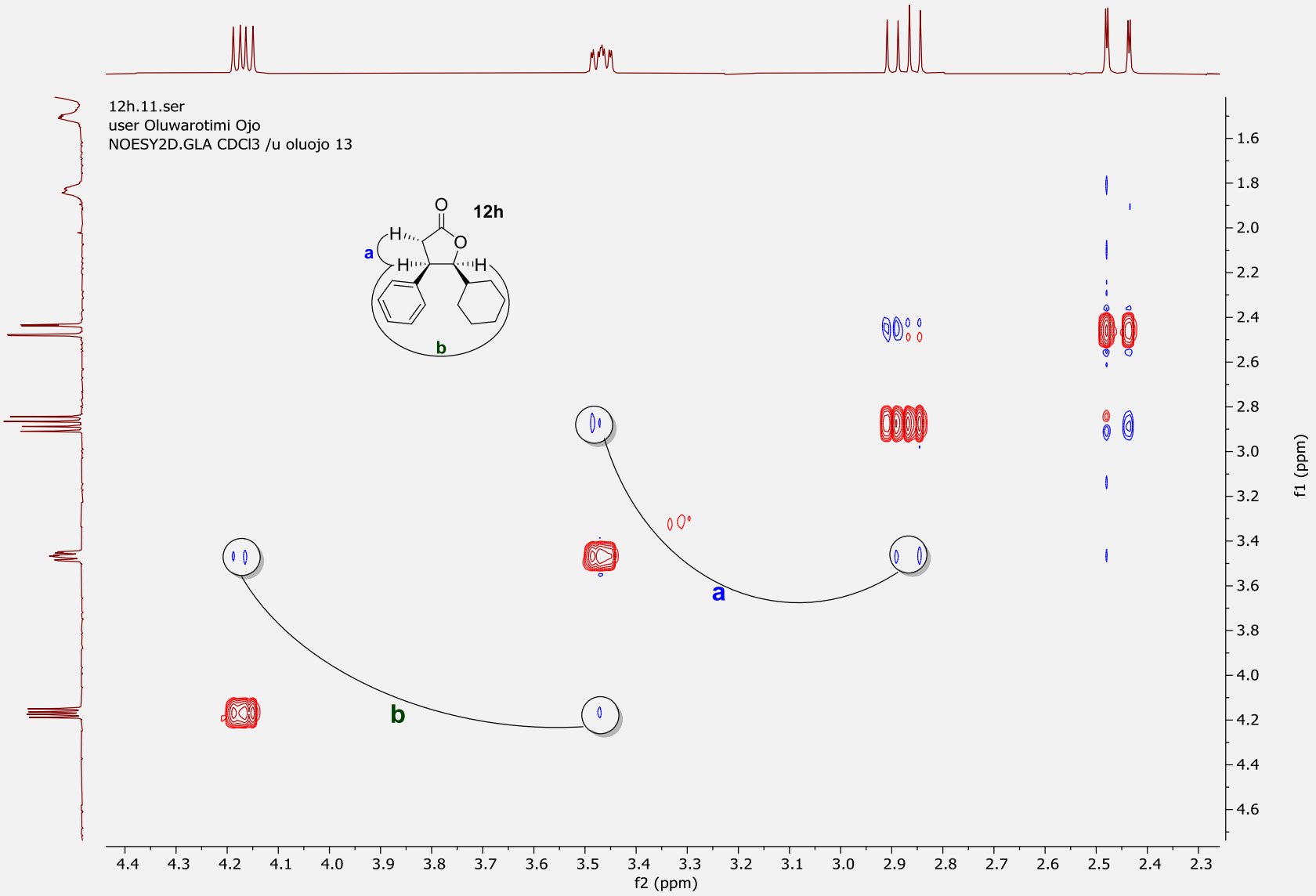

12g.10.fid
user Oluwarotimi Ojo
PROTON.GLA CDCl3 /u oluojo 12
(±)-12g
O
O
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
3.00
1.99
1.11
1.10
1.19
1.13
2.35
3.35
4.13
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

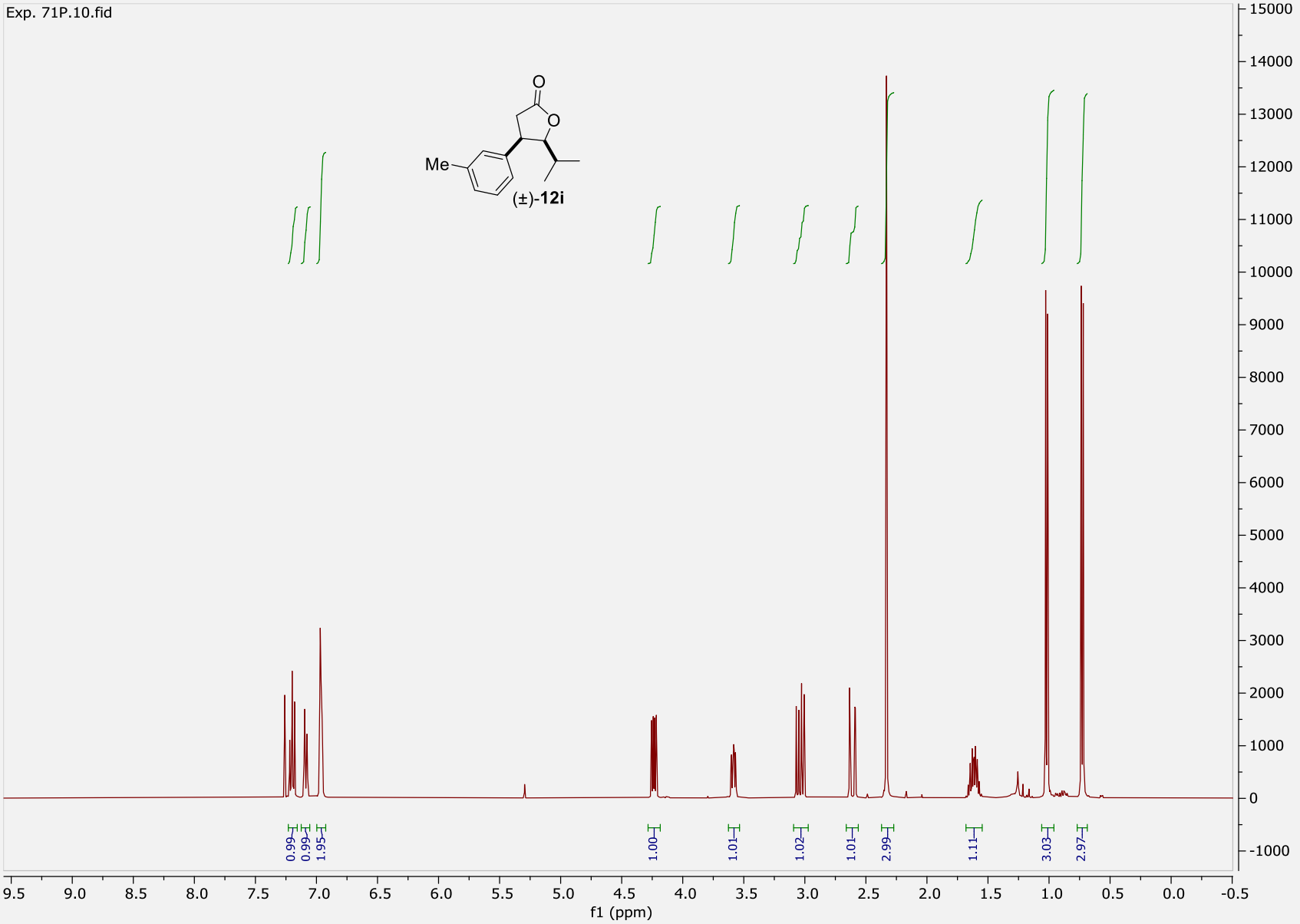

Feb11-2023.11.fid
Exp. 47 crude cyclopentane rac
177.41
139.30
128.82
128.21
127.68
89.39
44.70
40.36
38.01
30.93
27.96
25.61
25.39
(±)-12g
O
O
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

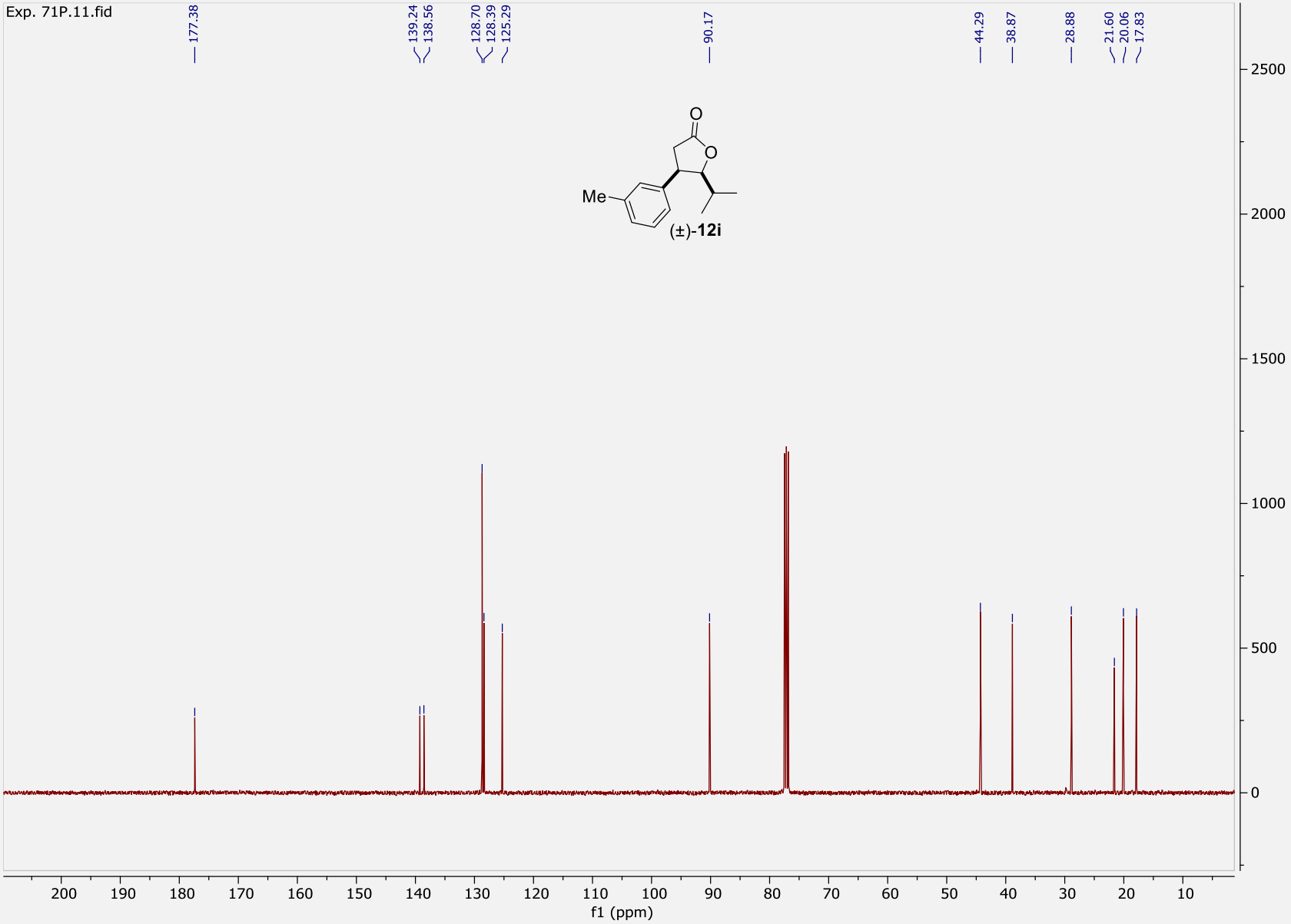

12g.11.ser
user Oluwarotimi Ojo
NOESY2D.GLA CDCl3 /u oluojo 12
12g
a
b
f2 (ppm)
f1 (ppm)

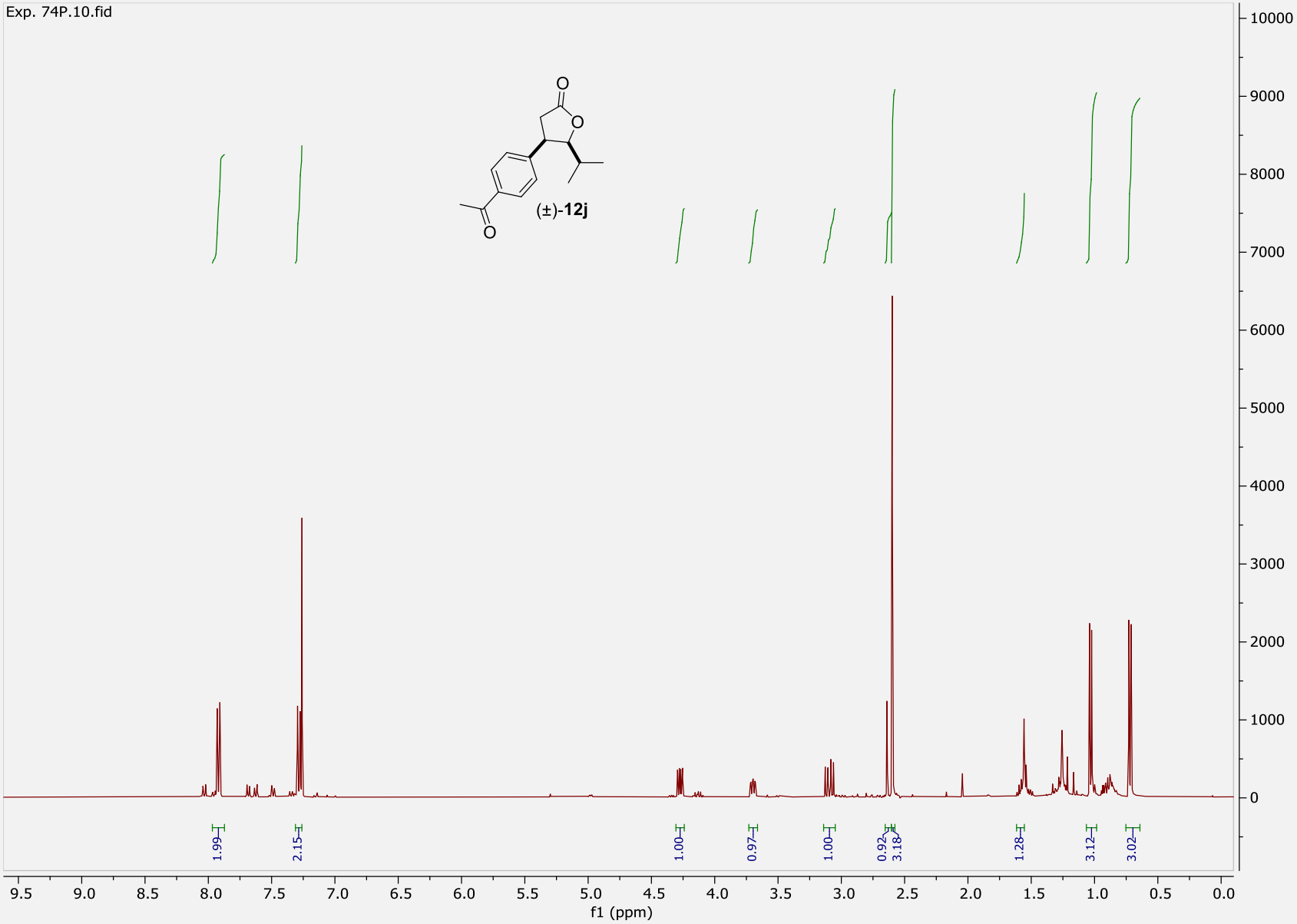

12h.10.fid
user Oluwarotimi Ojo
PROTON.GLA CDCl3 /u oluojo 13
(±)-12h
O
O
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
2.99
2.00
1.14
1.13
1.18
1.19
1.04
1.01
0.90
1.12
2.29
3.05
2.15
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

