Supporting Information

1. General Information

1.1 Reagents

Metals

Manganese powder -325 mesh (Alfa Aesar) was used as received and stored in a nitrogen filled glovebox. Nickel(II) chloride ethylene glycol dimethyl ether ($NiCl_2$•dme) was either purchased from Strem or synthesized according to the literature procedure [1] and stored in a nitrogen filled glovebox. The molar ratio of dme present in the $NiCl_2$•dme can vary between 0.1 and 1.0 was determined by elemental analysis and the molar mass of $NiCl_2$•dme_n was calculated accordingly.

Ligands

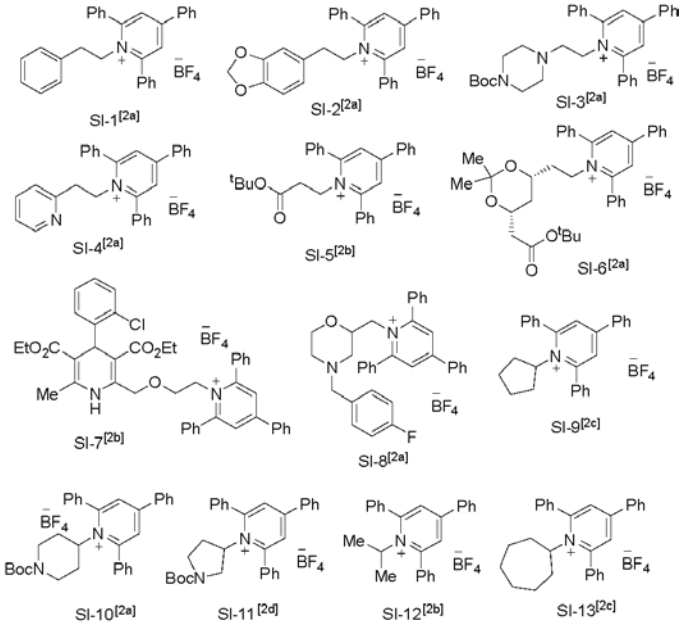
All ligands tested were from commercial suppliers and used as received.

Solvents

Anhydrous 1-methyl-2-pyrrolidinone (NMP) (Sigma Aldrich) was used as received and stored in a nitrogen filled glovebox. Tetrahydrofuran (THF) and dichloromethane (DCM) were purified by passage though activated alumina and molecular sieves in a solvent purification system and stored in a nitrogen-filled glovebox.

Preparation of Alkyl Pyridinium Salts

Pyridinium salts were prepared according to previously reported procedures.[2]

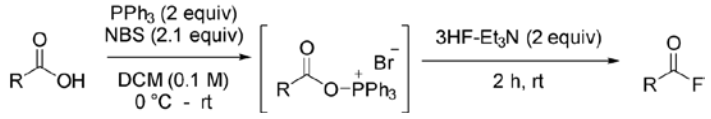


[1] L. G. L. Ward, J. R. Pipal, *Inorg. Synth*, **2007**, pp. 154–164.

[2] a) C. H. Basch, J. Liao, J. Xu, J. J. Piane, M. P. Watson, *J. Am. Chem. Soc*. **2017**, *139*, 5313-5316. b) J. Liao, C. H. Basch, M. E. Hoerrner, M. R. Talley, B. P. Boscoe, J. W. Tucker, M. R. Garnsey, M. P. Watson, *Org. Lett*. **2019***, 21*, 2941-2946. c) F. J. R. Klauck, M. J. James, F. Glorius, *Angew. Chem. Int. Ed*. **2017***, 56*, 12336-12339. d) S. Plunkett, C. H. Basch, S. O. Santana, M. P. Watson, *J. Am. Chem. Soc.* **2019**, *141*, 2257-2262.

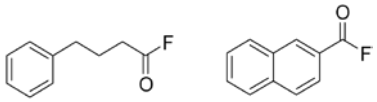
Preparation of Isolated Acyl Fluorides (GP-1)

In most cases acyl fluorides were generated *in situ* from carboxylic acids, but for a few examples, where separation of the product from the proton-sponge-derived side products was challenging, acyl fluorides were isolated and purified first using an adaptation of a known procedure.[3]

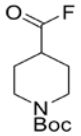


An oven dried round-bottom flask equipped with a magnetic stir bar was charged with carboxylic acid (2.0 mmol, 1.0 equiv) and triphenylphosphine (1.05 g, 4.0 mmol, 2.0 equiv). Anhydrous dichloromethane (20 mL) was added, the flask was capped, and the mixture was cooled with stirring to 0 °C using an ice-water bath. Once the mixture was cooled, *N*-bromosuccinimide (NBS) (0.75 g, 4.2 mmol, 2.1 equiv) was added in one portion with stirring. After two min of stirring, the ice-bath was removed and the mixture was further stirred at rt for 15 min. Subsequently, the flask was opened and triethylamine trihydrogen fluoride (0.65 mL, 4.0 mmol, 2.0 equiv) was added via micropipette. The resulting mixture was stirred for 2 h at rt before it was diluted with hexanes (60 mL). The resulting solution was further stirred for 10 min, during which a large amount of triphenylphosphine oxide and succinimide precipitate. The solids were removed by filtration through a short pad of silica gel (2 cm thick x 3 cm diameter) and the pad was washed with further portions of hexanes or a mixture of hexanes and ethyl acetate. The filtrate was then concentrated under reduced pressure on a rotary evaporator to afford the analytically pure acid fluoride.

The preparation and characterization of acyl fluorides SII-1 and SII-4 has been reported.[3]



***tert*-butyl 4-(fluorocarbonyl)piperidine-1-carboxylate (SII-2)**



Following **GP-1**, using 1-(*tert*-butoxycarbonyl)piperidine-4-carboxylic acid (0.458 g, 2 mmol, 1.0 equiv), PPh_3 (1.05 g, 4.0 mmol, 2.0 equiv), NBS (0.75 g, 4.2 mmol, 2.1 equiv) and 3HF-Et_3N (0.65 mL, 4.0 mmol, 2.0 equiv). Purified by filtration through a short plug of silica (2 cm thick × 3 cm diameter), washing the silica plug with a solvent mixture of hexanes and ethyl acetate (10:1) to obtain SII-2 as a colorless oil in 52% yield (241 mg).

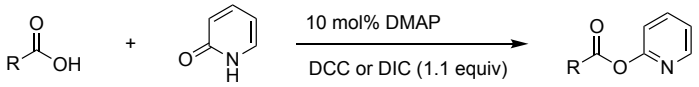
IR: 2975, 1834, 1687, 1417, 1162, 1127, 1013,863 cm^{-1}
^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.01 (d, *J* = 13.7 Hz, 2H), 2.93 (t, *J* = 12.4 Hz, 2H), 2.68 (tt, *J* = 10.6, 3.9 Hz, 1H), 1.96 (dt, *J* = 12.5, 3.8 Hz, 2H), 1.71 (dtd, *J* = 14.6, 10.8, 4.2 Hz, 2H), 1.46 (s, 9H).

[3] S. B. Munoz, H. Dang, X. Ispizua-Rodriguez, T. Mathew, G. K. S. Prakash, *Org. Lett.*, **2019**, *21*, 1659-1663.

^{13}C NMR (126 MHz, $CDCl_3$) δ 164.2 (d, $J_{C\text{-}F}$ = 366.1 Hz), 154.5, 80.0, 42.5, 39.5 (d, $J_{C\text{-}F}$ = 48.9 Hz), 28.4, 27.1.
^{19}F NMR (377 MHz, Chloroform-*d*) δ 37.25.
HR-MS (ESI) m/z calculated for $C_{11}H_{18}FNO_3$ $[M+Na]^+$ 254.1163, found 254.1159.

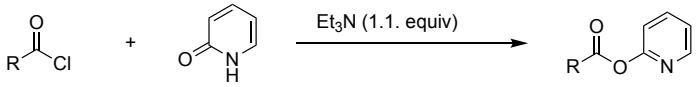
Preparation of 2-pyridyl esters (GP-2)
The 2-pyridyl esters can be prepared from carboxylic acid chlorides or, preferably, by direct esterification.[4]

Direct esterification of acid (GP-2A)



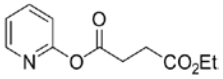
An oven dried round-bottom flask or scintillation vial equipped with a magnetic stir bar was charged with carboxylic acid (2.0 mmol, 1.0 equiv), *N*,*N*-dimethylaminopyridine (DMAP) (0.2 mmol, 0.1 equiv), and dichloromethane, resulting in a solution between 0.1-0.2 M in carboxylic acid). To this solution was added *N*,*N*-dicyclohexylcarbodiimide (DCC) (0.45 g, 2.2 mmol, 1.1 equiv) or *N*,*N*-diisopropylcarbodiimide (DIC) (0.35 mL, 2.2 mmol, 1.1 equiv). The reaction mixture was stirred vigorously for 10 min at rt before 2-pyridinone (0.19 g, 2.0 mmol, 1.0 equiv) was added as a solid. The reaction mixture was allowed to stir until the carboxylic acid was consumed, as determined by TLC analysis, before it was filtered through a short pad of silica gel (20 g, rinsed with additional 50 mL cold Et_2O). The filtrate was concentrated under reduced pressure and the residue was purified by column chromatography (SiO_2) to afford the corresponding 2-pyridyl ester.

Esterification via acid chloride (GP-2B)



An oven dried round-bottom flask equipped with a magnetic stir bar was charged with Et_3N (0.31 mL, 2.2 mmol, 1.1 equiv), 2-pyridinone (0.19 g, 2.0 mmol, 1.0 equiv) and anhydrous CH_2Cl_2 (10 mL). The reaction mixture was then cooled to 0 °C using an ice-water bath. The corresponding acyl chloride (2.0 mmol, 1.0 equiv) was then added drop-wise to the reaction mixture over 1 min before the cooling bath was removed. After stirring the reaction mixture for 6 h, the reaction mixture was filtered through a short pad of silica gel (5 g) and the silica pad was washed with ethyl acetate (3 × 20 mL). The filtrate was concentrated under reduced pressure and the residue was purified by flash chromatography to afford the corresponding 2-pyridyl esters.

ethyl pyridin-2-yl succinate (**SII-5**)



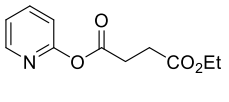
Following **GP-2A**, using 4-ethoxy-4-oxobutanoic acid (0.73 g, 5.0 mmol, 1.0 equiv), DMAP (0.06 g, 0.5 mmol, 0.1 equiv), DCC (1.13g, 5.5 mmol, 1.1 equiv) and 2-pyridinone (0.48 g, 5 mmol, 1.0

[4] B. Neises, W. Steglich, *Angew. Chem. Int. Ed*. **1978**, *17*, 522-524.

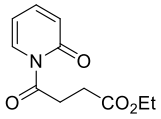
equiv) in 30 mL DCM for 12 h. The product was purified by flash silica gel chromatography (10-40% ethyl acetate/hexanes) to afford SII-5 as a colorless oil in 91% yield (1.02 g).

Alternatively, following **GP-2B**, using ethyl 4-chloro-4-oxobutyrate (1.4 mL, 10 mmol, 1.0 equiv), 2-pyridinone (0.95 g, 10 mmol, 1.0 equiv) and Et_3N (1.54 mL, 11 mmol, 1.1 equiv) in 20 mL of DCM. The product was purified by flash silica gel chromatography (10-40% ethyl acetate/hexanes) to afford SII-5 as a colorless oil in 81% yield (1.8 g).