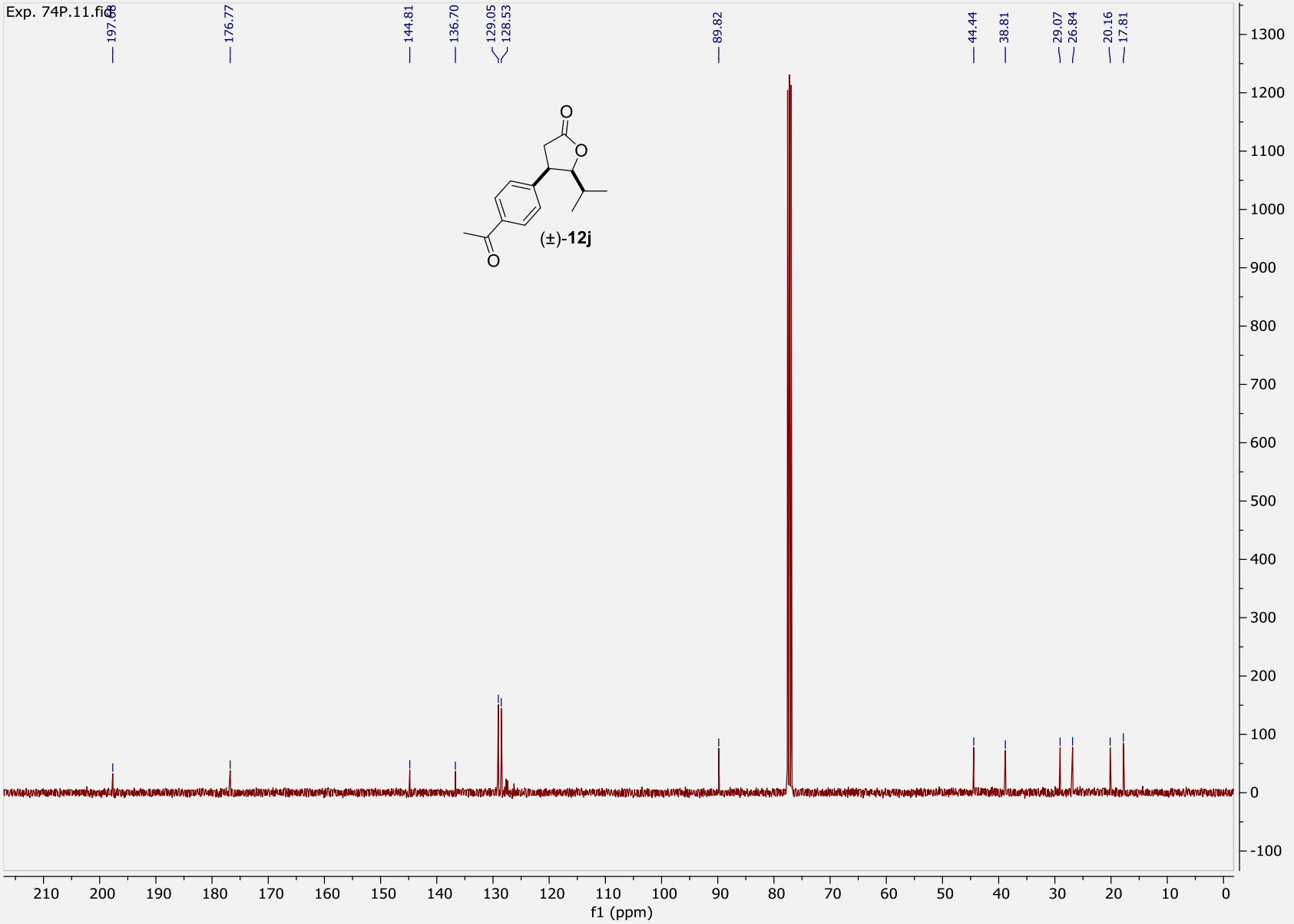

Dec10-2022.11.fid
HS-04 crude
177.18
139.14
128.75
127.95
127.49
88.71
44.05
38.51
37.86
29.95
27.76
26.13
25.11
25.07
(±)-12h
O
O
600
550
500
450
400
350
300
250
200
150
100
50
0
-50
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

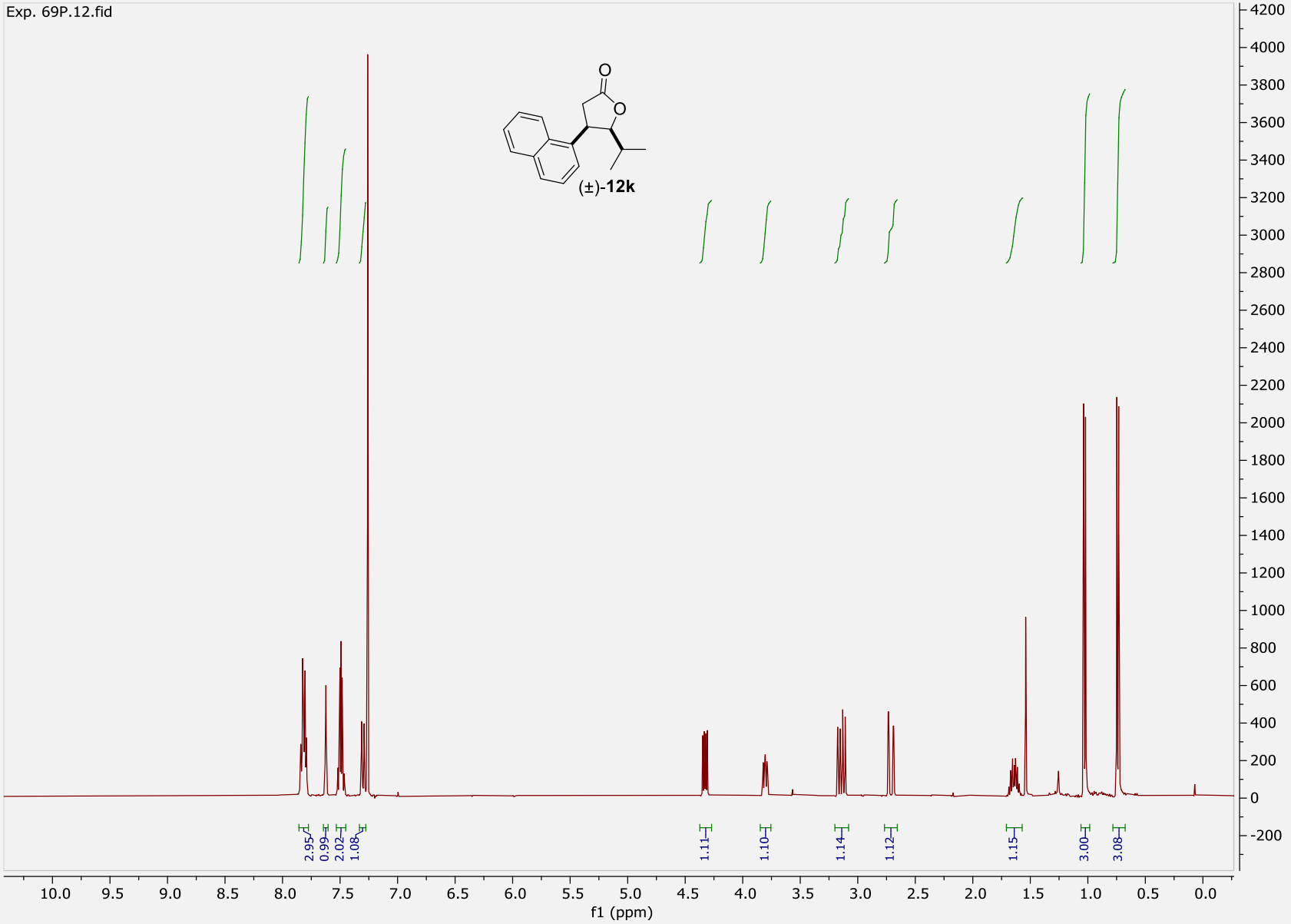

12h.11.ser
user Oluwarotimi Ojo
NOESY2D.GLA CDCl3 /u oluojo 13
12h
a
b
f2 (ppm)
f1 (ppm)

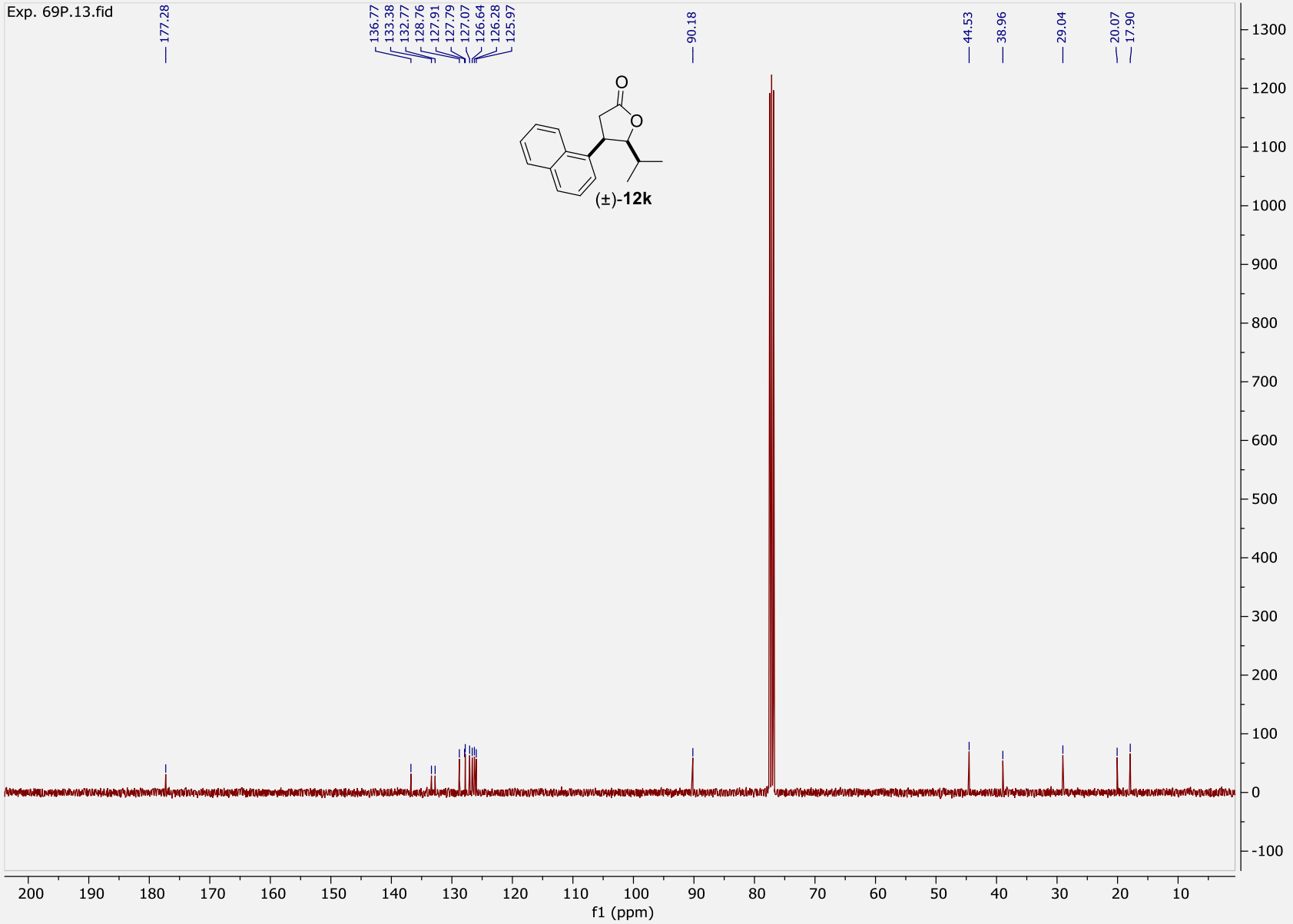

Exp. 71P.10.fid
O
O
Me
(±)-12i
0.99
0.99
1.95
1.00
1.01
1.02
1.01
2.99
1.11
3.03
2.97
f1 (ppm)

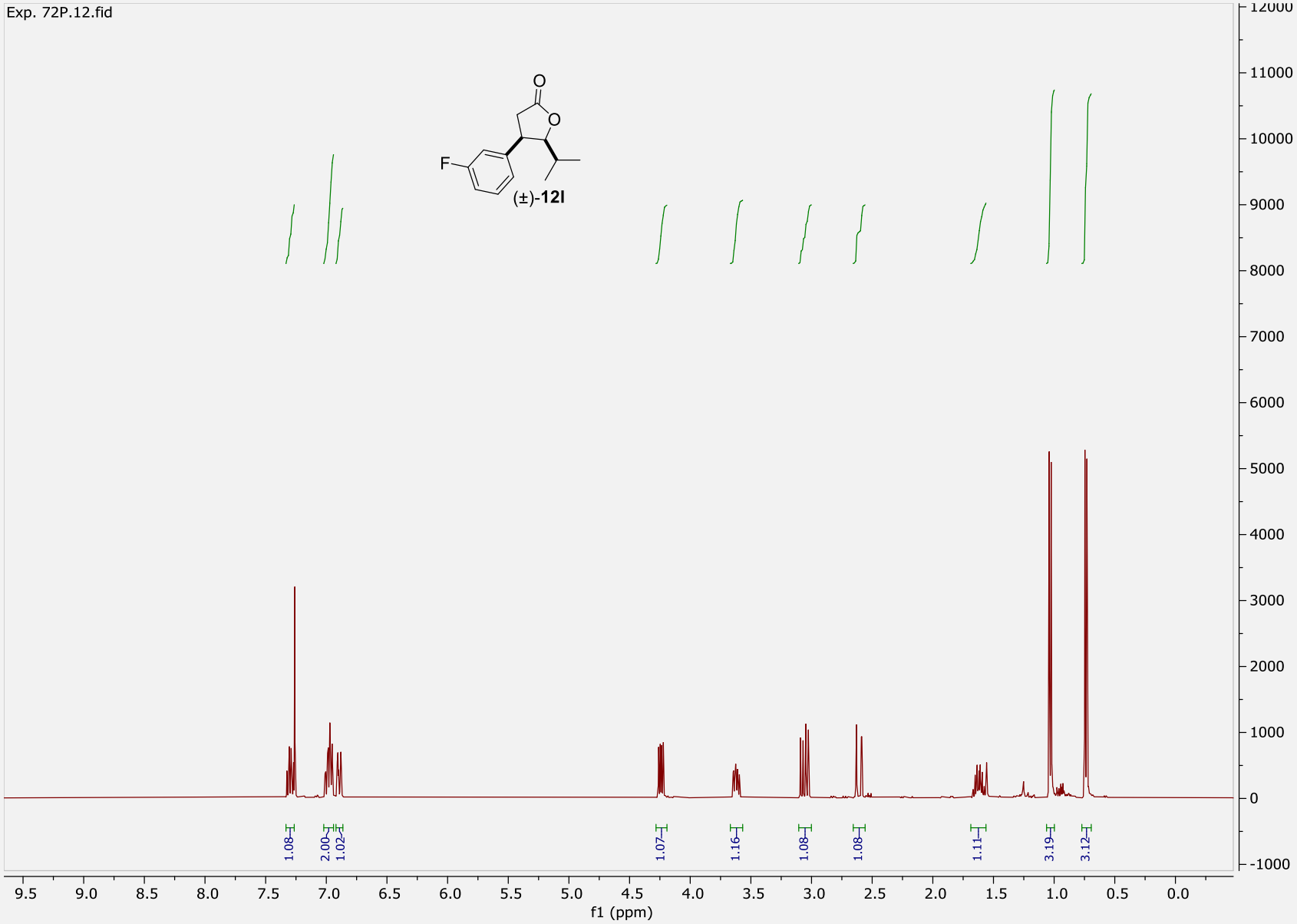

Exp. 71P.11.fid
177.38
139.24
138.56
128.70
128.39
125.29
90.17
44.29
38.87
28.88
21.60
20.06
17.83
O
O
Me
(±)-12i
2500
2000
1500
1000
500
0
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

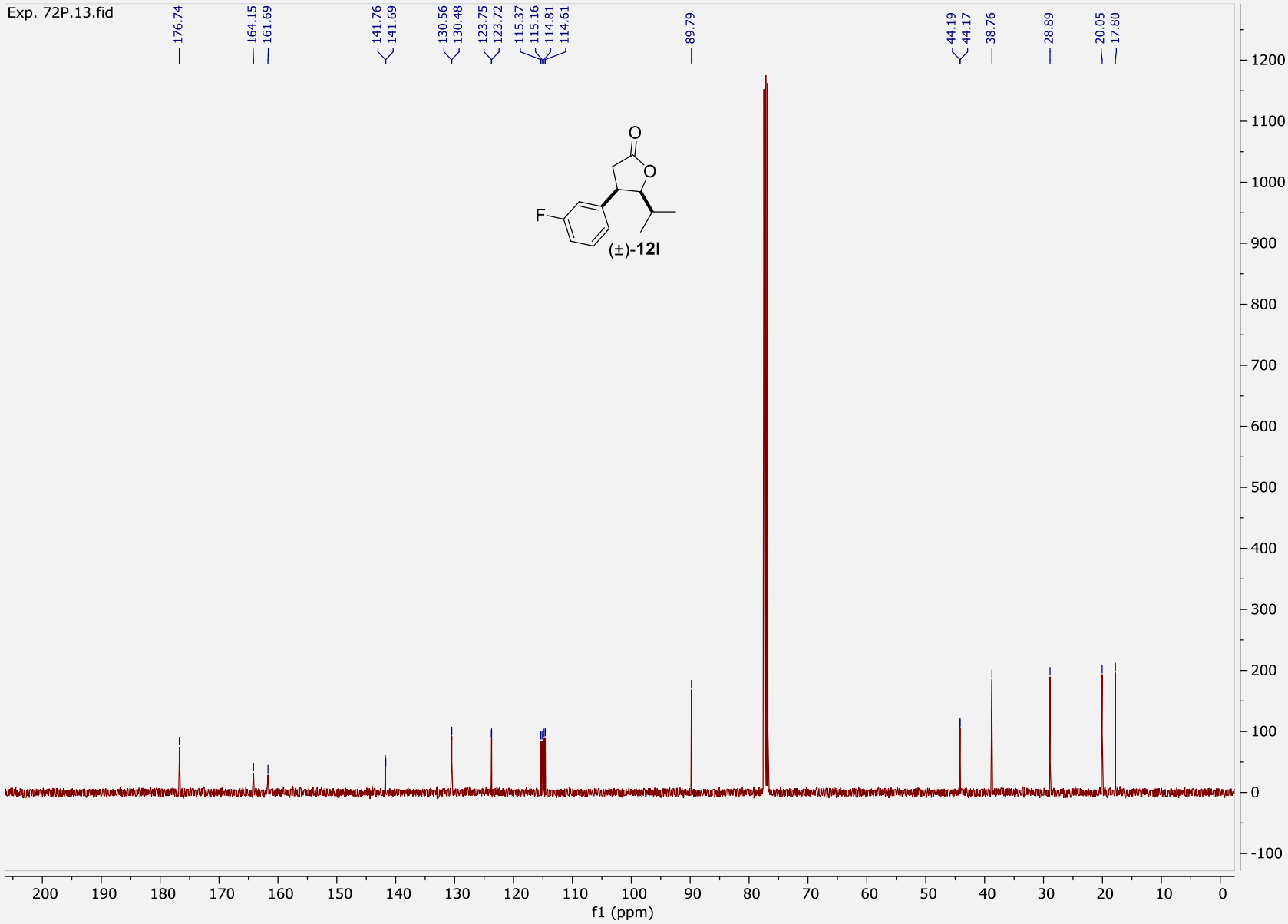

Exp. 74P.10.fid
(±)-12j
1.99
2.15
1.00
0.97
1.00
0.92
3.18
1.28
3.12
3.02
f1 (ppm)

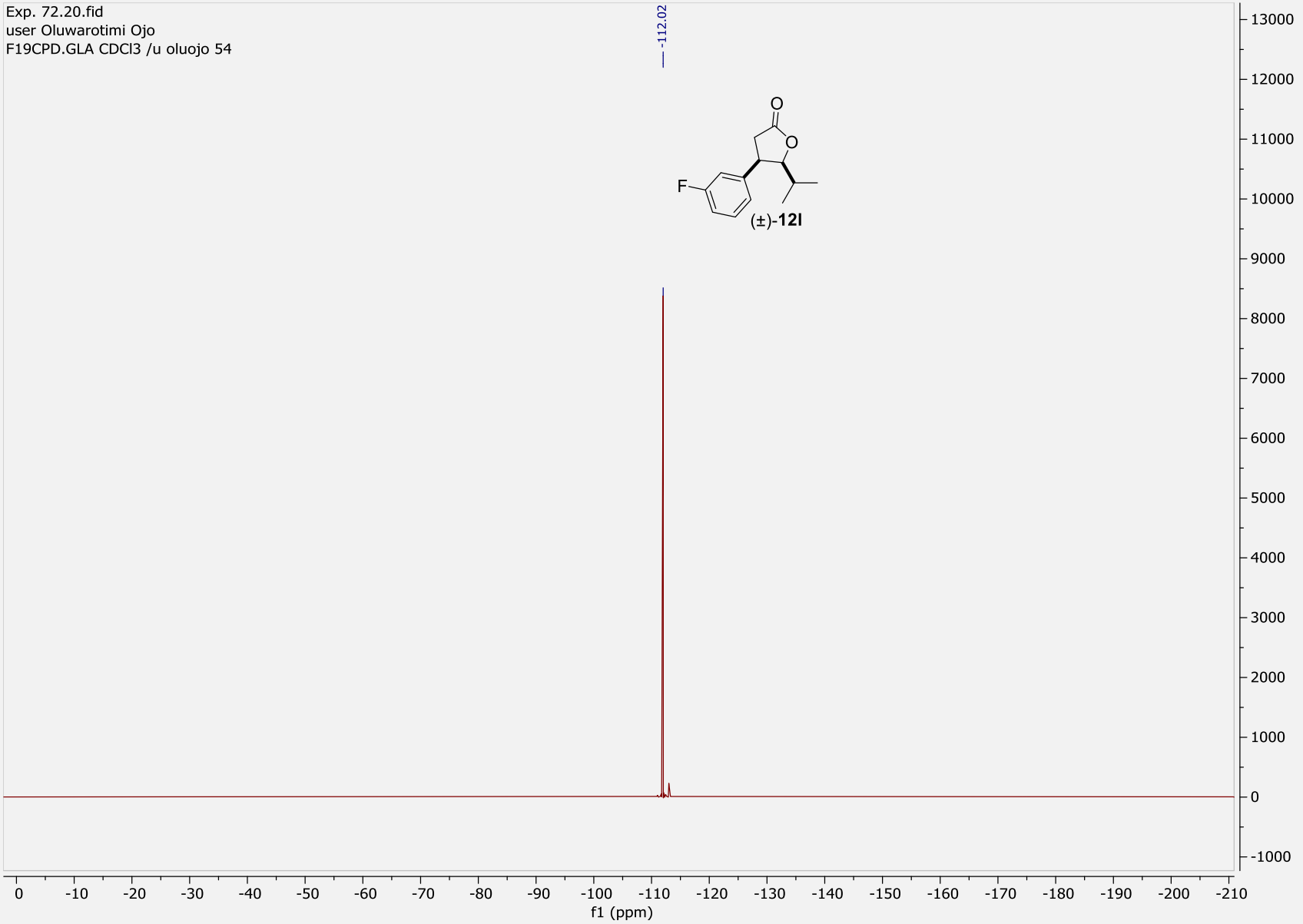

Exp. 74P.11.fid
197.88
176.77
144.81
136.70
129.05
128.53
89.82
44.44
38.81
29.07
26.84
20.16
17.81
(±)-12j
f1 (ppm)

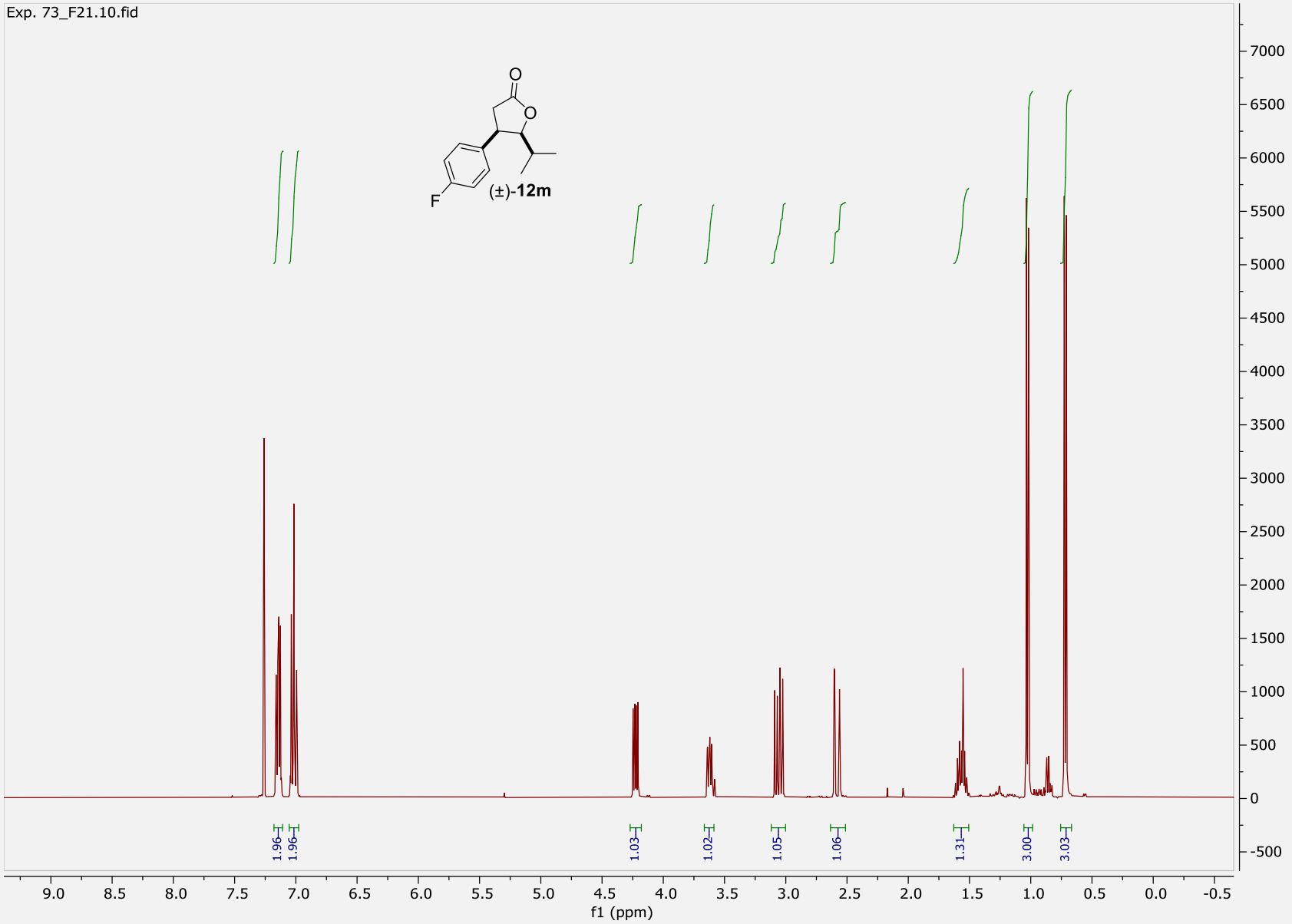

Exp. 69P.12.fid
(±)-12k
2.95
0.99
2.02
1.08
1.11
1.10
1.14
1.12
1.15
3.00
3.08
f1 (ppm)

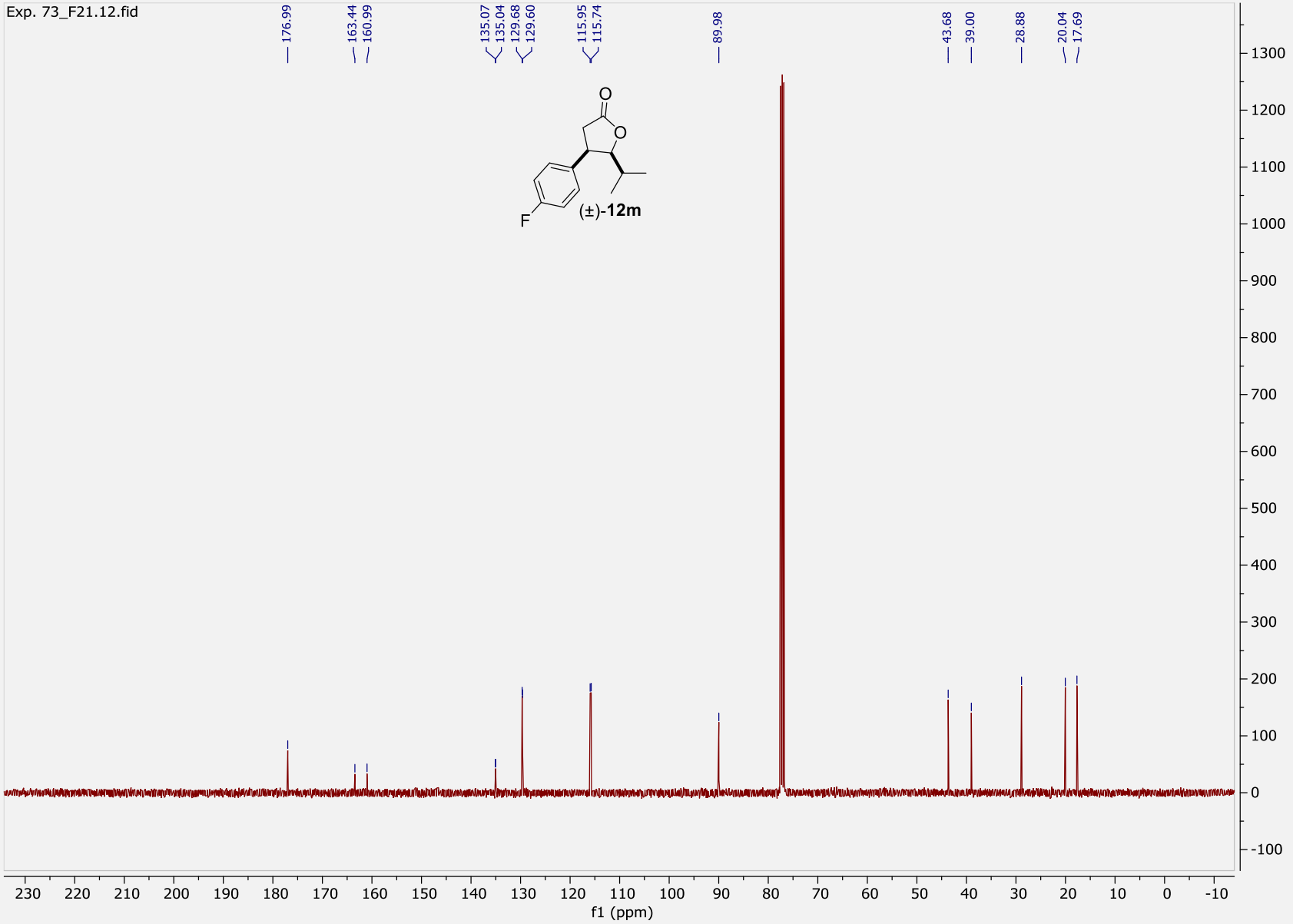

Exp. 69P.13.fid
177.28
136.77
133.38
132.77
128.76
127.91
127.79
127.07
126.64
126.28
125.97
90.18
44.53
38.96
29.04
20.07
17.90
(±)-12k
f1 (ppm)