Note: Compound SII-5 contains about 9% of the *N*-acylpyridinone (SII-5b) (10:1 ratio of SII-5 and SII-5b). Similar products have been reported sporadically in the literature from reactions with acid chlorides and there is some indication that the ratio depends upon substrate and conditions.[5] At this time, we are not sure if SII-5b can react in the same manner as SII-5 and yields for reactions with SII-5 were NOT adjusted for the lower amount of limiting reagent.]



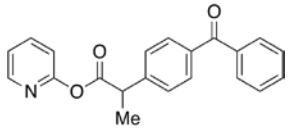
SII-5



SII-5b

IR 2935, 1763, 1591, 1202, 1122, 774 cm^{-1}
^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.40 (dd, *J* = 5.0, 1.9 Hz, 1H), 7.79 (td, *J* = 7.7, 2.0 Hz, 1H), 7.23 (dd, *J* = 7.3, 4.9 Hz, 1H), 7.11 (d, *J* = 8.1 Hz, 1H), 4.22 – 4.13 (m, 2H), 2.98 – 2.91 (m, 2H), 2.75 (t, *J* = 6.9 Hz, 2H), 1.27 (td, *J* = 7.1, 1.1 Hz, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 172.0, 170.6, 157.8, 148.6, 139.6, 122.1, 116.5, 60.9, 29.4, 29.0, 14.2.
HR-MS (ESI) m/z calculated for $C_{11}H_{13}NO_4$ $[M+H]^+$ 224.0917, found 224.0915.

pyridin-2-yl 2-(4-benzoylphenyl)propanoate (**SII-6**)

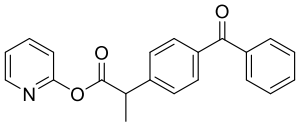


Following **GP-2A**, using Ketoprofen (1.0 g, 3.9 mmol, 1 equiv), DMAP (0.047 g 0.39 mmol, 0.1 equiv), DCC (0.887 g, 4.3 mmol, 1.1 equiv) and 2-pyridinone (0.371 g, 3.9 mmol, 1 equiv) in 26 mL of DCM. The product was purified by flash silica gel chromatography (20-60% diethyl ether/hexane) to afford SII-8 as a light yellow oil in 86% yield (1.11 g).

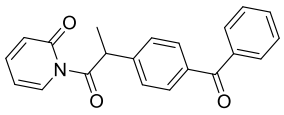
Alternatively, following **GP-2B**, using ketoprofen chloride (2.7 g, 10 mmol, 1 equiv), 2-pyridinone (0.95 g, 10 mmol, 1 equiv) and Et_3N (1.54 mL, 11 mmol, 1.1 equiv) in 30 mL of DCM. Purified by flash silica gel chromatography (20-60% diethyl ether/hexane) to afford SII-6 as a light yellow oil in 58% yield (1.93 g).

Note: as with **SII-5**, **SII-6** contains small amounts of the *N*-acylpyridinone **SII-6b**. In this case, the amount is only about 5% (20:1 ratio by NMR).]

[5] a) A. McKillop, M. J. Zelesko, E. C. Taylor, *Tetrahedron Lett.* **1968**, *9*, 4945-4948. b) L. Li, Y. Zhao, R. Cao, L. Li, G. Cai, J. Li, X. Qi, S. Chen, Z. Zhang, *Chem. Commun.* **2019**, *55*, 4407-4410.



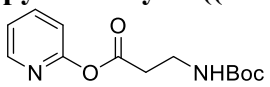
SII-6



SII-6b

IR: 2980, 1762, 1689, 1597, 1433, 1284, 1137, 1073, 721cm^{-1}
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.41 (dd, *J* = 5.0, 1.9 Hz, 1H), 7.89 (d, *J* = 1.8 Hz, 1H), 7.84 (dd, *J* = 8.1, 1.4 Hz, 2H), 7.81 – 7.73 (m, 2H), 7.69 (dt, *J* = 7.7, 1.5 Hz, 1H), 7.64 – 7.57 (m, 1H), 7.51 (dt, *J* = 10.3, 7.6 Hz, 3H), 7.26 – 7.21 (m, 1H), 7.00 (d, *J* = 8.1 Hz, 1H), 4.12 (q, *J* = 7.2 Hz, 1H), 1.70 (d, *J* = 7.1 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 196.4, 172.2, 157.9, 148.6, 134.0, 139.5, 138.1, 137.5, 132.5, 131.7, 130.1, 129.4, 129.3, 128.8, 128.3, 122.6, 116.2, 45.6, 18.5.
HR-MS (ESI) m/z calculated for $C_{21}H_{17}NO_3$ $[M+H]^+$ 332.1287, found 332.1276.

pyridin-2-yl 3-((tert-butoxycarbonyl)amino)propanoate (**SII-7**)



Following **GP-2A**, using 3-((tert-butoxycarbonyl)amino)propanoic acid (0.95 g, 5.0 mmol, 1.0 equiv), DMAP (0.06 g, 0.5 mmol, 0.1 equiv), DIC (0.86 mL, 5.5 mmol, 1.1 equiv) and 2-pyridinone (0.48 g, 5 mmol, 1.0 equiv) in 30 mL DCM for 12 h. The product was purified by flash silica gel chromatography (10-40% ethyl acetate/hexanes) to afford SII-7 as a colorless oil in 73% yield (0.97 g).
IR: 3362, 2989, 1762, 1590, 1426, 1280, 1143, 727 cm^{-1}
^{1}H NMR: (500 MHz, Chloroform-*d*) δ 8.42 (dd, *J* = 4.9, 2.0 Hz, 1H), 7.82 (td, *J* = 7.8, 2.0 Hz, 1H), 7.28 – 7.22 (m, 1H), 7.10 (d, *J* = 8.1 Hz, 1H), 5.12 (d, *J* = 6.5 Hz, 1H), 3.52 (q, *J* = 6.1 Hz, 2H), 2.86 (t, *J* = 5.9 Hz, 2H), 1.45 (s, 9H).
^{13}C NMR: (126 MHz, $CDCl_3$) δ 170.9, 157.6, 155.8, 148.6, 139.7, 122.3, 116.4, 79.5, 35.8, 34.9, 28.4.
HR-MS (ESI) m/z calculated for $C_{13}H_{18}N_2O_4$ $[M+Na]^+$ 289.1159, found 289.1156.

Preparation of 2-pyridyl thioesters
S-2-Pyridyl 4-phenylbutanoate was prepared according to our previous report.[6]

1.2 Methods

NMR Spectroscopy
^{1}H and ^{13}C NMR spectra were acquired on 400, 500, and 600 MHz Bruker NMR instruments. NMR chemical shifts are reported in ppm and are referenced to the residual solvent peak for $CDCl_3$ (δ = 7.26 ppm, ^{1}H NMR; δ = 77.16 ppm, ^{13}C NMR. Coupling constants (*J*) are reported in Hertz.

Gas Chromatography
GC analyses were performed on an Agilent 7890A GC equipped with dual DB-5 columns (20 m × 180 μm × 0.18 μm), dual FID detectors, and hydrogen as the carrier gas. A sample volume of 1

[6] For the preparation of *S*-2-pyridyl thioester (SII-3), see: J. Wang, B. P. Cary, P. D. Beyer, S. H. Gellman, D. J. Weix, *Angew. Chem., Int. Ed.* **2019**, *58*, 12081-12085; *Angew. Chem*. **2019**, *131*, 12209-12213.

μL was injected at a temperature of 300 °C and a 100:1 split ratio. The initial inlet pressure was 20.3 psi but varied as the column flow was held constant at 1.8 mL/min for the duration of the run. The initial oven temperature of 50 °C was held for 0.46 min followed by a temperature ramp of 65 °C/min up to 300 °C. The total run time was 5.0 min and the FID temperature was 325 °C.

GC/MS Analysis

GC/MS analyses were performed on a Shimadzu GCMS-QP2010 equipped with an RTX-5MS column (30 m × 0.25 mm × 0.25 μm) with a quadrupole mass analyzer using helium as the carrier gas. The analysis method used in all cases was 1 μL injection of sample, an injection temp of 250 °C, and a 20:1 split ratio. The initial inlet pressure was 8.1 psi, but varied as the column flow was held constant at 1.0 mL/min for the duration of the run. The interface temperature was held at 275 °C, and the ion source (EI^+, 30 eV) was held at 200 °C. The initial oven temperature was held at 60 °C for 1 min with the detector off, followed by a temperature ramp, with the detector on, to 300 °C at 20 °C/min. Total run time was 13.00 min.

Mass Spectrometry

High resolution mass spectra (HRMS) Mass spectrometry data was collected on a Thermo Q Exactive™ Plus (thermofisher.com) via flow injection with electrosprayionization or via ASAP-MS™ (asap -ms.com) by the chemistry mass spectrometry facility at the University of Wisconsin – Madison. The purchase of the Thermo Q Exactive Plus in 2015 was funded by NIH Award 1S10 OD020022 to the Department of Chemistry. The mass spectral data was also obtained at the University of Delaware mass spectrometry facility on a Q-Exactive Orbitrap.

Infrared Spectroscopy

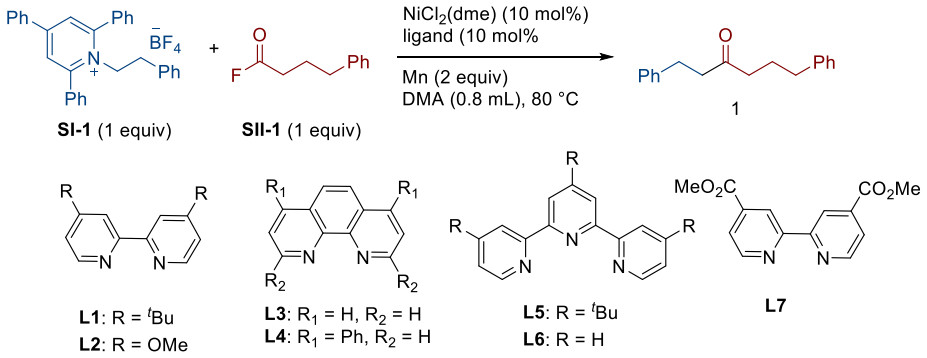
Infrared (IR) spectra were recorded on a Bruker Tensor 27 FT-IR and are reported in wavenumbers (cm^{-1}). Infrared (IR) spectra were also obtained on a Nicolet Magna 560 FT-IR spectrophotometer with material loaded onto a KBr plate.

Chromatography

Chromatography was performed on silica gel (EMD, silica gel 60, particle size 0.040-0.063 mm) using standard flash techniques, on a Teledyne Isco Rf-200 (detection at 210 nm and 280 nm), or on a Biotage Isolera One (detection at 210 nm and 400 nm or 254 nm and 280 nm, on KPsil columns or SNAPUltra columns respectively). Products were visualized by UV, PMA stain, or fractions were analyzed by GC.

2. Additional Optimization Tables

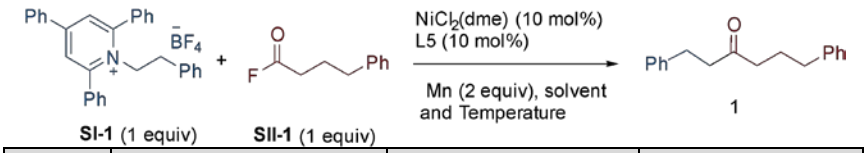
Table S2.1 Effect of Ligand on Reaction Yield for Coupling of Primary Alkyl Pyridinium Salts with Acyl Fluorides



Entry	Ligand	Yield of 1 (%)[a]
1	L1	10
2	L2	15
3	L3	31
4	L4	19
5	L5	52
6	L6	17
6	L7	<5

[a] GC yields vs 1,3,5-trimethoxybenzene standard; reaction run on a 0.125 mmol scale in 0.8 mL of solvent for 24 h.