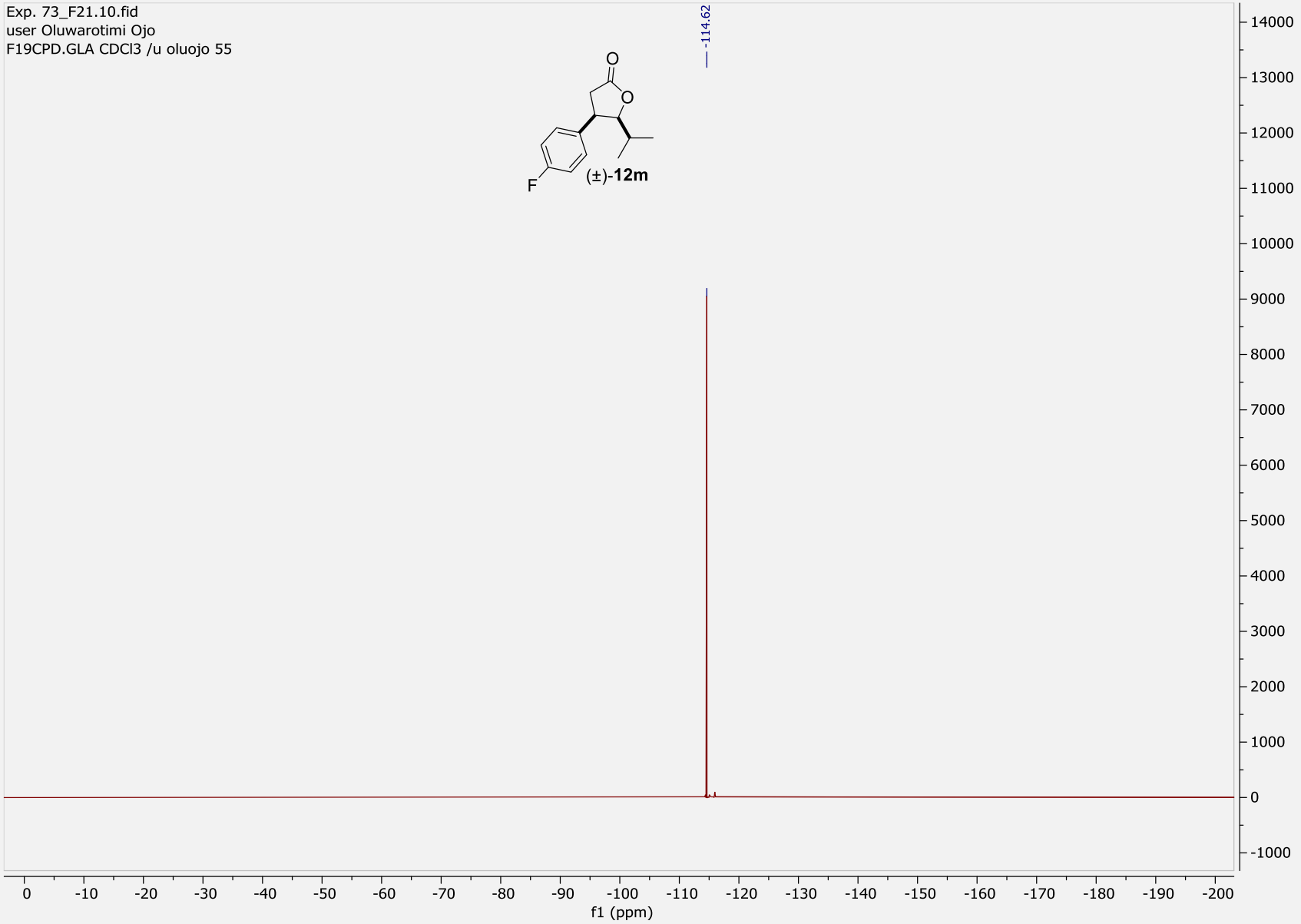

Exp. 72P.12.fid
O
O
F
(±)-12l
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
1.08
2.00
1.02
1.07
1.16
1.08
1.08
1.11
3.19
3.12
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

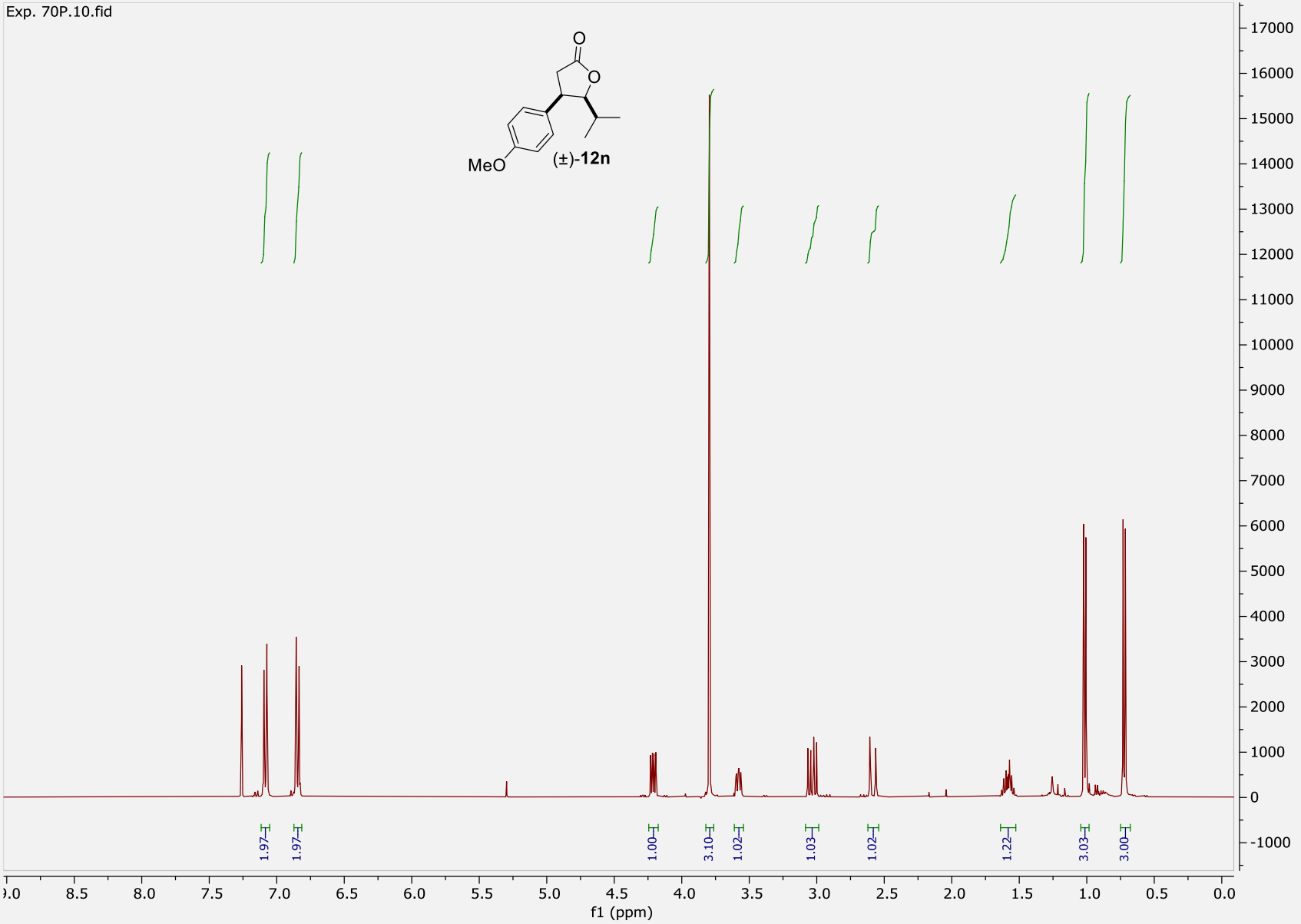

Exp. 72P.13.fid
176.74
164.15
161.69
141.76
141.69
130.56
130.48
123.75
123.72
115.37
115.16
114.81
114.61
89.79
44.19
44.17
38.76
28.89
20.05
17.80
F
O
O
(±)-12l
f1 (ppm)

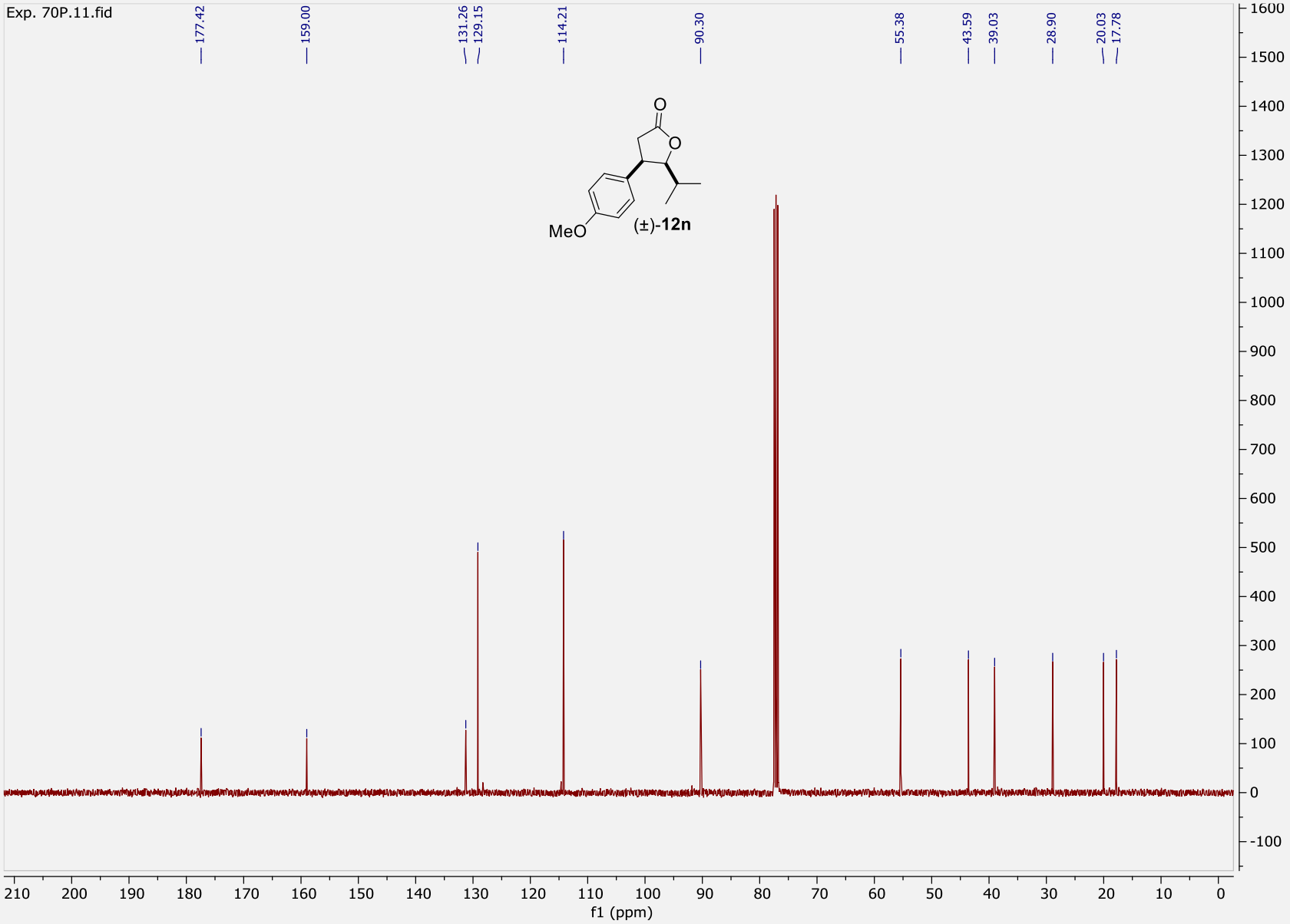

Exp. 72.20.fid
user Oluwarotimi Ojo
F19CPD.GLA CDCl3 /u oluojo 54
-112.02
O
O
F
(±)-12l
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

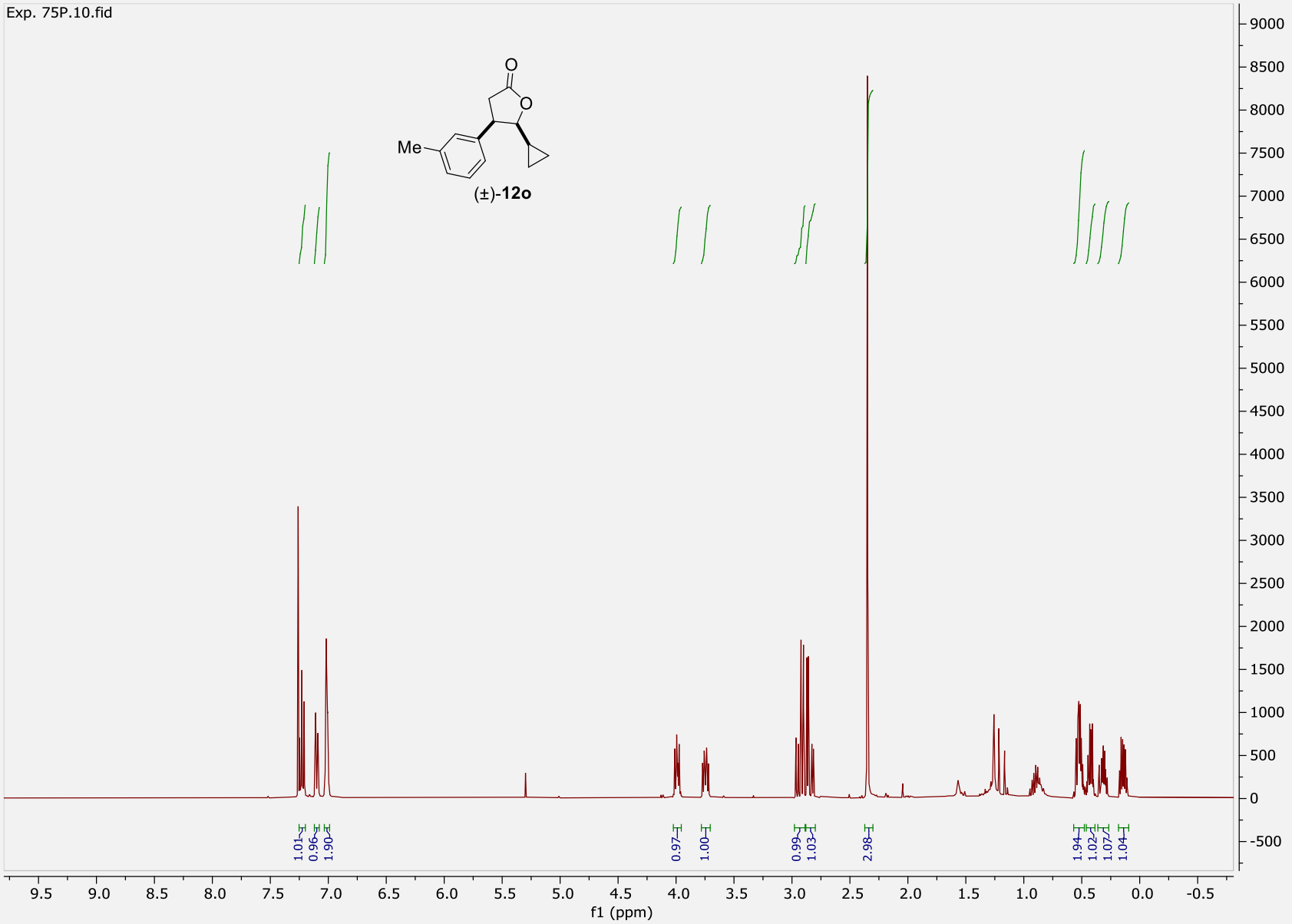

Exp. 73_F21.10.fid
O
O
F
(±)-12m
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
1.96
1.96
1.03
1.02
1.05
1.06
1.31
3.00
3.03
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

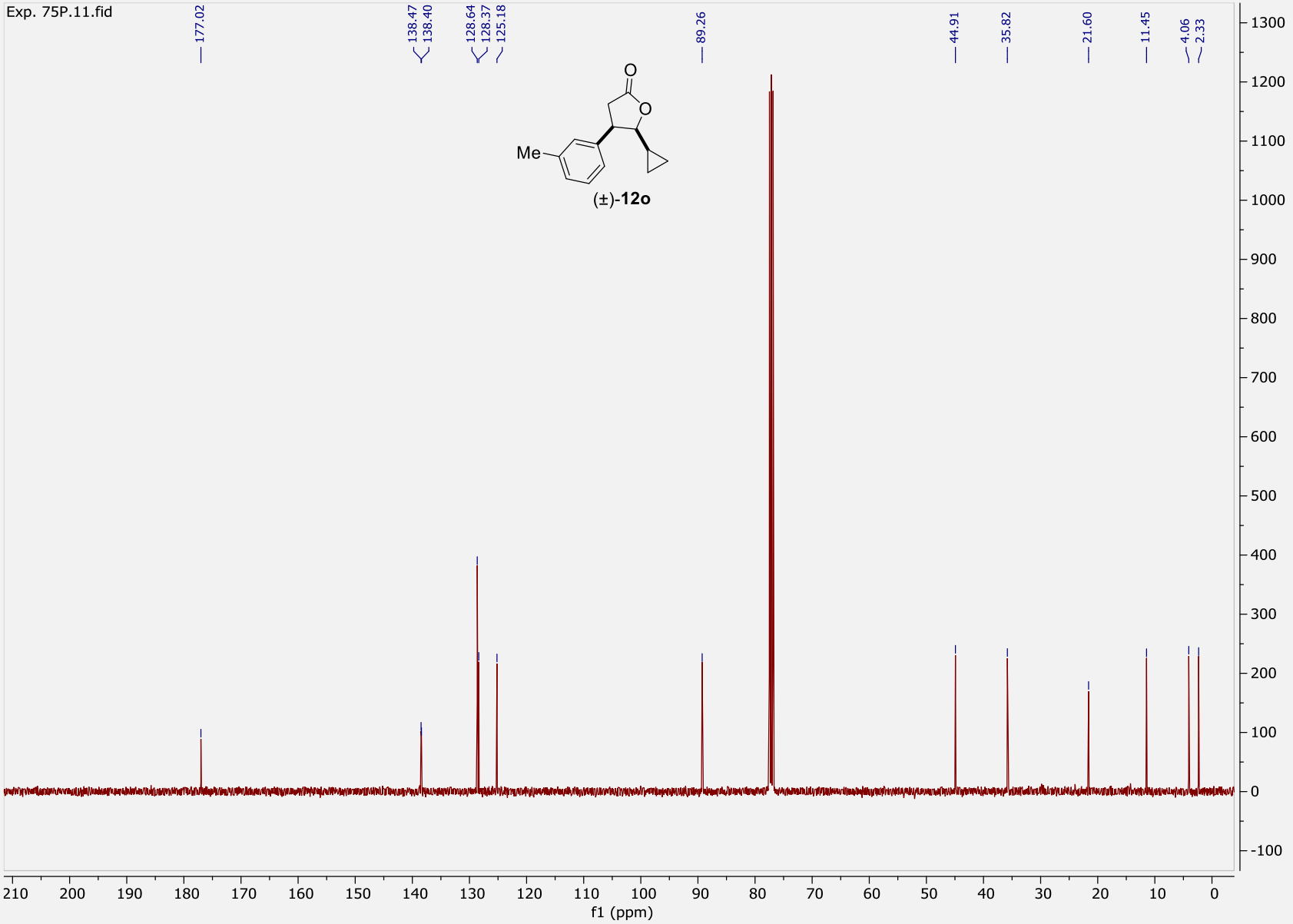

Exp. 73_F21.12.fid
176.99
163.44
160.99
135.07
135.04
129.68
129.60
115.95
115.74
89.98
43.68
39.00
28.88
20.04
17.69
O
O
F
(±)-12m
f1 (ppm)

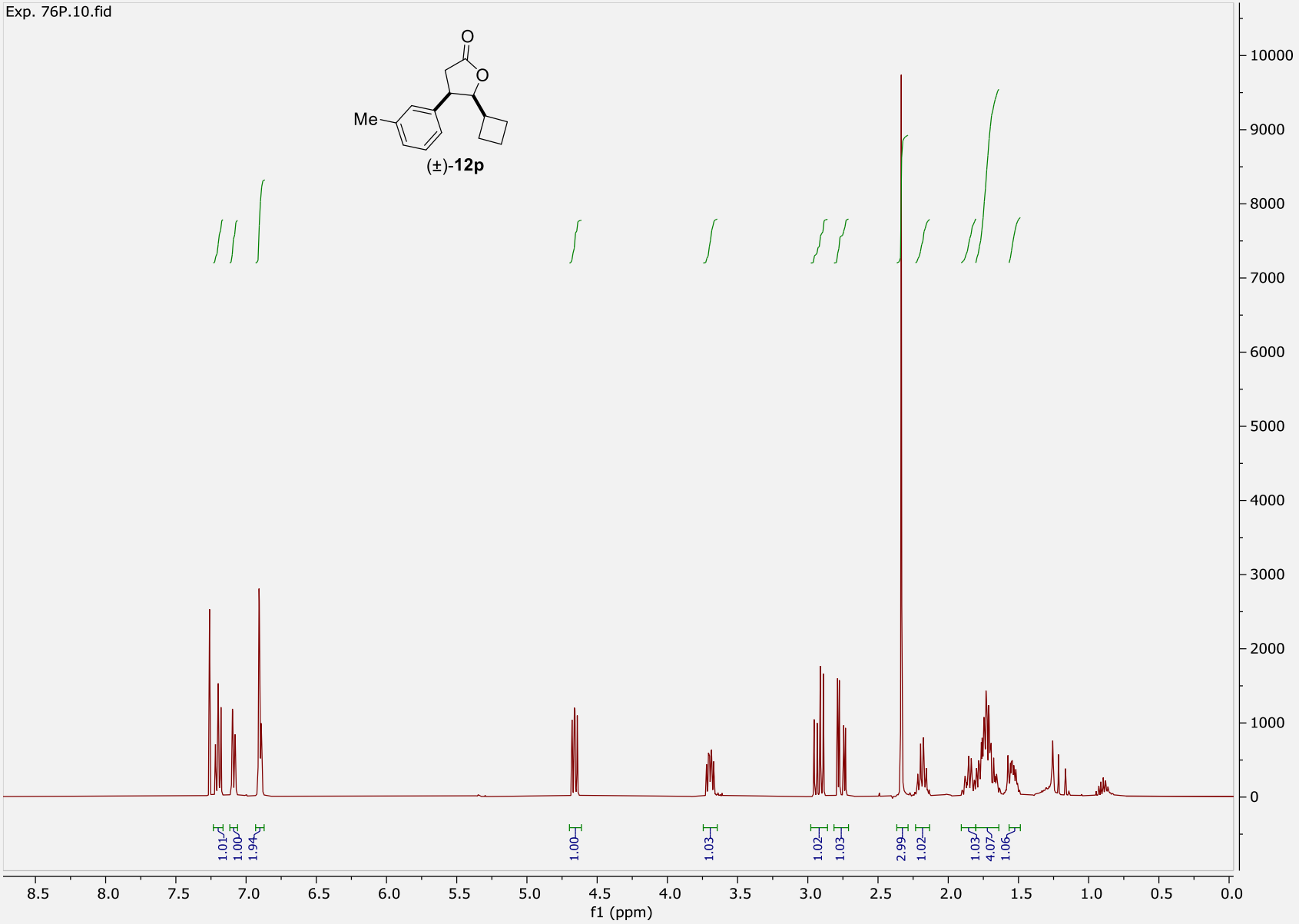

Exp. 73_F21.10.fid
user Oluwarotimi Ojo
F19CPD.GLA CDCl3 /u oluojo 55
-114.62
O
O
F
(±)-12m
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

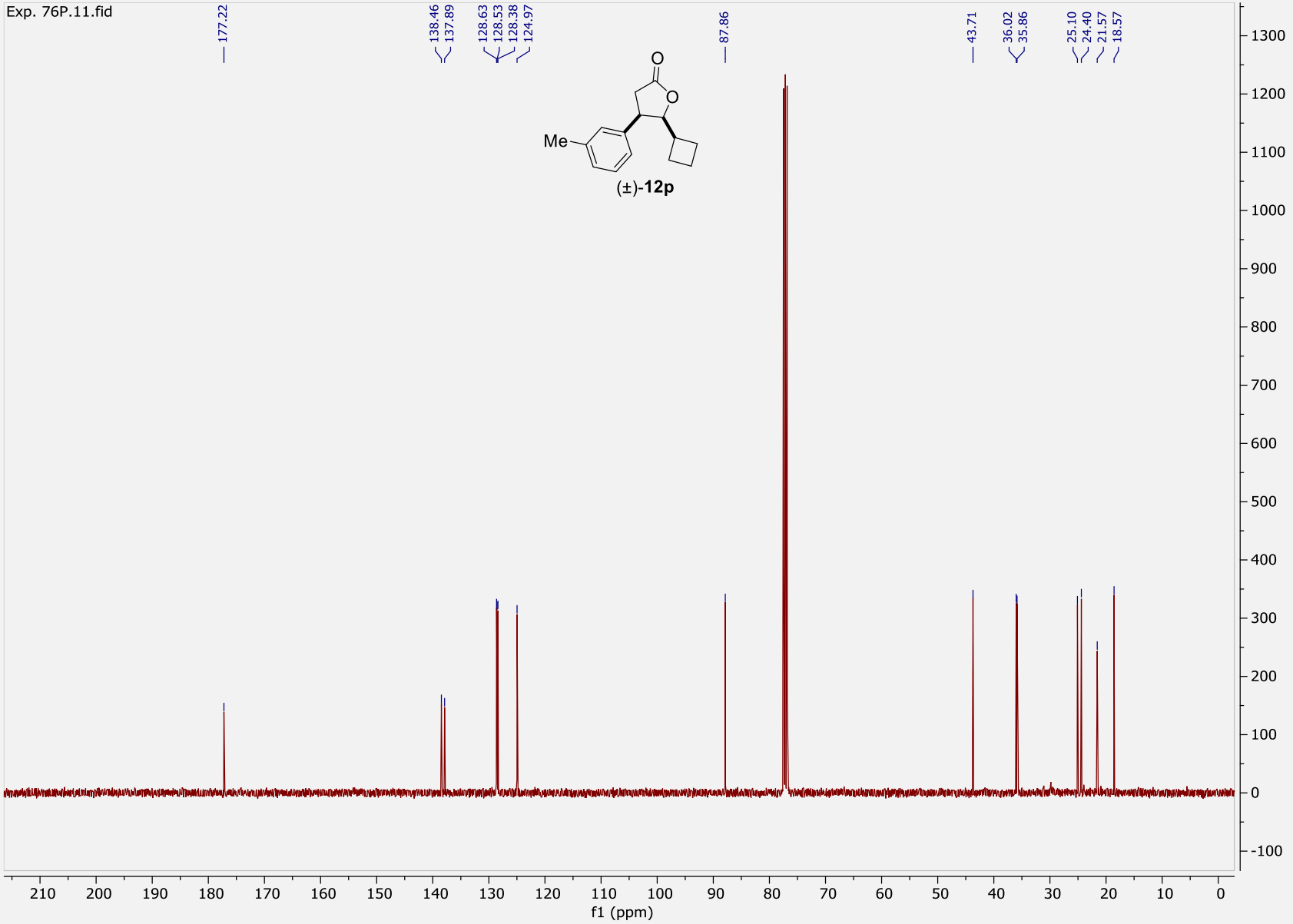

Exp. 70P.10.fid
MeO
(±)-12n
1.97
1.97
1.00
3.10
1.02
1.03
1.02
1.22
3.03
3.00
f1 (ppm)

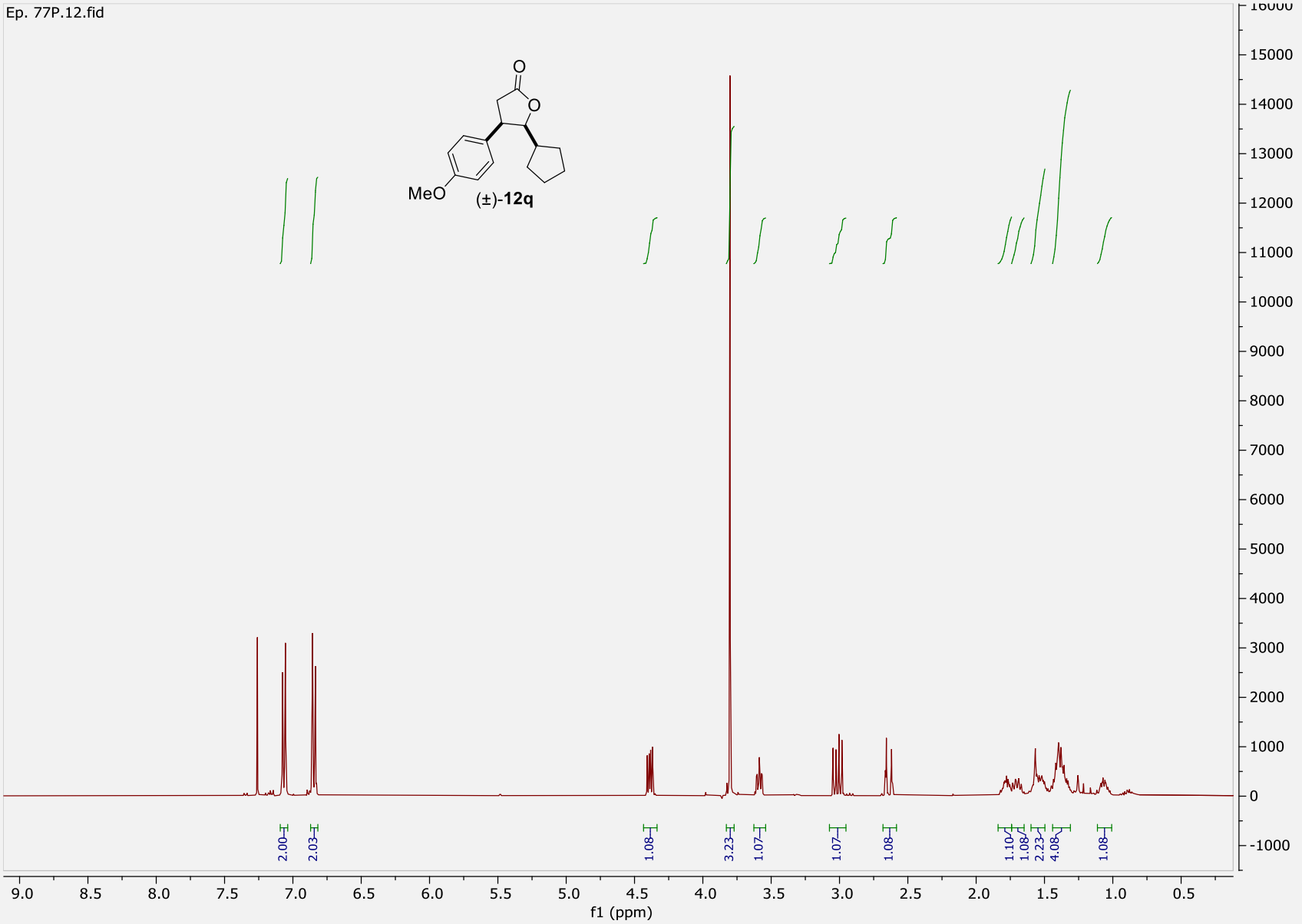

Exp. 70P.11.fid
177.42
159.00
131.26
129.15
114.21
90.30
55.38
43.59
39.03
28.90
20.03
17.78
MeO
(±)-12n
f1 (ppm)