Table S2.2 Solvent and Temperature Effect on Reaction Yield for the Coupling of Primary Alkyl Pyridinium Salts with Acyl Fluorides

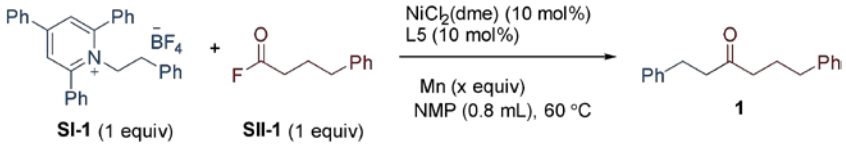


SI-1 (1 equiv) **SII-1** (1 equiv)

Entry	Solvent	Temperature (°C)	Yield of 1 (%)[a]
1	**DMA**	80	49
2	**THF**	80	24
3	**NMP**	80	55
4	**DMF**	80	65
5	**DMF**	40	18
6	**DMF**	60	72
7	**NMP**	60	84
8	**NMP with L7**	60	46
9	**DMA**	60	66

[a] GC yields vs 1,3,5-trimethoxybenzene standard; reaction run on a 0.125 mmol scale in 0.8 mL of solvent for 24 h.

Table S2.3 Influence of Reductant Stoichiometry on the Coupling of Primary Alkyl Pyridinium Salts with Acyl Fluorides

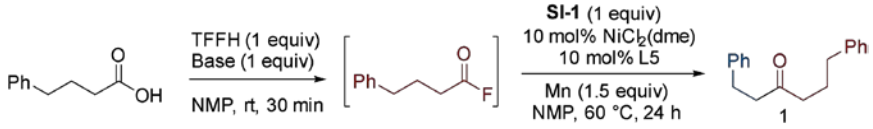


SI-1 (1 equiv) **SII-1** (1 equiv)

Entry	Equiv of Mn	Yield of 1 (%)[a]
1	1.1	82
2	1.5	82
3	1.8	76
4	2	78

[a] GC yields vs trimethoxybenzene standard; reaction run on a 0.125 mmol scale in 0.8 mL of solvent.

Table S2.4 Comparison of *in-situ* Acyl Fluoride Generation Methods for the Coupling of Primary Alkyl Pyridinium Salts



Entry	Base	Yield of Acyl Fluoride (%)[a]	Yield of 1 (%)[b]
1	Et_3N	96	31
2	iPr_2NEt	95	26
3	Proton Sponge	96	81

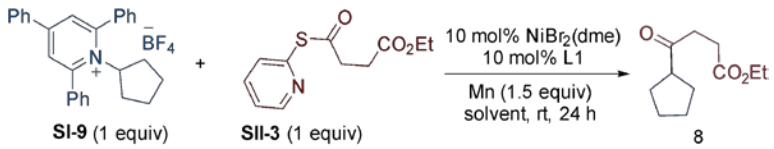
[a] ^{19}F NMR yield vs trifluorotoluene standard; reaction run on a 0.125 mmol scale in 0.8 mL of solvent. [b] GC yields vs 1,3,5-trimethoxybenzene standard. TFFH = Fluoro-N,N,N′,N′-bis(tetramethylene)formamidinium hexafluorophosphate; proton sponge = 1,8-bis(dimethylamino)naphthylene.

Commonly used organic bases, such as triethyl amine and diisopropyl ethyl amine, were effective for acyl fluoride generation, but interfered with the nickel-catalyzed acylation reaction (entries 1-2). 1,8-Bis(dimethylamino)naphthylene (proton sponge, entry 3) was effective for both steps. This inhibition of catalysis by triethylamine and diisopropyl ethyl amine or their salts has been observed by in some cross-electrophile coupling reactions,[7] but not others.[8]

[7] T. Koyanagi, A. Herath, A. Chong, M. Ratnikov, A. Valiere, J. Chang, V. Molteni, J. Loren, *Org. Lett*. **2019**, *21*, 816-820.

[8] a) L. K. G. Ackerman, L. L. Anka-Lufford, M. Naodovic, D. J. Weix, *Chem. Sci.* **2015**, *6*, 1115-1119. b) Y. Zhao, D. J. Weix, *J. Am. Chem. Soc.* **2014**, *136*, 48-51.

Table S2.5. Effect of Solvent Choice on the Coupling of Secondary Alkyl Pyridinium Salts with 2-Pyridyl Esters

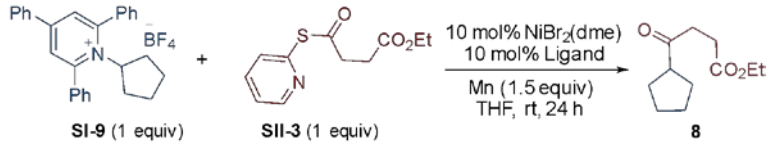


SI-9 (1 equiv) SII-3 (1 equiv) 8

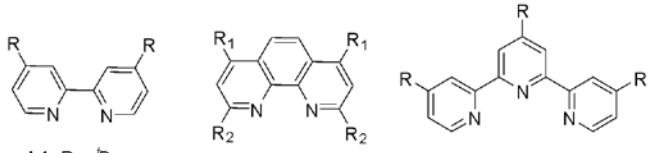
Entry	Solvent	Yield of 8 (%)[a]
1	DMA	10
2	DMF	Trace
3	NMP	6
4	THF	41
5	DMPU	none

[a] GC yields vs trimethoxybenzene standard; reaction run on a 0.125 mmol scale in 0.8 mL of solvent.

Table S2.6. Effect of Ligand Choice on Coupling of Secondary Alkyl Pyridinium Salts with *S*-2-Pyridyl Thioesters.



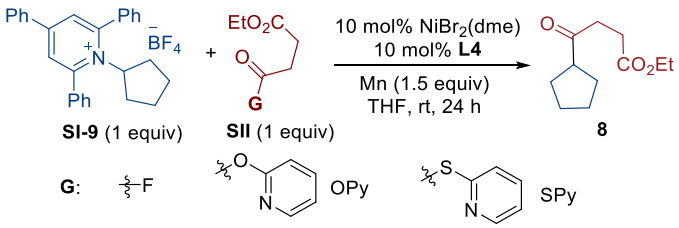
SI-9 (1 equiv) SII-3 (1 equiv) 8



Entry	Ligand	Yield of 1 (%)[a]
1	L1	35
2	L2	41
3	L3	33
4	L4	48
5	L5	33

[a] GC yields vs trimethoxybenzene standard; reaction run on a 0.125 mmol scale in 0.8 mL of solvent.

Table S2.7. Effect of Activating Group on Coupling of Secondary Alkyl Pyridinium Salts.



Entry	AG	Yield of 1 (%)[a]
1	F	Trace
2	SPy	46
3	OPy	66
4	OPy with 1.5 equiv of pyridinium salts	72

[a] GC yields vs trimethoxybenzene standard; reaction run on a 0.125 mmol scale in 0.8 mL of solvent for 24 h. **G** = Activating Group.

3. General Procedures

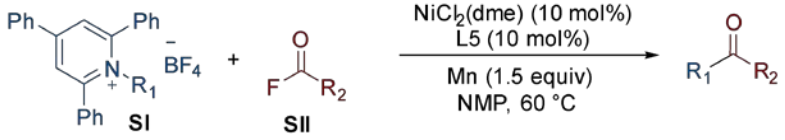
3.1 General Procedure for Reaction Optimization (GP-3)

For experimental convenience, reactions were set up in an N_2 filled glove box. An oven-dried 1-dram vial equipped with a PTFE-coated stir-bar was charged with $NiCl_2$(dme) (0.0125 mmol, 0.1 equiv), ligand (0.0125 mmol, 0.1 equiv) and 1,3,5-trimethoxybenzene (10 mg, 0.06 mmol, 0.48 equiv). The vial was then added to the listed solvent (0.8 mL) and the solution was allowed to stir for ~30 min. To the resulting solution was added the pyridinium salt (0.125 mmol, 1 equiv) and the carboxylic acid derivative (0.125 mmol, 1 equiv), followed by Mn powder (11 mg, 0.19 mmol, 1.5 equiv). The vial was then capped with a screw cap fitted with a PTFE-faced silicon septum, removed from the glovebox, and placed on a stir plate (1200 rpm) and allowed to stir for 24 h at listed temperature.

GC Analysis

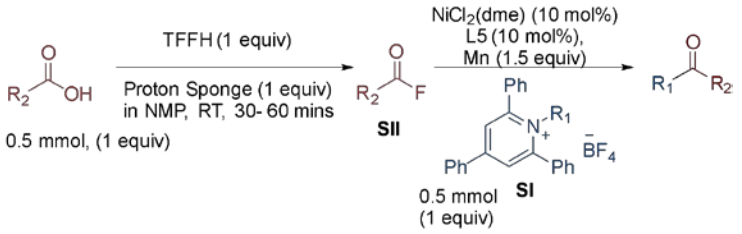
The reactions were monitored by GC analysis, by taking a 10 μL aliquot of the crude reaction mixture with a gas-tight syringe. The aliquot was diluted with ethyl acetate (0.50 mL), quenched with 200 μL H_2O, filtered through a 2-cm silica plug in a Pasteur pipette, and the filtrate was collected in a GC vial. The resulting solution was analyzed by GC and yields were determined based on the peak area of the analyte compared to the known 1,3,5-trimethoxybenzene internal standard.

3.2 General Procedure 4 (GP-4): Cross-Electrophile Coupling of Primary Alkyl Pyridinium Salts with Acyl Fluorides



For experimental convenience, reactions were set up in an N_2 filled glove box. An oven-dried 2-dram vial equipped with a PTFE-coated stir-bar was charged with $NiCl_2$(dme) (11 mg, 0.050 mmol, 0.10 equiv), **L5** (20 mg, 0.050 mmol, 0.10 equiv). NMP (3 mL) was added to the vial and the mixture was stirred for ~30 min, resulting in a blue solution. Pyridinium salt (0.50 mmol, 1.0 equiv) and acyl fluoride (0.50 mmol, 1.0 equiv) were then added to the vial, followed by the addition of Mn powder (41 mg, 0.75 mmol, 1.5 equiv). The vial was then capped with a screw cap fitted with a PTFE-faced silicon septum, removed from the glovebox, and placed on the stir plate (1200 rpm) and allowed to stir for 24 h at 60 °C. Upon reaction completion (judged by TLC, PMA stain), the mixture was filtered through a pad of celite (3 g), the celite pad was washed with ethyl acetate (3 × 5 mL), and the filtrate was concentrated under reduced pressure. The residue was diluted with ethyl acetate (30 mL) and washed with 5% aq NH_4OH (2 × 20 mL). The organic layer was washed with sat. $NaCl_{aq}$ (20 mL), dried over $MgSO_4$, and filtered through a fritted glass funnel to remove the drying agent. The filtrate was then concentrated in vacuo and purified by chromatography (SiO_2) to obtain the desired ketone product.