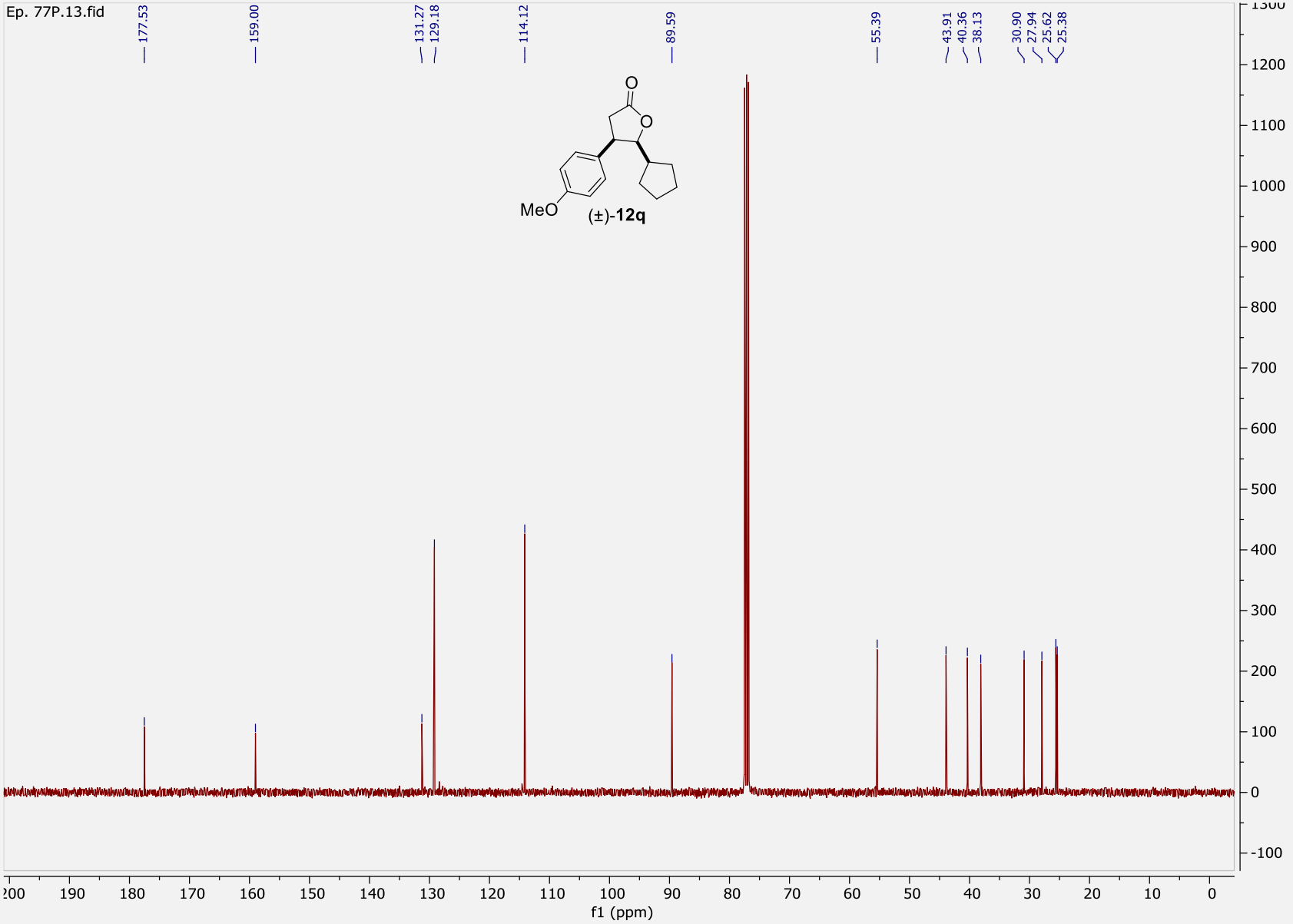

Exp. 75P.10.fid
Me
(±)-12o
1.01
0.96
1.90
0.97
1.00
0.99
1.03
2.98
1.94
1.02
1.07
1.04
f1 (ppm)

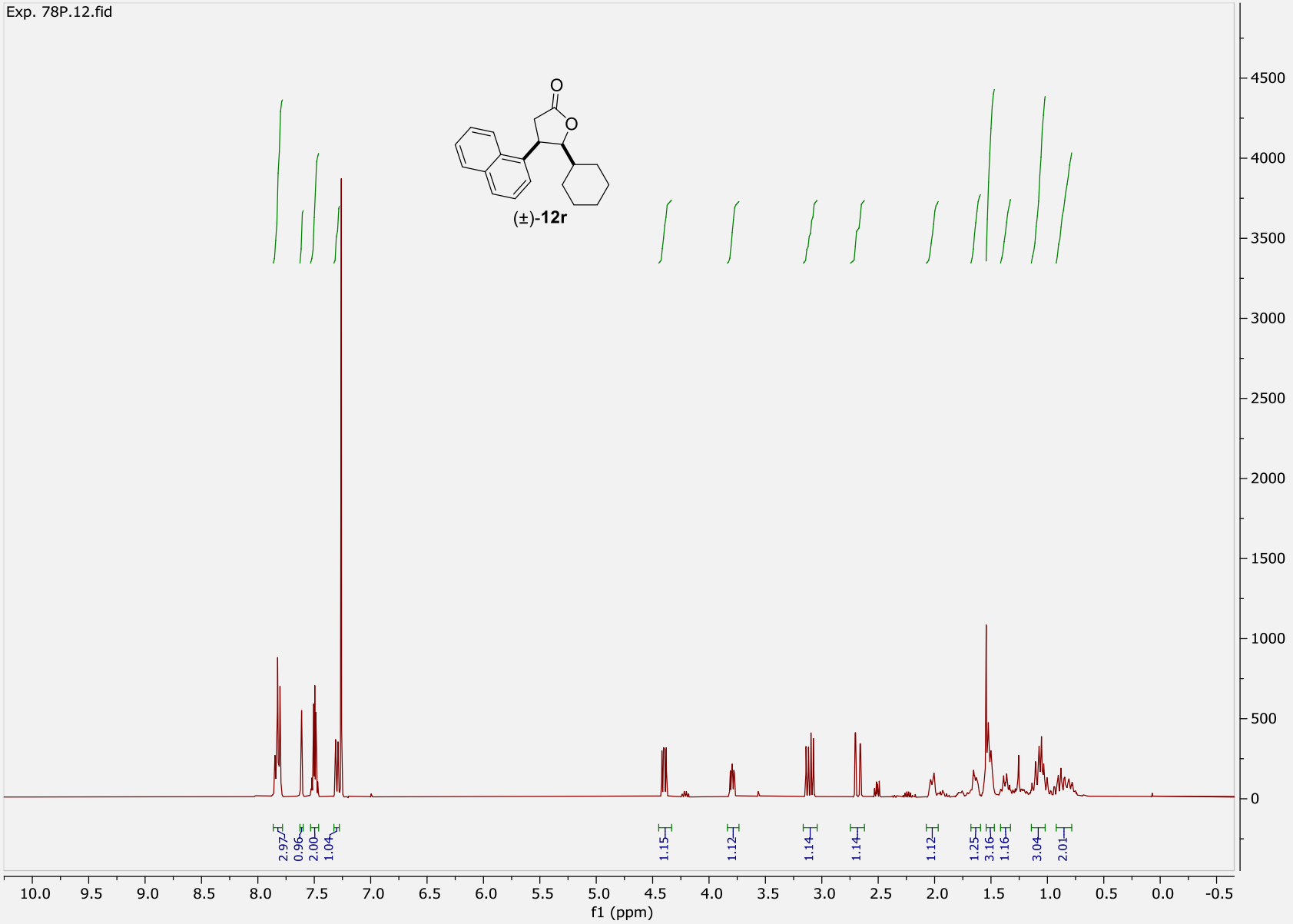

Exp. 75P.11.fid
177.02
138.47
138.40
128.64
128.37
125.18
89.26
44.91
35.82
21.60
11.45
4.06
2.33
O
O
Me
(±)-12o
f1 (ppm)

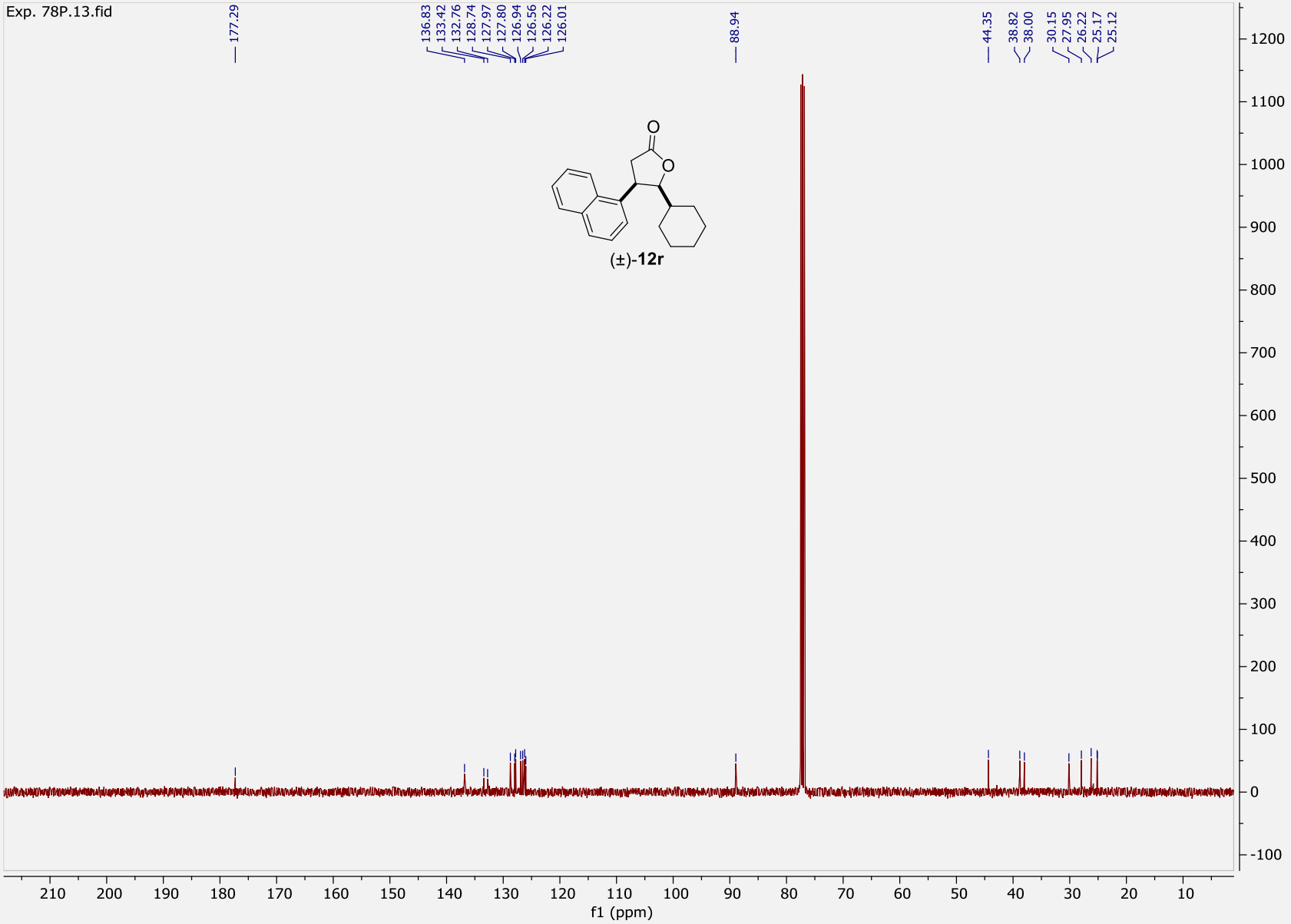

Exp. 76P.10.fid
Me
(±)-12p
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
1.01
1.00
1.94
1.00
1.03
1.02
1.03
2.99
1.02
1.03
4.07
1.06
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

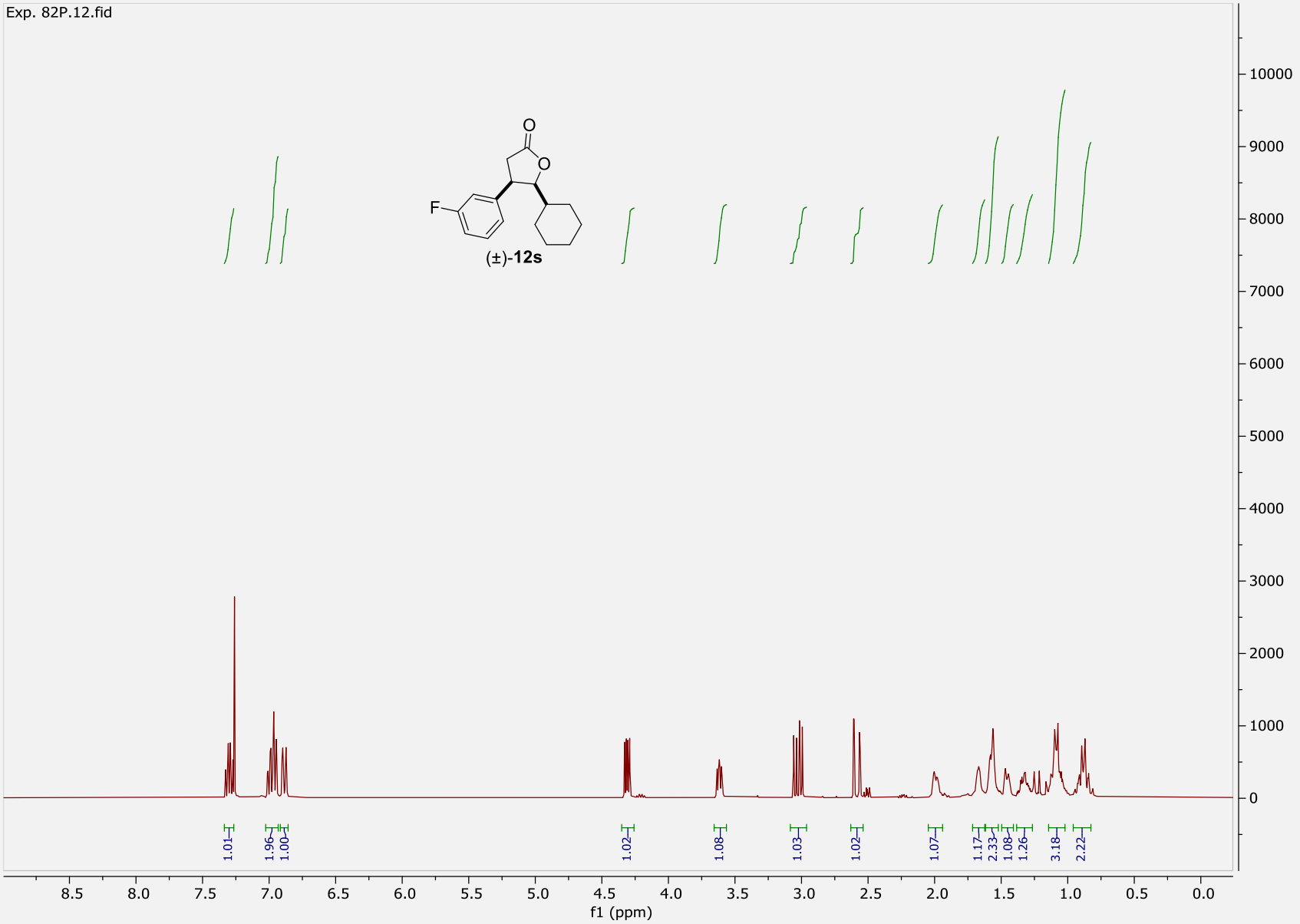

Exp. 76P.11.fid
177.22
138.46
137.89
128.63
128.53
128.38
124.97
87.86
43.71
36.02
35.86
25.10
24.40
21.57
18.57
O
O
Me
(±)-12p
f1 (ppm)

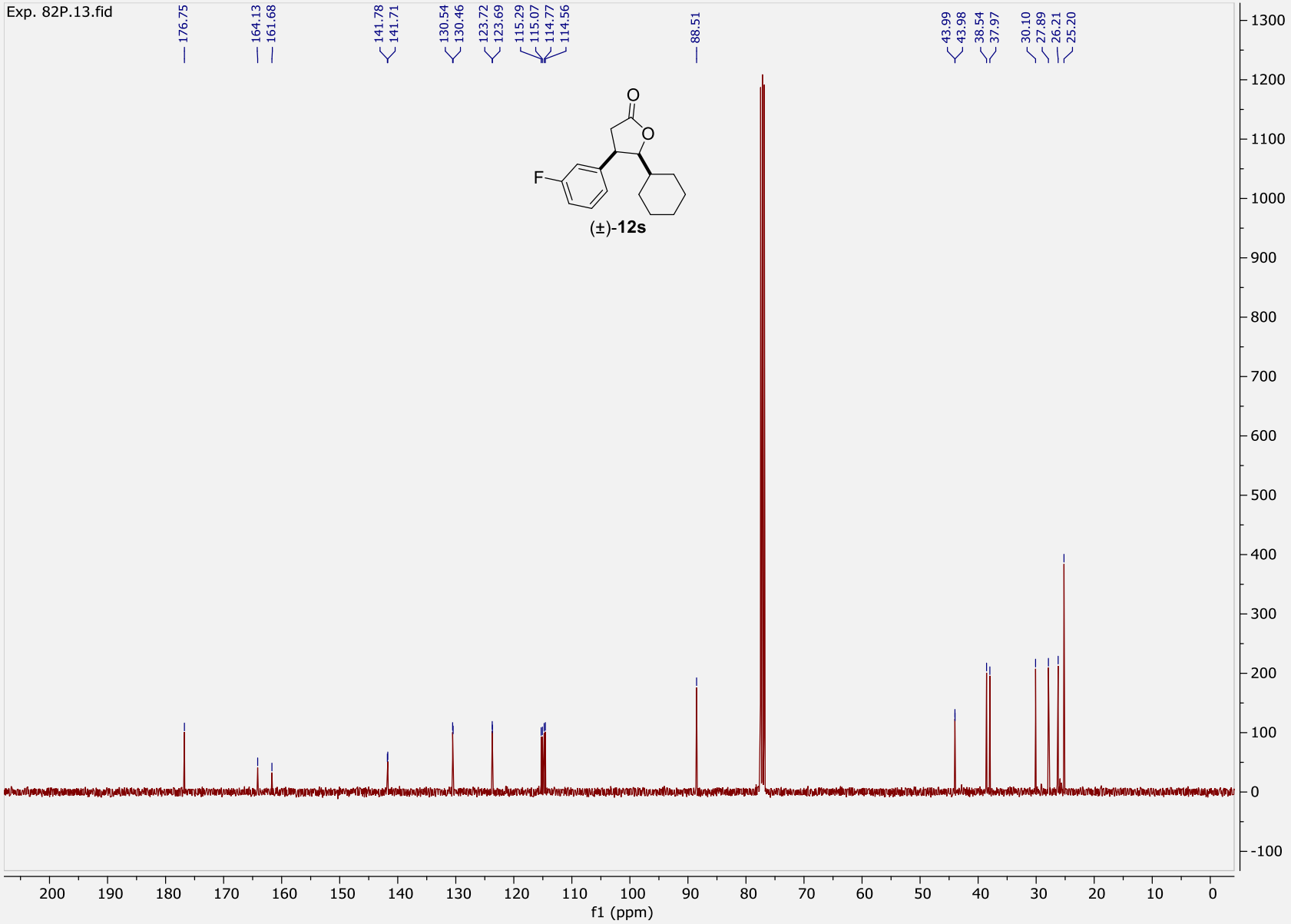

Ep. 77P.12.fid
O
O
MeO
(±)-12q
2.00
2.03
1.08
3.23
1.07
1.07
1.08
1.10
1.08
2.23
4.08
1.08
f1 (ppm)

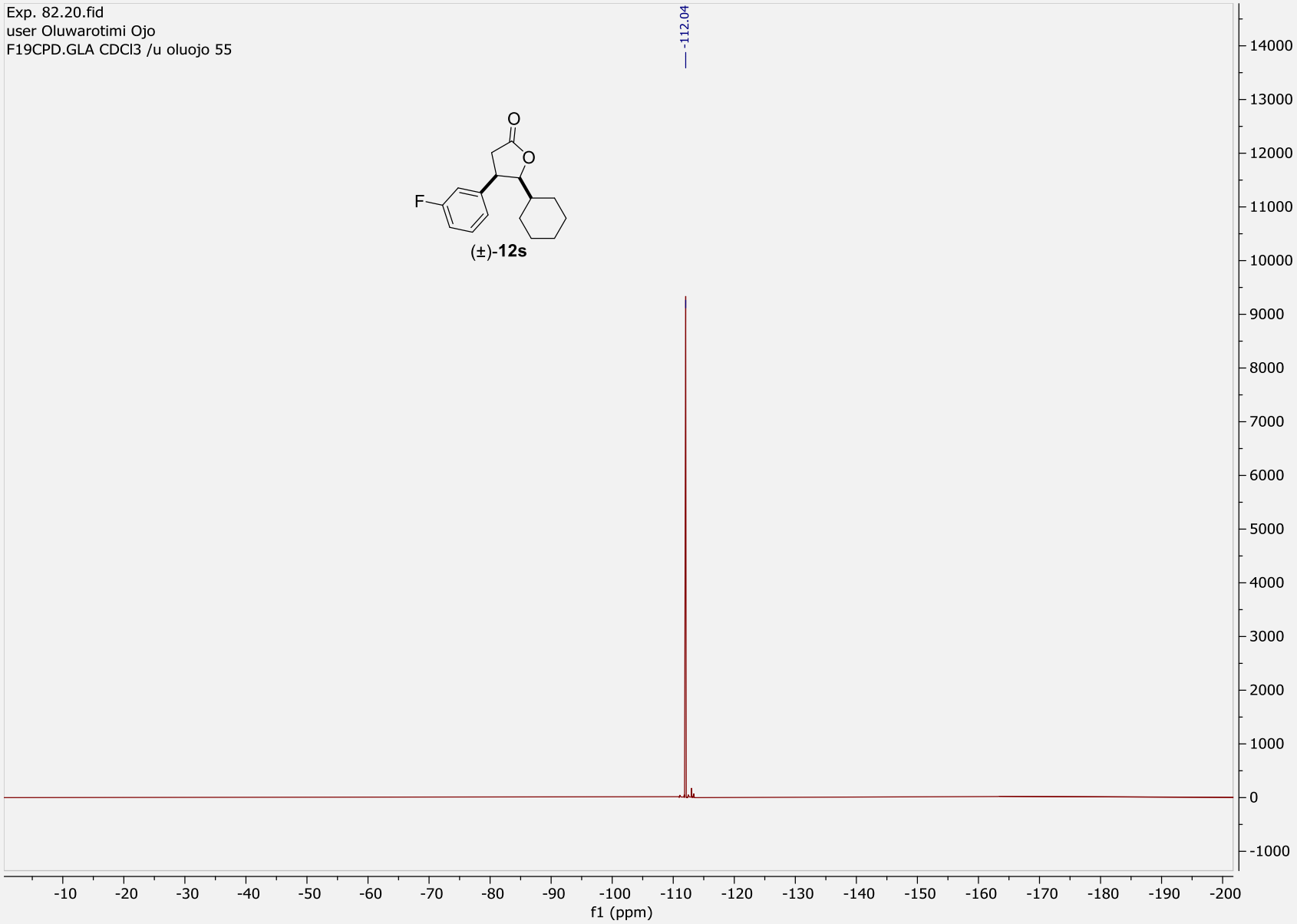

Ep. 77P.13.fid
177.53
159.00
131.27
129.18
114.12
89.59
55.39
43.91
40.36
38.13
30.90
27.94
25.62
25.38
MeO
(±)-12q
f1 (ppm)

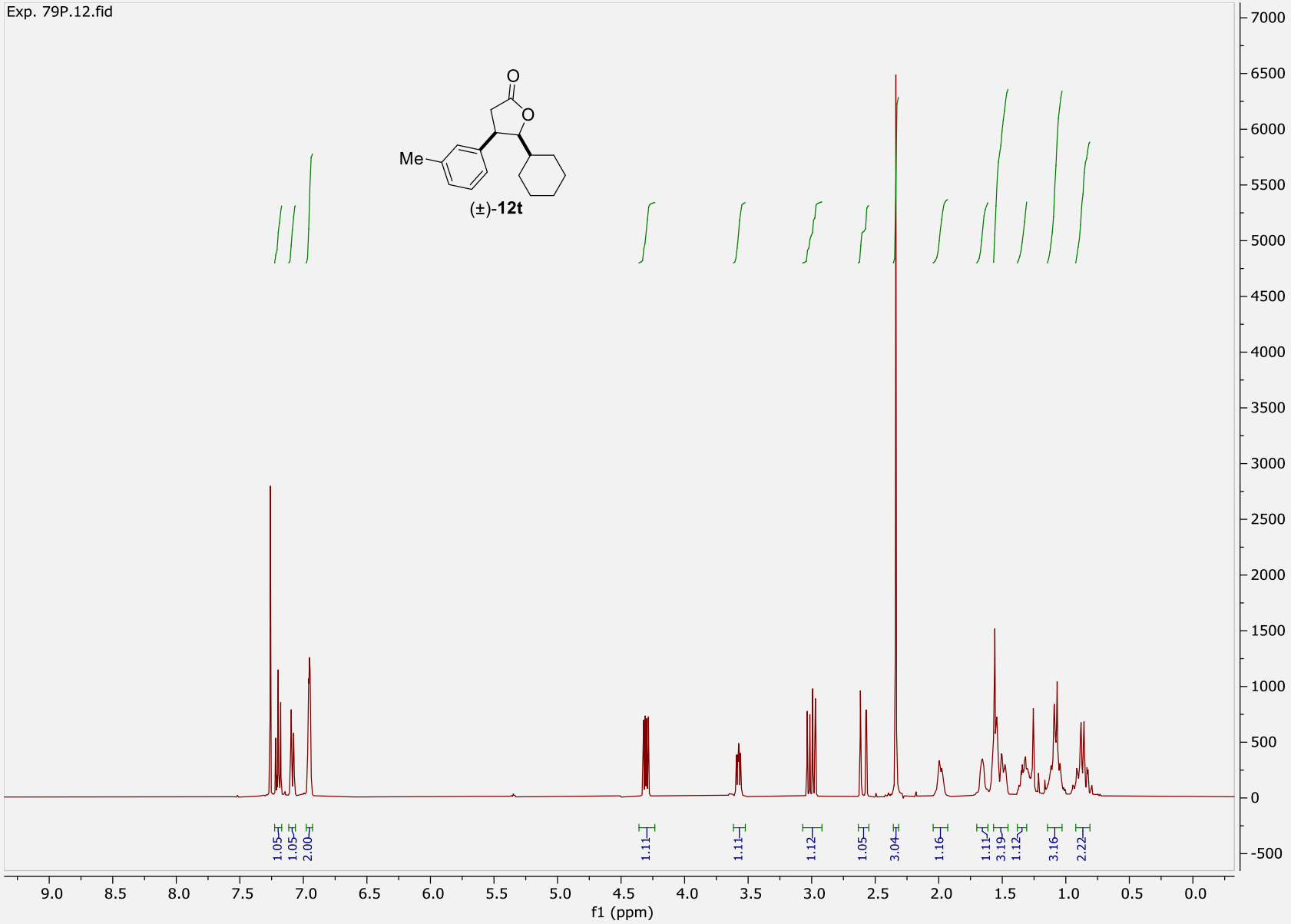

Exp. 78P.12.fid
(±)-12r
4500
4000
3500
3000
2500
2000
1500
1000
500
0
2.97
0.96
2.00
1.04
1.15
1.12
1.14
1.14
1.12
1.25
3.16
1.16
3.04
2.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

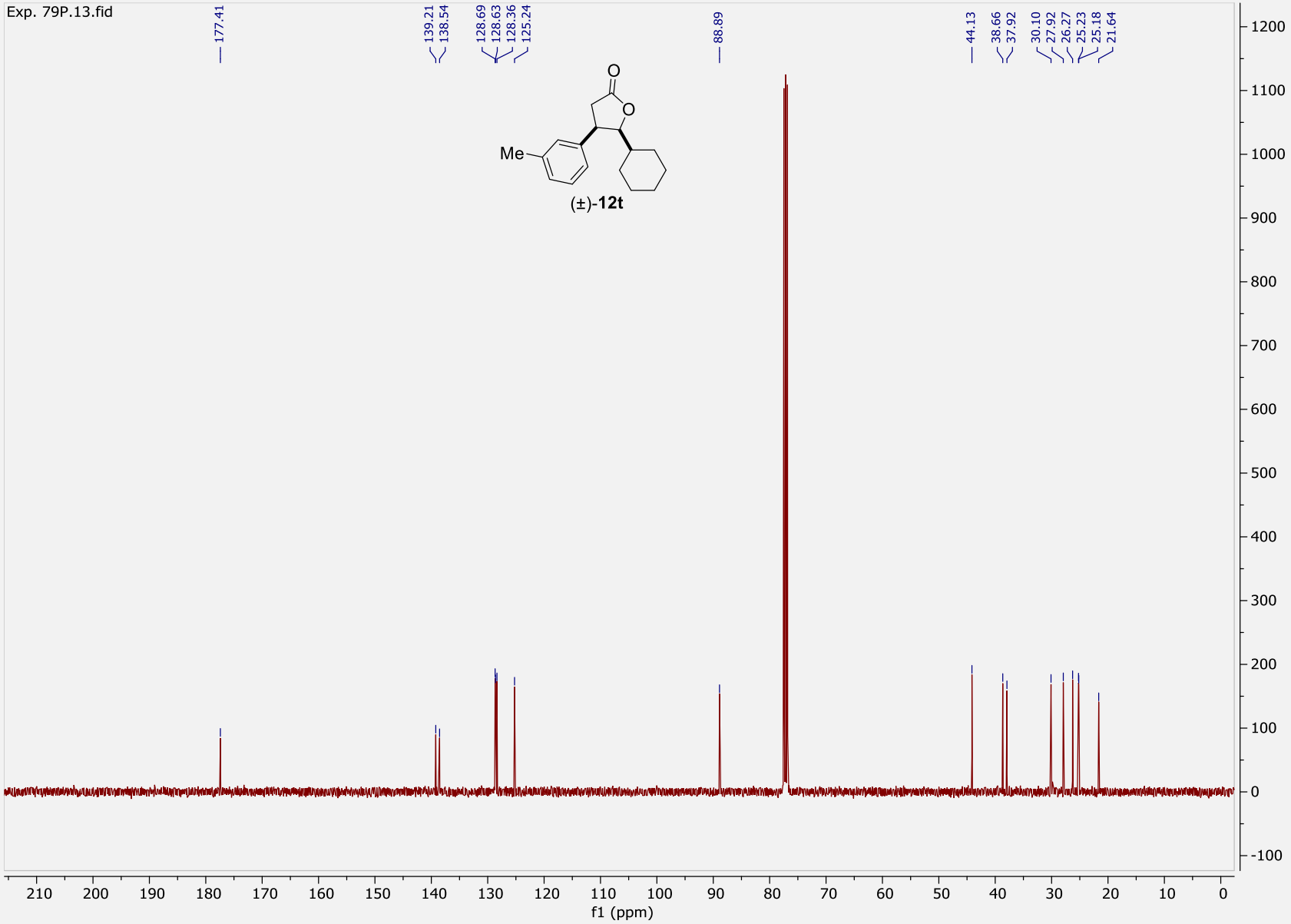

Exp. 78P.13.fid
177.29
136.83
133.42
132.76
128.74
127.97
127.80
126.94
126.56
126.22
126.01
88.94
44.35
38.82
38.00
30.15
27.95
26.22
25.17
25.12
O
O
(±)-12r
f1 (ppm)