3.3 General Procedure 5 (GP-5): Cross-Electrophile Coupling of Primary Alkyl Pyridinium Salts with *in-situ*-Generated Acyl Fluorides



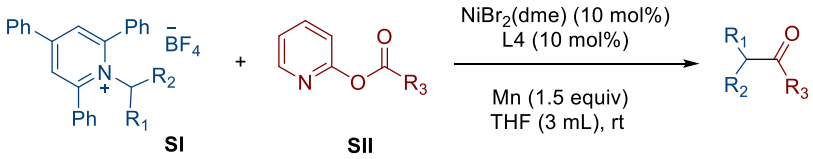
For experimental convenience, reactions were set up in an N_2 filled glove box. An oven-dried 2-dram vial equipped with a PTFE-coated stir-bar was charged with TFFH (132 mg, 0.50 mmol, 1 equiv), proton sponge (107 mg, 0.50 mmol, 1 equiv), NMP (1.0 mL) and carboxylic acid (0.50 mmol, 1.0 equiv). The reaction vial was then capped with a screw cap fitted with a PTFE-faced silicon septum, and the mixture was stirred for 30 to 60 min.

In parallel, a catalyst solution was prepared by adding $NiCl_2$(dme) (11 mg, 0.050 mmol, 0.10 equiv) and **L5** (20 mg, 0.05 mmol, 0.10 equiv) to a 1-dram vial equipped with a PTFE-coated stir bar. NMP (1mL) was then added and the mixture was stirred for 20 mins, resulting in a light green solution.

The nickel catalyst solution was then transferred by a 1 mL micropipette to the acyl fluoride solution, followed by the addition of additional NMP (1 mL). To this mixture was added pyridinium salt (0.50 mmol) and Mn powder (41 mg, 0.75 mmol, 1.5 equiv). The vial was then

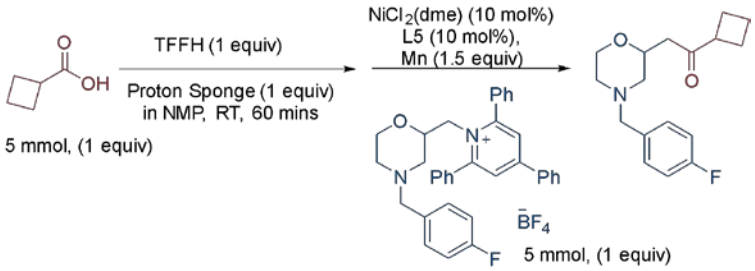
capped with a screw cap fitted with a PTFE-faced silicon septum, removed from the glovebox, and placed on a pre-heated (60 °C) stir plate (1200 rpm) and allowed to stir for 24 h. The mixture was filtered through a pad of celite (20 g), the celite pad was washed with ethyl acetate (3 × 5 mL), and the filtrate was concentrated under reduced pressure. The residue was diluted with ethyl acetate (30 mL) and washed with 5% aq NH_4OH (2 × 20 mL). The organic layer was further washed with sat. $NaCl_{aq}$ (20 mL), dried over $MgSO_4$, and filtered through a fritted glass funnel to remove the drying agent. The filtrate was again concentrated in vacuo and the residue was purified by chromatography (SiO_2) to obtain the desired ketone product.

3.4 General Procedure 6 (GP-6): Cross-Electrophile Coupling of Secondary Alkyl Pyridinium Salts with 2-Pyridyl Esters



For experimental convenience, reactions were set up in an N_2 filled glove box. An oven-dried 2-dram vial equipped with a PTFE-coated stir-bar was charged with $NiBr_2$(dme) (16 mg, 0.05 mmol, 0.1 equiv), **L4** (17 mg, 0.050 mmol, 0.1 equiv). THF (3 mL) was added to the vial and the mixture was stir for ~ 30 min, resulting in a green solution. Pyridinium salt (0.50-0.75 mmol, 1.0 - 1.5 equiv) and 2-pyridyl ester (0.50 mmol, 1.0 equiv) were then added to the vial, followed by the addition of Mn powder (41 mg, 0.75 mmol, 1.5 equiv). The vial was then capped with a screw cap fitted with a PTFE-faced silicon septum, removed from the glovebox, and placed on the stir plate (1200 rpm) and allowed to stir for 24 h at room temperature. The mixture was filtered through a pad of celite (20 g), the celite pad was washed with ethyl acetate (3 × 5 mL), and the filtrate was concentrated in vacuo and purified by chromatography (SiO_2) to obtain the desired ketone product.

3.5 Preparative-Scale Benchtop Procedure



A catalyst solution was prepared on the benchtop by charging a 20-mL scintillation vial with a PTFE-coated stir bar, $NiCl_2$•dme (110 mg, 0.5 mmol, 0.1 equiv), **L5** (200 mg, 0.5 mmol, 0.1 equiv). The scintillation vial was capped with a rubber septum and evacuated and backfilled with N_2 for three times via a needle connected to a vacuum line. Air-free, anhydrous NMP (10 mL) was added to the scintillation vial a gas tight syringe. The mixture was stirred at rt for 20 min, resulting in a homogeneous, green solution.

To a second 20-mL scintillation vial equipped with a PTFE-coated stir bar was added pyridinium salt SI-8 (3.01 g, 5.0 mmol, 1 equiv) and NMP (10 mL). The mixture was stirred until homogeneous.

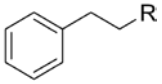
An N_2-flushed 100-mL Schlenk flask was charged with TFFH (1.32 g, 5.0 mmol, 1 equiv), proton sponge (1.07 g, 5.0 mmol, 1 equiv), NMP (10 mL) and cyclobutanecarboxylic acid (500.6 mg, 5.0 mmol, 1 equiv) under a positive N_2 flow and stoppered with a rubber stopper. The reaction mixture was then stirred at rt for 1 h to ensure complete formation of the acyl fluoride. The catalyst solution and the pyridinium salt solution were then transferred to the Schlenk flask containing the acid fluoride via gas tight syringes. Mn powder (410 mg, 7.5 mmol) was then added to the Schlenk flask in one portion under a positive nitrogen flow, the flask was re-capped, and the flask was placed into a pre-heated 60 °C oil bath to stir (1200 RPM) for 24 h.

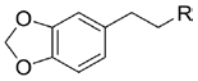
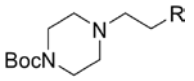
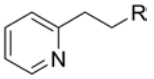
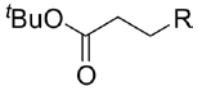
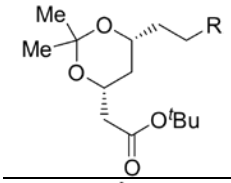
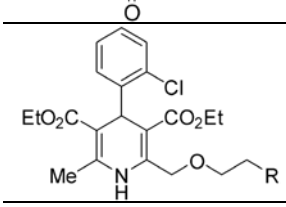
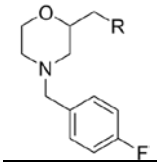
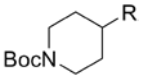
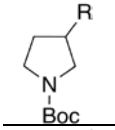
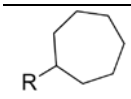
Isolation and Purification

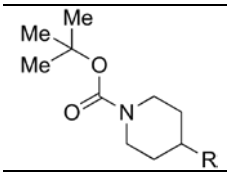
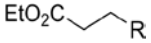
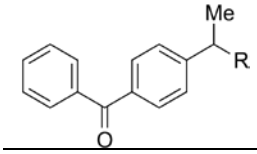
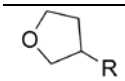
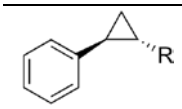
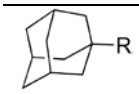
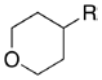
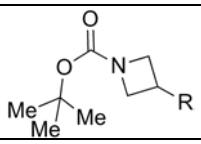
After 24 h, the Schlenk flask was removed from the oil bath and cooled to rt. The mixture was filtered through a pad of celite (200 g), the celite pad was washed with ethyl acetate (3 × 50 mL), and the resulting solution was concentrated under reduced pressure. The crude mixture was diluted with ethyl acetate (100 mL) and washed with 5% aq NH_4OH (2 × 60 mL). The organic layer was washed with sat. $NaCl_{aq}$ (100 mL), dried over $MgSO_4$, and filtered through a fritted glass funnel to remove the drying agent. The filtrate was then concentrated in vacuo and the residue was purified by chromatography (10-50% ethyl acetate in hexanes) to afford 0.743 g (51% yield) of the desired product as light yellow oil. Characterization was consistent with the material synthesized on small scale (**19** in **Section 4**).

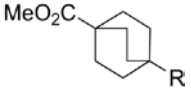
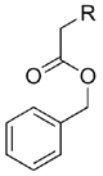
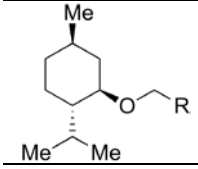
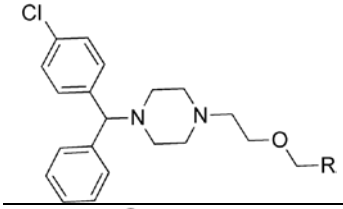
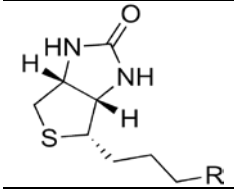
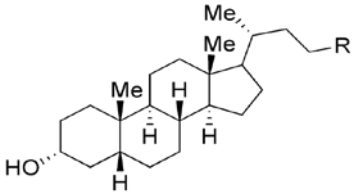
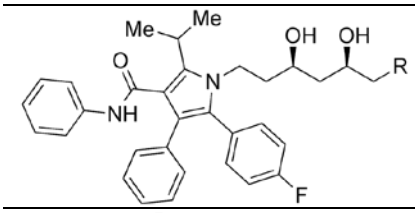
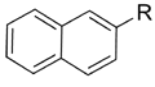
4. Analysis of Substrate Availability

We conducted a retrospective comparison of our alkyl fragments, comparing amines, bromides, and carboxylic acid derivatives of all the alkyls used in this study. In each case, the least expensive option is highlighted in blue, with the supplier noted in parentheses. In more than half of the examples, the amine is the least expensive. There are several cases that amines are affordable while the corresponding bromide and carboxylic acid precursors are either not commercially available or not reported in literature. Meanwhile, we have noticed a chemical space difference between amines and carboxylic acids; this difference is particularly significant in pharmaceutical intermediates. In our substrate scope, there are three amine-derived drug intermediates where their corresponding carboxylic acids fragments are not commercially available. Also, there are six carboxylic acid derived natural products and drug intermediates, which are readily available with reasonable price, where their corresponding amine fragments are not available. Hence, in these cases, the ketone synthesis can be challenging without readily available starting materials.

compound	**amine** ($R = NH_2$)	**bromide** ($R = Br$)	**carboxylic acid** ($R = CO_2H$)
<structure of alkyl fragment>	bottle size/price (supplier) [CAS#]		
	50 mL/$30.6 (SigmaAldrich) [64-04-0]	5g/$10 100g/$40 (Combi-Blocks) [103-63-9]	5g/$10 100g/$40 (SigmaAldrich) [501-52-0]

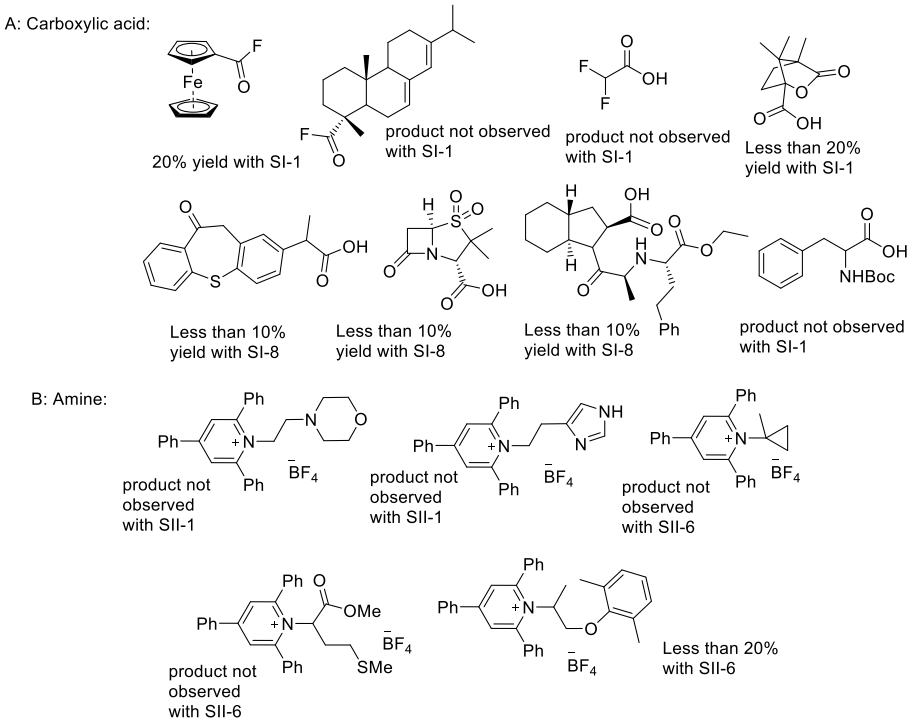
	1g/$10 100g/$245 (Combi-Blocks) [1484-85-1]	custom syntheses available [57587-02-7]	1g/$25 100g/$840 (Combi-Blocks) [2815-95-4]
	1g/$20 25g/$260 (Combi-Blocks) [192130-34-0]	100mg/$88 5g/$700 (Combi-Blocks) [655225-01-7]	1g/$50 25g/$720 (Combi-Blocks) [242459-97-8]
	1g/$10 100g/$285 (Combi-Blocks) [2706-56-1]	1g/$30 100g/$850 (Combi-Blocks) [72996-65-7] (HBr)	1g/$84 25g/$920 (Combi-Blocks) [15197-75-8]
	HCl Salt 5 g/$20 500 g/$960 (Combi-Blocks) [58620-93-2]	1 g/$15 50 g/$550 (Combi-Blocks) [55666-43-8]	1 g/$10 500 g/$960 (Combi-Blocks) [15026-17-2]
	1g/$10 100g/$200 (Combi-Blocks) [125995-13-3]	one patent ref [1380521-19-6]	unknown compound
	1g/$84 5g/$264 (AK Scientfic) [88150-42-9]	unknown compound (including PG variants)	unknown compound (including PG variants)
	1g/$14 25g/$114 (Combi-Blocks) [112914-13-3]	custom syntheses available [1508657-04-2]	custom syntheses available [763903-53-3]
	1g/$10 100g/$125 (Combi-Blocks) [87120-72-7]	1g/$13 4×25g/$420 (Combi-Blocks) [180695-79-8]	5g/$10 500g/$180 (Combi-Blocks) [84358-13-4]
	100 mL/$16 2500 mL/$61 (Alfa Aesar) [75-31-0]	250 mL/$47 2500 mL/$347 (Alfa Aesar) [75-26-3]	100 mL/$21 2500 mL/$82 (Alfa Aesar) [79-31-2]
	1g/$13 4x25g/$370 (Combi-Blocks) [186550-13-0]	1g/$50 4x25g/$1250 (Combi-Blocks) [939793-16-5]	1g/$10 4x25g/$295 (Combi-Blocks) [59378-75-5]
	1g/$14 100g/$107 (Combi-Blocks) [1003-03-8]	1g/$14 100g/$30 (Oakwood) [137-43-9]	1g/$14 100g/$187 (Oakwood) [3400-45-1]
	1g/$14 100g/$234 (Combi-Blocks) [5452-35-7]	1g/$21 100g/$494 (Combi-Blocks) [2404-35-5]	1g/$27 100g/$721 (Combi-Blocks) [1460-16-8]

	25g/$47 500g/$701 (Combi-Blocks) [87120-72-7]	5g/$34 100g/$561 (Combi-Blocks) [180695-79-8]	5g/$14 500g/$241 (Combi-Blocks) [84358-13-4]
	HCl Salt 5 g/$14 25 g/$21 100 g/$54 500 g/$201 (Combi-Blocks) [4244-84-2]	5g/$14 25g/$21 100g/$66 (Combi-Blocks) [539-74-2]	1g/$14 5g/$34 25g/$107 100g/$321 (Combi-Blocks) [1070-34-4]
	custom syntheses available [482620-67-7]	custom syntheses available [79316-80-6]	25g/$229 (TCI America) [22071-15-4]
	5g/$14 25g/$17 100g/$30 (Oakwood) [765-30-0]	5g/$27 25g/$54 100g/$161 (Combi-Blocks) [4333-56-6]	5g/$15 25g/$21 100g/$47 (Oakwood) [1759-53-1]
	1g/$201 5g/$801 (Combi-Blocks) [88675-24-5]	1g/$101 25g/$1,201 (Combi-Blocks) [19311-37-6]	1g/$14 100g/$481 (Combi-Blocks) [89364-31-8]
	1g/$200 5g/$799 (Aurum Pharmatech) [3721-28-6]	custom syntheses available [32523-76-5]	5g/$97 10g/$181 (Pharmablock) [23020-15-7]
	1g/$34 5g/$115 25g/$346 100g/$1,002 (Oakwood) [2516-34-9]	1g/$21 5g/$54 10g/$94 25g/$147 (Oakwood) [4399-47-7]	1g/$14 5g/$18 25g/$34 100g/$101 (Oakwood) [3721-95-7]
	5g/$37.20 25g/$175 100g/$479 (Sigma) [768-94-5]	5g/$14 25g/$27 100g/$67 (Combi-Blocks) [768-90-1]	1g/$14 5g/$17 25g/$27 100g/$54 (Oakwood) [828-51-3]
	1g/$10 5g/$25 25g/$80 100g/$240 (Combi-Blocks) [33024-60-1] (HCl)	1g/$17 5g/$54 10g/$94 25g/$194 (Combi-Blocks) [25637-16-5]	1g/$10 5g/$12 25g/$40 100g/$120 (Combi-Blocks) [5337-03-1]
	1g/$14 5g/$54 10g/$94 25g/$187	1g/$81 5g/$241 10g/$387 25g/$907	1g/$14 5g/$27 25g/$94 100g/$301

	(Combi-Blocks) [193269-78-2]	(Combi-Blocks) [1064194-10-0]	(Combi-Blocks) [142253-55-2]
	250mg/$127 1g/$294 5g/$1,014 10g/$1,414 (Combi-Blocks) [135908-33-7]	1g/$478 5g/$1,435 (Pharmablock) [23062-51-3]	1g/$47 5g/$141 10g/$241 25g/$441 (Combi-Blocks) [18720-35-9]
	1g/$136 5g/$264 (Aurum Pharmatech) [1738-68-7]	5g/$14 25g/$19 100g/$51 500g/$167 (Oakwood) [5437-45-6]	1g/$41 5g/$121 10g/$207 25g/$421 (Combi-Blocks) [103-40-2]
	Not Available	Not Available	1g/$41 5g/$107 25g/$321 (Combi-Blocks) [40248-63-3]
	Not Available	Not Available	1g/$41 5g/$107 10g/$201 25g/$334 (Combi-Blocks) [83881-52-1]
	Not Available	Not Available	1g/$14 5g/$21 25g/$67 100g/$214 (Combi-Blocks) [58-85-5]
	Not Available	Not Available	1g/$14 5g/$27 25g/$101 100g/$201 (Combi-Blocks) [434-13-9]
	Not Available	Not Available	250mg/$46 1g/$90 5g/$274 (Oakwood) [134523-00-5]
	1g/$29 5g/$117 10g/$199 25g/$393 (Oakwood) [91-59-8]	25g/$27 100g/$81 (Combi-Blocks) [580-13-2]	1g/$13 5g/$14 25g/$33 100g/$79 (Oakwood) [93-09-4]

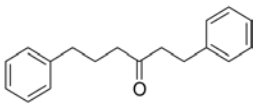
5 Table S5 Low Yielding Substrates

Substrates that were tested, but provided yields ≤20%, are listed below. In particular, more hindered tertiary carboxylic acids and pyridinium salts that could β-eliminate or chelate were more problematic. Few triphenylpyridinium salts of tertiary carbinamines can be synthesized due to steric hinderance. We did not observe product in a test reaction with one of the few accessible ones derived from 1-methylcyclopropylamine.



6. Specific Procedures and Product Characterization

1,6-diphenylhexan-3-one (1)

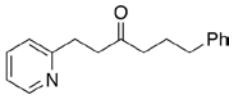


Following GP-4, pyridinium salt SI-1 (250 mg, 0.50 mmol, 1.0 equiv) and 4-phenylbutanoyl fluoride, SII-1, (83 mg, 0.50 mmol, 1.0 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (50:1 hexanes/ ethyl acetate) to afford 0.103 g (82%) of the desired product as light yellow oil.

Alternatively, the product could also be prepared following GP-5. Pyridinium salt SI-1 (250 mg, 0.50 mmol, 1.0 equiv) and 4-phenylbutanoic acid (82 mg, 0.50 mmol, 1.0 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (50:1 hexanes/ ethyl acetate) to afford 0.081 g (64%) of the desired product as light yellow oil.

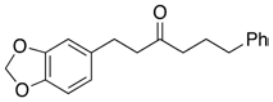
IR 2929, 1710, 1495, 1452, 1029, 696 cm^{-1}
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 – 7.23 (m, 4H), 7.21 – 7.07 (m, 6H), 2.88 (t, *J* = 7.7 Hz, 2H), 2.69 (t, *J* = 7.6 Hz, 2H), 2.59 (t, *J* = 7.6 Hz, 2H), 2.38 (t, *J* = 7.3 Hz, 2H), 1.89 (p, *J* = 7.4 Hz, 2H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 209.8, 141.6, 141.1, 128.48, 128.46, 128.4, 128.3, 126.1, 125.9, 44.3, 42.1, 35.1, 29.8, 25.1.
HRMS (ESI+) m/z calcd for $C_{18}H_{20}O$ $[M+Na]^+$ 275.1406, found 275.1402.

6-phenyl-1-(pyridin-2-yl)hexan-3-one (2)



Following GP-5, pyridinium salt SI-4 (250 mg, 0.5 mmol, 1.0 equiv) and 4-phenylbutanoic acid (82 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (20-75% diethyl ether/hexane) to afford 0.057 g (45%) of the desired product as light yellow oil. The product was contaminated with diethyl ether and acetone and the reported yield was adjusted based upon ^{1}H NMR. This unusual reporting method was necessitated by the closure of our labs due to the COVID-19 pandemic.
IR 2925, 1717, 1653, 1558, 1450, 1012, 700 cm^{-1}
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46 (d, *J* = 4.9 Hz, 1H), 7.55 (td, *J* = 7.6, 1.9 Hz, 1H), 7.26 (d, *J* = 7.4 Hz, 2H), 7.18 – 7.11 (m, 4H), 7.09 – 7.04 (m, 1H), 3.04 (t, *J* = 7.2 Hz, 2H), 2.87 (t, *J* = 7.2 Hz, 2H), 2.58 (t, *J* = 7.6 Hz, 2H), 2.43 (t, *J* = 7.3 Hz, 2H), 1.89 (p, *J* = 7.4 Hz, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 209.9, 160.5, 149.2, 141.6, 136.3, 128.5, 125.9, 123.2, 121.2, 100.0, 42.1, 41.6, 35.1, 31.7, 25.3.
HRMS (ESI) m/z calcd for $C_{17}H_{19}NO$ $[M+H]^+$ 254.1545, found 254.1536.