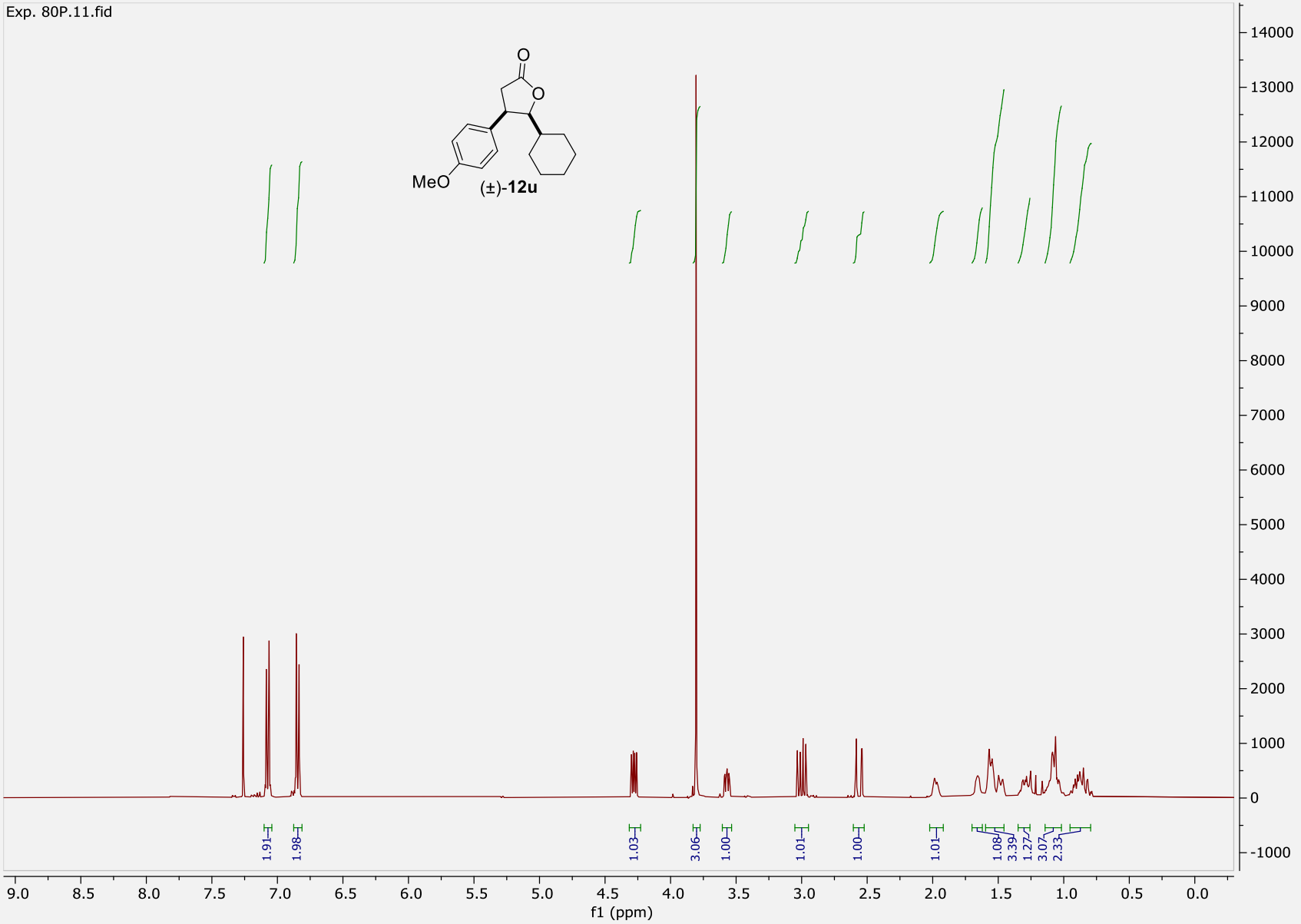

Exp. 82P.12.fid
O
O
F
(±)-12s
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
1.01
1.96
1.00
1.02
1.08
1.03
1.02
1.07
1.17
2.33
1.08
1.26
3.18
2.22
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

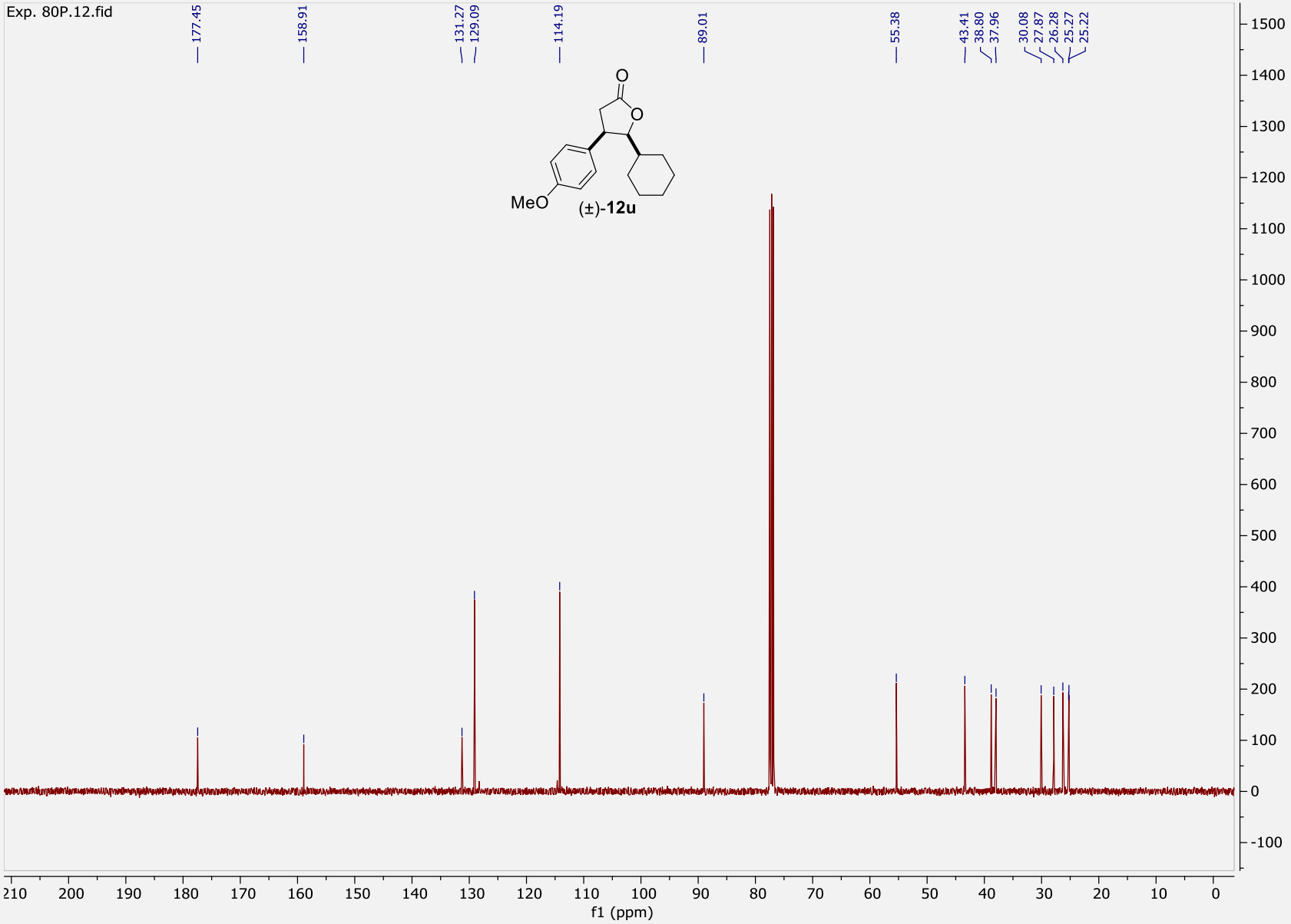

Exp. 82P.13.fid
176.75
164.13
161.68
141.78
141.71
130.54
130.46
123.72
123.69
115.29
115.07
114.77
114.56
88.51
43.99
43.98
38.54
37.97
30.10
27.89
26.21
25.20
O
O
F
(±)-12s
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

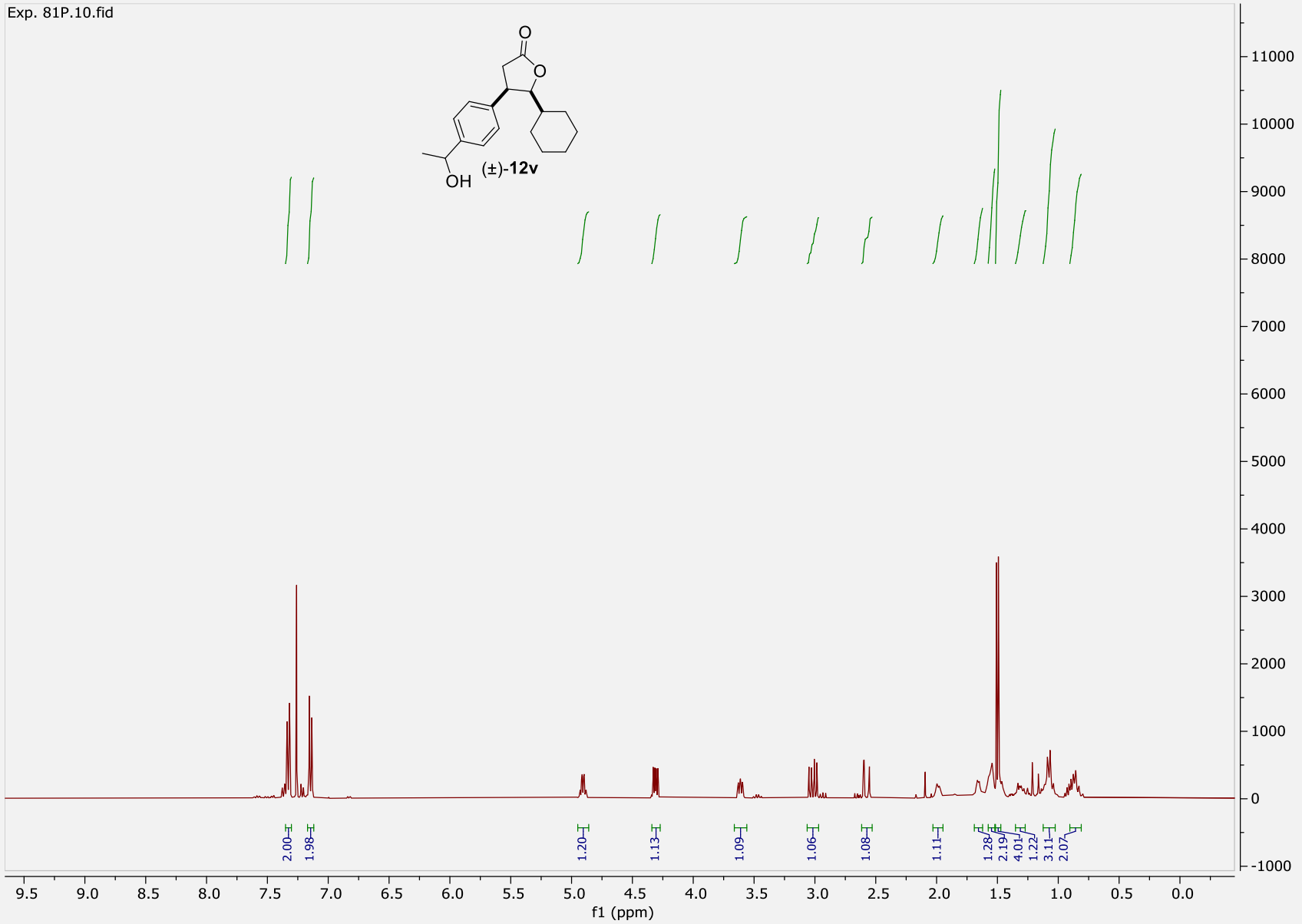

Exp. 82.20.fid
user Oluwarotimi Ojo
F19CPD.GLA CDCl3 /u oluojo 55
-112.04
O
O
F
(±)-12s
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

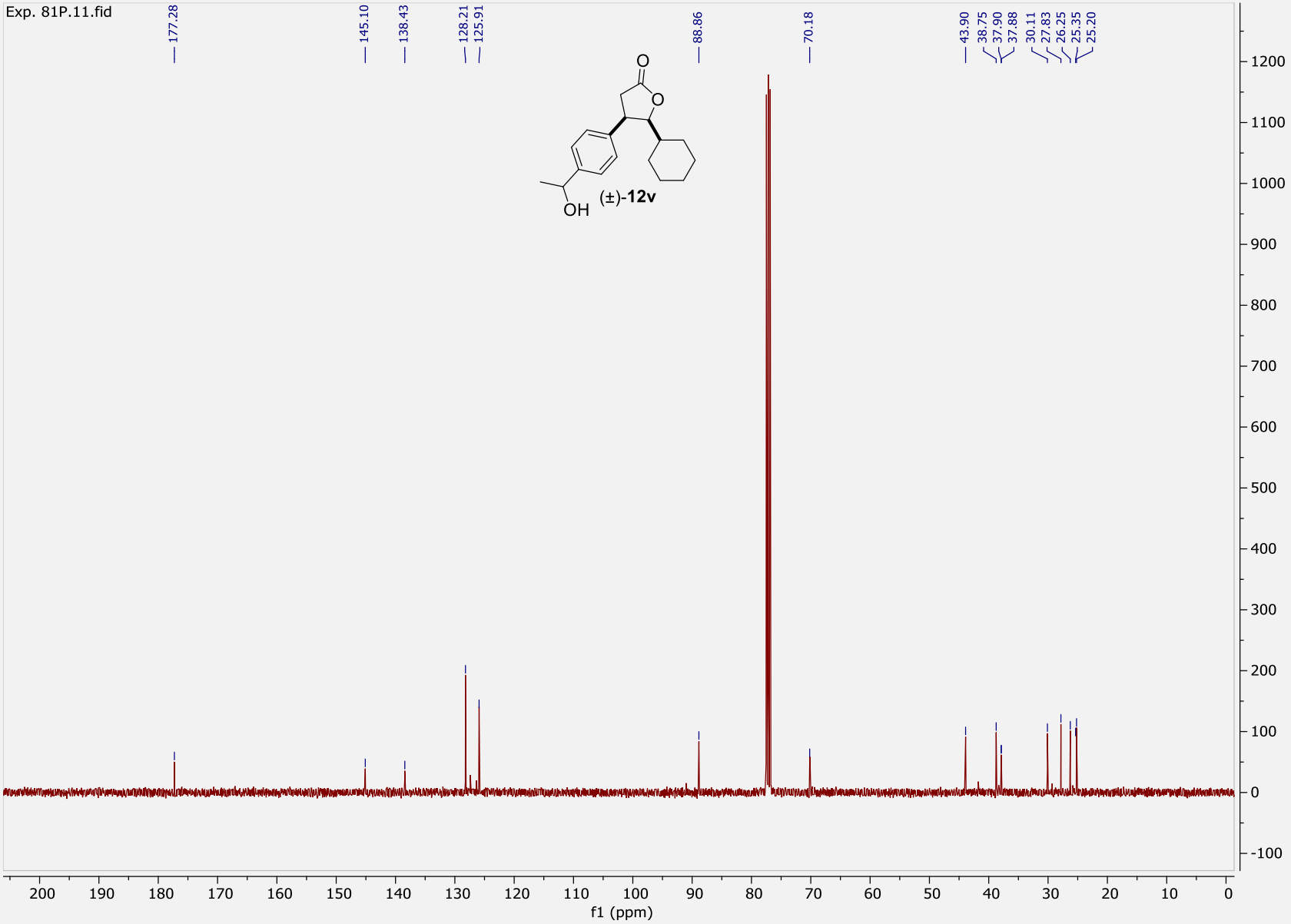

Exp. 79P.12.fid
O
O
Me
(±)-12t
1.05
1.05
2.00
1.11
1.11
1.12
1.05
3.04
1.16
1.11
3.19
1.12
3.16
2.22
f1 (ppm)


Exp. 79P.13.fid
177.41
139.21
138.54
128.69
128.63
128.36
125.24
88.89
44.13
38.66
37.92
30.10
27.92
26.27
25.23
25.18
21.64
Me
(±)-12t
f1 (ppm)


Exp. 80P.11.fid
O
O
MeO
(±)-12u
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
1.91
1.98
1.03
3.06
1.00
1.01
1.00
1.01
1.08
3.39
1.27
3.07
2.33
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


Exp. 80P.12.fid
177.45
158.91
131.27
129.09
114.19
89.01
55.38
43.41
38.80
37.96
30.08
27.87
26.28
25.27
25.22
MeO
(±)-12u
f1 (ppm)


Exp. 81P.10.fid
O
O
OH
(±)-12v
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
2.00
1.98
1.20
1.13
1.09
1.06
1.08
1.11
1.28
2.19
4.01
1.22
3.11
2.07
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


Exp. 81P.11.fid
177.28
145.10
138.43
128.21
125.91
88.86
70.18
43.90
38.75
37.90
37.88
30.11
27.83
26.25
25.35
25.20
O
O
OH
(±)-12v
f1 (ppm)