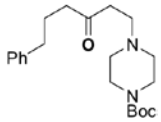
1-(benzo[*d*][1,3]dioxol-5-yl)-6-phenylhexan-3-one (3)



Following GP-5, pyridinium salt SI-2 (272 mg, 0.5 mmol, 1.0 equiv) and 4-phenylbutanoic acid (82 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (5-20% ethyl acetate/hexane) to afford 0.111 g (75%) of the desired product as yellow oil.

IR 2923, 1712, 1502, 1489, 1443, 1245, 1039, 928, 700 cm^{-1}
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.22 – 7.18 (m, 2H), 7.13 – 7.09 (m, 1H), 7.08 – 7.05 (m, 2H), 6.63 (d, *J* = 7.9 Hz, 1H), 6.58 (d, *J* = 1.7 Hz, 1H), 6.53 (dt, *J* = 8.0, 1.2 Hz, 1H), 5.83 (s, 2H), 2.72 (t, *J* = 7.5 Hz, 2H), 2.57 (dd, *J* = 8.1, 7.1 Hz, 2H), 2.52 (t, *J* = 7.6 Hz, 2H), 2.31 (t, *J* = 7.3 Hz, 2H), 1.87 – 1.77 (m, 2H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 209.8, 147.6, 145.8, 141.6, 134.9, 128.5, 128.4, 126.0, 121.1, 108.8, 108.2, 100.8, 44.6, 42.2, 35.1, 29.5, 25.1.
HRMS (ESI) m/z calcd for $C_{19}H_{20}O_3$ $[M+H]^+$ 297.1491, found 297.1483.

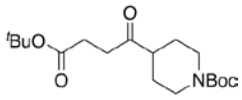
***tert*-butyl 4-(3-oxo-6-phenylhexyl)piperazine-1-carboxylate (4)**



Following GP-4, pyridinium salt SI-3 (303 mg, 0.5 mmol, 1.0 equiv) and 4-phenylbutanoyl fluoride, SII-1, (83 mg, 0.50 mmol, 1.0 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (20-50% diethyl ether/hexane) to afford 0.076 g (42%) of the desired product as yellow oil.

IR 2927, 1687, 1495, 1029, 694 cm^{-1}
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 – 7.25 (m, 2H), 7.24 – 7.11 (m, 3H), 3.39 (t, *J* = 5.0 Hz, 4H), 2.63 (q, *J* = 8.0 Hz, 4H), 2.56 (t, *J* = 6.7 Hz, 2H), 2.44 (t, *J* = 7.3 Hz, 2H), 2.35 (t, *J* = 5.0 Hz, 4H), 1.92 (p, *J* = 7.4 Hz, 2H), 1.45 (s, 9H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 209.5, 154.7, 141.5, 128.5, 128.4, 126.0, 79.7, 52.9, 52.8, 43.6, 42.2, 40.3, 35.1, 28.4, 25.1.
HRMS (ESI) m/z calcd for $C_{21}H_{32}N_2O_3$ $[M+H]^+$ 361.2486, found 361. 2484.

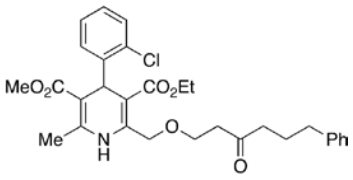
***tert*-butyl 4-(4-(*tert*-butoxy)-4-oxobutanoyl)piperidine-1-carboxylate (5)**



Following GP-5, pyridinium salt SI-5 (262 mg, 0.5 mmol, 1.0 equiv) and 1-(tert-butoxycarbonyl)piperidine-4-carboxylic acid (115 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (20-75% diethyl ether/hexane) to afford 0.0713 g (55%) of the desired product as light yellow oil.

IR 2976, 1695, 1422, 1366, 1238, 1159, cm^{-1}
^{1}H NMR (600 MHz, $CDCl_3$) δ 4.09 (s, 2H), 2.76 (d, *J* = 13.9 Hz, 2H), 2.69 (t, *J* = 6.5 Hz, 2H), 2.53 – 2.48 (m, 3H), 1.83 (d, *J* = 13.3 Hz, 2H), 1.59 – 1.51 (m, 2H), 1.44 (s, 9H), 1.42 (s, 9H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 210.4, 172.0, 154.7, 80.6, 79.6, 48.5, 43.1, 35.2, 29.1, 28.4, 28.1, 27.5.
HRMS (ESI) m/z calcd for $C_{18}H_{31}NO_5$ $[M+H]^+$ 342.2281, found 342.2269

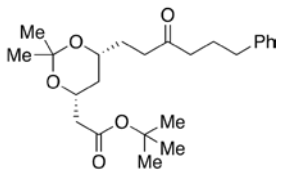
3-ethyl 5-methyl 4-(2-chlorophenyl)-6-methyl-2-(((3-oxo-6-phenylhexyl)oxy)methyl)-1,4-dihydropyridine-3,5-dicarboxylate (6)



Following GP-5, pyridinium salt SI-7 (787 mg, 0.5 mmol, 1 equiv) and 4-phenylbutanoic acid (82 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (20-60% diethyl ether/hexane) to afford 0.135 g (50%) of the desired product as yellow oil. The product contains ethyl acetate and a small amount of an amlodipine-derived side product, presumably hydrodeaminated amlodipine, based upon analysis of the NMR spectra and comparisons with literature reports.[2b,9] The reported yield is adjusted for these impurities. This unusual reporting method was necessitated by the closure of our labs due to the COVID-19 pandemic.

IR 3321, 2946, 1695, 1485, 1280, 1207, 1098, 700 cm^{-1}
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (s, 1H), 7.42 (dd, *J* = 7.8, 1.7 Hz, 1H), 7.32 (t, *J* = 7.4 Hz, 2H), 7.27 – 7.22 (m, 2H), 7.22 – 7.18 (m, 2H), 7.14 (td, *J* = 7.5, 1.4 Hz, 1H), 7.05 (td, *J* = 7.6, 1.8 Hz, 1H), 5.44 (d, *J* = 7.8 Hz, 1H), 4.83 – 4.67 (m, 2H), 4.06 (dtq, *J* = 9.6, 6.3, 3.3, 2.5 Hz, 2H), 3.73 (tt, *J* = 9.6, 4.7 Hz, 2H), 3.64 (d, *J* = 2.7 Hz, 3H), 2.77 (t, *J* = 5.2 Hz, 2H), 2.67 (dd, *J* = 9.2, 5.7 Hz, 2H), 2.53 (s, 3H), 2.52 – 2.43 (m, 2H), 1.99 (p, *J* = 7.4 Hz, 2H), 1.21 (q, *J* = 6.9 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 209.4, 168.2, 167.4, 146.3, 146.1, 145.2, 141.4, 132.3, 131.6, 129.2, 128.49, 128.48, 127.3, 126.9, 126.1, 103.8, 101.1, 68.7, 65.8, 59.7, 50.8, 42.3, 42.2, 37.2, 35.1, 25.2, 19.0, 14.3.
HRMS (ESI) m/z calcd for $C_{30}H_{34}ClNO_6$ $[M+H]^+$ 540.2153, found 540.2147.

***tert*-butyl 2-((4*R*,6*R*)-2,2-dimethyl-6-(3-oxo-6-phenylhexyl)-1,3-dioxan-4-yl)acetate (7)**

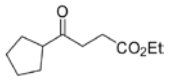


Following GP-5, pyridinium salt SI-6 (652 mg, 0.5 mmol, 1 equiv) and 4-phenylbutanoic acid (82 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (20-60% diethyl ether/hexane) to afford 0.103 g (64%) of the desired product as yellow oil.

IR 2937, 1729, 1368, 1200, 1154, 950, 700 cm^{-1}
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.21 (t, *J* = 7.6 Hz, 2H), 7.13 – 7.07 (m, 3H), 4.15 (dtd, *J* = 11.7, 6.6, 2.5 Hz, 1H), 3.78 – 3.70 (m, 1H), 2.54 (t, *J* = 7.6 Hz, 2H), 2.43 – 2.31 (m, 4H), 2.23 – 2.19 (m, 1H), 1.83 (p, *J* = 7.5 Hz, 2H), 1.70 (dtd, *J* = 14.8, 7.5, 3.8 Hz, 1H), 1.57 (dtd, *J* = 14.1, 8.1, 6.0 Hz, 1H), 1.46 (dt, *J* = 12.7, 2.4 Hz, 1H), 1.37 (s, 9H), 1.35 – 1.33 (m, 2H), 1.32 (s, 3H), 1.26 (s, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 210.5, 170.2, 141.6, 128.5, 128.4, 125.9, 98.7, 80.6, 67.9, 66.2, 42.7, 42.7, 42.0, 38.2, 36.5, 35.1, 30.1, 28.1, 25.3, 19.7.
HRMS (ESI) m/z calcd for $C_{24}H_{36}O_5$ $[M+H]^+$ 405.2641, found 405.2631.

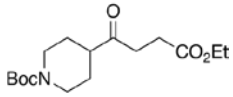
[9] A. S. Lakshmi Devi, Y. Srinivasa Rao, M. Satish, G. Jyothi, K. Babu Rao, T. Omdutt, *Magn. Reson. Chem.* **2007**, *45*, 688-691.

ethyl 4-cyclopentyl-4-oxobutanoate (8)



Following GP-6, pyridinium salt SI-9 (347 mg, 0.75 mmol, 1.5 equiv) and SII-5 (112 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (0-5% diethyl ether/hexane) to afford 0.070 g (71%) of the desired product as light yellow oil. The spectra data matches the previous report.[10]
^{1}H NMR (500 MHz, $CDCl_3$) δ 4.13 (qd, J = 7.1, 2.3 Hz, 2H), 2.91 (pd, J = 8.2, 7.7, 1.8 Hz, 1H), 2.78 (td, J = 6.6, 2.0 Hz, 2H), 2.58 (td, J = 6.6, 2.3 Hz, 2H), 1.89 – 1.72 (m, 4H), 1.72 – 1.53 (m, 4H), 1.26 (td, J = 7.2, 2.3 Hz, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 211.2, 172.9, 60.6, 51.3, 36.1, 28.9, 28.1, 26.0, 14.2.

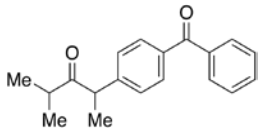
***tert*-butyl 4-(4-ethoxy-4-oxobutanoyl)piperidine-1-carboxylate (9)**



Following GP-6, pyridinium salt SI-10 (434 mg, 0.75 mmol, 1.5 equiv) and SII-5 (112 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (20-50% diethyl ether/hexane) to afford 0.091 g (58%) of the desired product as light yellow oil.

IR 2926, 1736, 1692, 1423, 1366, 1170, 1133, 1023, 859, 770 cm^{-1}
^{1}H NMR (600 MHz, $CDCl_3$) δ 4.09 (q, *J* = 7.2 Hz, 4H), 2.77 (d, *J* = 14.7 Hz, 2H), 2.72 (t, *J* = 6.5 Hz, 2H), 2.56 (t, *J* = 6.5 Hz, 2H), 2.49 (tt, *J* = 11.4, 3.7 Hz, 1H), 1.81 (d, *J* = 13.2 Hz, 2H), 1.52 (dtd, *J* = 13.4, 11.7, 4.3 Hz, 2H), 1.42 (s, 9H), 1.22 (t, *J* = 7.1 Hz, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 210.3, 172.7, 154.7, 79.6, 60.7, 48.6, 43.2, 35.0, 28.44, 28.43, 27.9, 27.5, 14.2.
HRMS (ESI) m/z calcd for $C_{16}H_{27}NO_5$ $[M+H]^+$ 314.1967, found 314.1955.

2-(4-benzoylphenyl)-4-methylpentan-3-one (10)

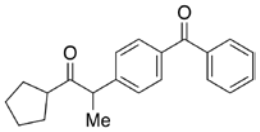


Following GP-6, pyridinium salt SI-12 (328 mg, 0.75 mmol, 1.5 equiv) and SII-6 (166 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (5-50% diethyl ether/hexane) to afford 0.086 g (62%) of the desired product as colorless oil.

[10] J. Wang, B. P. Cary, P. D. Beyer, S. H. Gellman, D. J. Weix, *Angew. Chem. Int. Ed*. **2019**, *58*, 12081-12085.

IR 2971, 1712, 1660, 1448, 1281, 1016, 720 cm^{-1}
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 – 7.77 (m, 2H), 7.70 – 7.64 (m, 2H), 7.62 – 7.57 (m, 1H), 7.52 – 7.40 (m, 4H), 4.02 (q, *J* = 6.9 Hz, 1H), 2.71 (p, *J* = 6.9 Hz, 1H), 1.41 (d, *J* = 7.0 Hz, 3H), 1.09 (d, *J* = 7.0 Hz, 3H), 0.95 (d, *J* = 6.7 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 214.2, 196.5, 141.1, 138.1, 137.4, 132.6, 131.8, 130.1, 129.6, 129.0, 128.8, 128.3, 50.8, 39.5, 19.1, 18.30, 18.26.
HRMS (ESI) m/z calcd for $C_{19}H_{20}O_2$ $[M+H]^+$ 281.1542, found 281.1536.

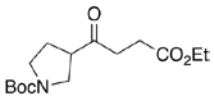
2-(4-benzoylphenyl)-1-cyclopentylpropan-1-one (11)



Following GP-6, pyridinium salt SI-9 (347 mg, 0.75 mmol, 1.5 equiv) and SII-6 (166 mg, 0.5 mmol, 1 equiv)were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (2-20% diethyl ether/hexane) to afford 0.099 g (65%) of the desired product as yellow oil.

IR 2957, 1708, 1660, 1448, 1317, 1281, 720 cm^{-1}
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 – 7.77 (m, 2H), 7.71 – 7.65 (m, 2H), 7.62 – 7.57 (m, 1H), 7.52 – 7.41 (m, 4H), 3.95 (q, *J* = 6.9 Hz, 1H), 2.98 – 2.85 (m, 1H), 1.89 – 1.80 (m, 1H), 1.73 – 1.59 (m, 4H), 1.54 – 1.47 (m, 2H), 1.42 (d, *J* = 7.0 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 213.0, 196.6, 141.2, 138.1, 137.4, 132.6, 131.8, 130.1, 129.7, 129.0, 128.8, 128.3, 52.2, 50.1, 30.3, 29.1, 26.09, 18.1.
HRMS (ESI) m/z calcd for $C_{21}H_{22}O_2$ $[M+H]^+$ 307.1698, found 307.1691.

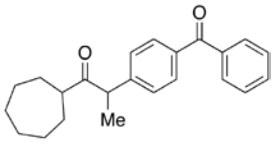
***tert*-butyl (*R*)-3-(4-ethoxy-4-oxobutanoyl)pyrrolidine-1-carboxylate (12)**



Following GP-6, pyridinium salt SI-11 (423 mg, 0.75 mmol, 1.5 equiv) and SII-5 (112 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (20-50% diethyl ether/hexane) to afford 0.099 g (55%) of the desired product as colorless oil.

IR 2924, 1696, 1407, 1166, 878, 772 cm^{-1}
^{1}H NMR (400 MHz, DMSO-d_6, VT NMR conducted at 100 °C) δ 4.09 (p, *J* = 7.2 Hz, 3H), 3.52 – 3.37 (m, 2H), 3.35 – 3.21 (m, 3H), 2.87 – 2.71 (m, 2H), 2.53 (d, *J* = 6.7 Hz, 1H), 2.16 – 2.03 (m, 1H), 1.96 (dq, *J* = 12.6, 7.6 Hz, 1H), 1.43 (d, *J* = 1.2 Hz, 9H), 1.21 (td, *J* = 7.1, 4.4 Hz, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 207.7, 207.6, 172.6, 172.3, 154.4, 79.6, 60.7, 50.3, 49.4, 47.0, 45.4, 45.2, 36.1, 30.3, 29.7, 29.1, 28.9, 28.5, 28.2, 27.9, 27.8, 14.14, 14.12.
HRMS (ESI) m/z calcd for $C_{15}H_{25}NO_5$ $[M+H]^+$ 300.1811, found 300.1800.

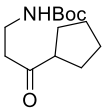
2-(4-benzoylphenyl)-1-cycloheptylpropan-1-one (13)



Following GP-6, pyridinium salt SI-13 (369 mg, 0.75 mmol, 1.5 equiv) and SII-6 (166 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (5-25% diethyl ether/hexane) to afford 0.093 g (56%) of the desired product as colorless oil.

IR 2925, 2853, 1710, 1660, 1597, 1447, 1317, 1281, 719 cm^{-1}
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 – 7.76 (m, 2H), 7.71 – 7.65 (m, 2H), 7.64 – 7.56 (m, 1H), 7.53 – 7.39 (m, 4H), 4.00 (q, *J* = 7.0 Hz, 1H), 2.62 (tt, *J* = 9.2, 4.4 Hz, 1H), 1.83 (ddq, *J* = 12.2, 5.4, 3.2, 2.7 Hz, 1H), 1.76 – 1.45 (m, 10H), 1.41 (d, *J* = 7.0 Hz, 3H), 1.30 (td, *J* = 8.8, 4.6 Hz, 1H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 213.7, 196.6, 141.1, 138.1, 137.5, 132.6, 131.8, 130.1, 129.7, 128.9, 128.8, 128.3, 51.1, 51.1, 30.5, 29.8, 28.3, 28.3, 26.7, 26.4, 18.5.
HRMS (ESI) m/z calcd for $C_{23}H_{26}O_2$ $[M+H]^+$ 335.2011, found 335.2002.

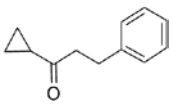
tert-butyl (3-cyclopentyl-3-oxopropyl)carbamate (14)



Following GP-6, pyridinium salt SI-9 (347 mg, 0.75 mmol, 1.5 equiv) and SII-7 (133 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (0-20% ethyl acetate/hexane) to afford 0.089 g (74%) of the desired product as colorless oil.

IR 3377, 2959, 2870, 1698. 1502, 1452, 1365, 1248, 1166 cm^{-1}
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.04 (s, 1H), 3.36 (q, *J* = 6.0 Hz, 2H), 2.85 (p, *J* = 8.0 Hz, 1H), 2.69 (t, *J* = 5.7 Hz, 2H), 1.87 – 1.76 (m, 2H), 1.76 – 1.52 (m, 6H), 1.42 (s, 9H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 212.9, 155.9, 79.2, 51.5, 41.4, 35.3, 28.7, 28.4, 26.0.
HRMS (ESI) m/z calcd for $C_{13}H_{23}NO_3$ $[M+Na]^+$ 264.1570, found 264.1569.

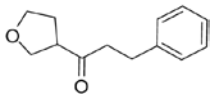
1-cyclopropyl-3-phenylpropan-1-one (15)



Following GP-5, pyridinium salt SI-1 (250 mg, 0.5 mmol, 1 equiv) and cyclopropanecarboxylic acid (43 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (0-5% diethyl ether/hexane) to afford 0.063 g (73%) of the desired product as light yellow oil.

IR 3007, 1696, 1385, 1101, 698 cm^{-1}
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 – 7.23 (m, 2H), 7.22 – 7.16 (m, 3H), 2.96 – 2.84 (m, 4H), 1.91 (tt, *J* = 7.8, 4.6 Hz, 1H), 1.06 – 0.99 (m, 2H), 0.85 (dq, *J* = 7.3, 3.7 Hz, 2H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 209.9, 141.2, 128.5, 128.3, 126.1, 45.0, 30.0, 20.6, 10.7.
HRMS (ESI) m/z calcd for $C_{12}H_{14}O$ $[M+H]^+$ 175.1117, found 175.1117.

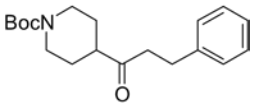
3-phenyl-1-(tetrahydrofuran-3-yl)propan-1-one (16)



Following GP-5, pyridinium salt SI-1 (250 mg, 0.5 mmol, 1 equiv) and tetrahydro-3-furoic acid (58 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (0-20% ethyl acetate/hexane) to afford 0.049 g (47%) of the desired product as colorless oil.

IR 2929, 1708, 1490, 1444, 1264, 733 cm^{-1}
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 – 7.25 (m, 2H), 7.22 – 7.15 (m, 3H), 3.91 – 3.86 (m, 1H), 3.83 (dd, *J* = 8.8, 6.2 Hz, 2H), 3.76 (dt, *J* = 8.4, 7.0 Hz, 1H), 3.16 (tt, *J* = 8.3, 6.6 Hz, 1H), 2.92 (t, *J* = 7.2 Hz, 2H), 2.87 – 2.72 (m, 2H), 2.10 – 1.98 (m, 2H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 209.0, 140.9, 128.6, 128.3, 126.2, 69.3, 68.3, 51.2, 43.7, 29.7, 28.8.
HRMS (ESI) m/z calcd for $C_{13}H_{16}O_2$ $[M+H]^+$ 205.1223, found 205.1223.

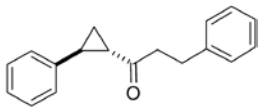
tert-butyl 4-(3-phenylpropanoyl)piperidine-1-carboxylate (17)



Following GP-5, pyridinium salt SI-1 (250 mg, 0.5 mmol, 1 equiv) and 1-(tert-butoxycarbonyl)piperidine-4-carboxylic acid (115 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (0-20% ethyl acetate/hexane) to afford 0.103 g (65%) of the desired product as light yellow oil.

IR 2928, 2855, 1707, 1677, 1495, 1278, 1030, 704
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 – 7.24 (m, 2H), 7.21 – 7.15 (m, 3H), 4.07 (s, 2H), 2.89 (t, *J* = 7.5 Hz, 2H), 2.81 – 2.69 (m, 4H), 2.42 (tt, *J* = 11.4, 3.7 Hz, 1H), 1.76 (d, *J* = 13.2 Hz, 2H), 1.54 – 1.46 (m, 2H), 1.45 (s, 9H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 211.2, 154.7, 141.1, 128.5, 128.3, 126.2, 79.6, 48.8, 43.2, 42.2, 29.7, 28.4, 27.4.
HRMS (ESI) m/z calcd for $C_{19}H_{27}NO_3$ $[M+Na]^+$ 340.1883, found 340.1880.

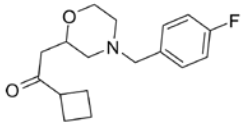
3-phenyl-1-((1S,2S)-2-phenylcyclopropyl)propan-1-one (18)



Following GP-5, pyridinium salt SI-1 (250 mg, 0.5 mmol, 1 equiv) and trans-2-Phenylcyclopropane-1-carboxylic acid (81 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (0-5% ethyl acetate/hexane) to afford 0.098 g (78%) of the desired product as light yellow oil.

IR 3027, 1693, 1398, 1096, 744 cm^{-1}
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.19 (dd, *J* = 8.2, 6.9 Hz, 4H), 7.11 (qd, *J* = 7.0, 6.5, 2.4 Hz, 4H), 6.98 (dd, *J* = 7.3, 1.7 Hz, 2H), 2.91-2.80 (m, 4H), 2.42 (ddd, *J* = 9.1, 6.6, 4.0 Hz, 1H), 2.09 (dt, *J* = 8.1, 5.0 Hz, 1H), 1.59 (ddd, *J* = 9.3, 5.2, 4.2 Hz, 1H), 1.27 (ddd, *J* = 8.1, 6.5, 4.2 Hz, 1H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 208.0, 141.0, 140.3, 128.5, 128.4, 126.5, 126.1, 126.1, 45.5, 32.4, 30.0, 29.0, 19.0.
HRMS (ESI) m/z calcd for $C_{18}H_{18}O$ $[M+H]^+$ 251. 1430, found 251.1428.

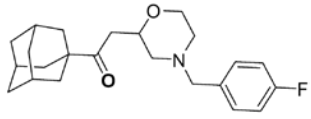
1-cyclobutyl-2-(4-(4-fluorobenzyl)morpholin-2-yl)ethan-1-one (19)



Following GP-5, pyridinium salt SI-8 (301 mg, 0.5 mmol, 1 equiv) and cyclobutanecarboxylic acid (50 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (10-50% ethyl acetate/hexane) to afford 0.091 g (62%) of the desired product as light yellow oil.

IR 2942, 2865, 1705, 1508, 1397, 1218, 1107, 840, 765 cm^{-1}
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 – 7.29 (m, 2H), 7.06 – 6.99 (m, 2H), 3.99 (dddd, *J* = 9.9, 7.5, 5.1, 2.3 Hz, 1H), 3.79 (ddd, *J* = 11.3, 3.3, 1.8 Hz, 1H), 3.65 (td, *J* = 11.3, 2.5 Hz, 1H), 3.44 (d, *J* = 2.8 Hz, 2H), 3.26 (pd, *J* = 8.6, 1.2 Hz, 1H), 2.72 (dt, *J* = 11.1, 2.0 Hz, 1H), 2.63 – 2.54 (m, 2H), 2.32 (dd, *J* = 16.0, 5.1 Hz, 1H), 2.28 – 2.18 (m, 2H), 2.17 – 2.07 (m, 3H), 1.95 (dq, *J* = 11.2, 8.8 Hz, 1H), 1.86 (dd, *J* = 11.1, 9.8 Hz, 1H), 1.83 – 1.75 (m, 1H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 209.2, 162.1 (d, J_{C-F} = 245.0 Hz), 133.5 (d, J_{C-F} = 3.2 Hz), 130.5 (d, J_{C-F} = 7.8 Hz), 115.1 (d, J_{C-F} = 21.2 Hz), 71.9, 66.7, 62.3, 58.2, 52.7, 46.0, 44.0, 24.11, 24.09, 17.6.
^{19}F NMR (377 MHz, $CDCl_3$) δ -115.68**.**
HRMS (ESI) m/z calcd for $C_{17}H_{22}FNO_2$ $[M+H]^+$ 292.1707, found 292.1705.

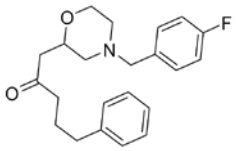
1-((3r,5r,7r)-adamantan-1-yl)-2-(4-(4-fluorobenzyl)morpholin-2-yl)ethan-1-one (20)



Following GP-5, pyridinium salt SI-8 (301 mg, 0.5 mmol, 1 equiv) and 1-adamantanecarboxylic acid (90 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (10-50% ethyl acetate/hexane) to afford 0.106 g (57%) of the desired product as light yellow oil.

IR 2906, 2848, 1694, 1506, 1219, 1110, 1047, 828 cm^{-1}
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 – 7.26 (m, 2H), 7.02 – 6.96 (m, 2H), 4.08 – 4.00 (m, 1H), 3.78 (ddd, *J* = 11.4, 3.3, 1.8 Hz, 1H), 3.66 (td, *J* = 11.3, 2.5 Hz, 1H), 3.51 – 3.36 (m, 2H), 2.86 – 2.70 (m, 2H), 2.59 (dq, *J* = 11.4, 2.1 Hz, 1H), 2.36 (dd, *J* = 17.0, 5.8 Hz, 1H), 2.12 (td, *J* = 11.3, 3.3 Hz, 1H), 2.03 (p, *J* = 3.1 Hz, 3H), 1.86 (dd, *J* = 11.1, 9.8 Hz, 1H), 1.77 (d, *J* = 2.9 Hz, 6H), 1.76 – 1.63 (m, 6H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 212.7, 162.0 (d, $J_{C\text{-}F}$ = 244.9 Hz), 133.5 (d, $J_{C\text{-}F}$ = 3.3 Hz), 130.5 (d, $J_{C\text{-}F}$ = 7.9 Hz), 115.1 (d, $J_{C\text{-}F}$ = 21.2 Hz), 71.9, 66.7, 62.3, 58.3, 52.7, 46.5, 40.0, 37.9, 36.5, 27.9.
^{19}F NMR (377 MHz, $CDCl_3$) δ -115.81.
HRMS (ESI) m/z calcd for $C_{23}H_{30}FNO_2$ $[M+H]^+$ 372.2333, found 372. 2329.

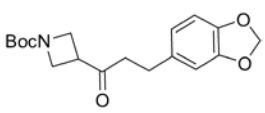
1-(4-(4-fluorobenzyl)morpholin-2-yl)-5-phenylpentan-2-one (21)



Following GP-5, pyridinium salt SI-8 (301 mg, 0.5 mmol, 1 equiv) and 4-phenylbutanoic acid (82 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (10-40% ethyl acetate/hexane) to afford 0.145 g (82%) of the desired product as light yellow oil.

IR 3025, 2930, 1712, 1507, 1218, 1107, 698 cm^{-1}
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27 (dd, *J* = 8.3, 6.0 Hz, 4H), 7.22 – 7.10 (m, 3H), 6.99 (t, *J* = 8.6 Hz, 2H), 3.98 (dddd, *J* = 10.1, 7.5, 4.8, 2.3 Hz, 1H), 3.79 (ddd, *J* = 11.5, 3.3, 1.8 Hz, 1H), 3.64 (td, *J* = 11.3, 2.5 Hz, 1H), 3.44 (s, 2H), 2.70 (dt, *J* = 11.0, 2.0 Hz, 1H), 2.60 (td, *J* = 9.3, 7.8, 4.0 Hz, 4H), 2.50 – 2.40 (m, 2H), 2.33 (dd, *J* = 15.7, 4.8 Hz, 1H), 2.13 (td, *J* = 11.3, 3.3 Hz, 1H), 1.95 – 1.82 (m, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 208.3, 162.1 (d, $J_{C\text{-}F}$ = 244.9 Hz), 141.6, 133.3 (d, $J_{C\text{-}F}$ = 3.2 Hz), 130.5, (d, $J_{C\text{-}F}$ = 7.9 Hz), 128.5, 128.4, 125.9, 115.1 (d, $J_{C\text{-}F}$ = 21.3 Hz), 72.0, 66.7, 62.3, 58.0, 52.7, 46.6, 42.8, 35.0, 24.9.
^{19}F NMR (377 MHz, $CDCl_3$) δ -115.68.
HRMS (ESI) m/z calcd for $C_{22}H_{26}FNO_2$ $[M+H]^+$ 365.2020, found 365.2016.

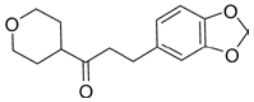
***tert*-butyl 3-(3-(benzo[d][1,3]dioxol-5-yl)propanoyl)azetidine-1-carboxylate (22)**



Following GP-5, pyridinium salt SI-2 (272 mg, 0.5 mmol, 1 equiv) and 1-Boc-azetidine-3-carboxylic acid (100 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (10-30% ethyl acetate/hexane) to afford 0.077 g (46%) of the desired product as light yellow oil.

IR 2888, 1704, 1489, 1245, 1128, 1037, 775 cm^{-1}
^{1}H NMR (500 MHz, $CDCl_3$) δ 6.71 (d, *J* = 7.9 Hz, 1H), 6.69 – 6.64 (m, 1H), 6.64 – 6.57 (m, 1H), 5.92 (s, 2H), 3.99 (d, *J* = 7.7 Hz, 4H), 3.36 (p, *J* = 7.6 Hz, 1H), 2.84 (t, *J* = 7.4 Hz, 2H), 2.69 (t, *J* = 7.4 Hz, 2H), 1.43 (s, 9H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 207.1, 156.2, 147.7, 146.0, 134.4, 121.1, 108.8, 108.3, 100.9, 79.8, 50.5, 42.6, 38.7, 29.3, 28.3.
HRMS (ESI) m/z calcd for $C_{18}H_{23}NO_5$ $[M+Na]^+$ 356.1468, found 356. 1467.

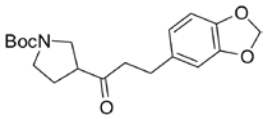
3-(benzo[d][1,3]dioxol-5-yl)-1-(tetrahydro-2H-pyran-4-yl)propan-1-one (23)



Following GP-5, pyridinium salt SI-2 (272 mg, 0.5 mmol, 1 equiv) and tetrahydropyran-4-yl-carboxylic acid (65 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (10-30% ethyl acetate/hexane) to afford 0.098 g (75%) of the desired product as colorless oil.

IR 2949, 2843, 1705, 1487, 1240, 1036, 809 cm^{-1}
^{1}H NMR (500 MHz, $CDCl_3$) δ 6.71 (d, *J* = 7.9 Hz, 1H), 6.66 (d, *J* = 1.6 Hz, 1H), 6.62 (dd, *J* = 7.9, 1.7 Hz, 1H), 5.91 (s, 2H), 3.98 (ddd, *J* = 11.5, 4.3, 2.6 Hz, 2H), 3.39 (td, *J* = 11.4, 2.8 Hz, 2H), 2.82 (t, *J* = 7.7 Hz, 2H), 2.76 – 2.69 (m, 2H), 2.50 (tt, *J* = 11.0, 4.2 Hz, 1H), 1.74 – 1.62 (m, 4H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 210.9, 147.7, 145.9, 134.9, 121.1, 108.8, 108.3, 100.8, 67.2, 47.7, 42.2, 29.4, 28.0.
HRMS (ESI) m/z calcd for $C_{15}H_{18}O_4$ $[M+H]^+$ 263.1278, found 263.1276.

***tert*-butyl 3-(3-(benzo[d][1,3]dioxol-5-yl)propanoyl)pyrrolidine-1-carboxylate (24)**

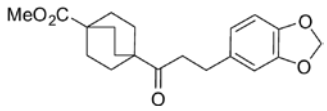


Following GP-5, pyridinium salt SI-2 (272 mg, 0.5 mmol, 1 equiv) and 1-boc-pyrrolidine-3-carboxylic acid (108 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (20-50% ethyl acetate/hexane) to afford 0.099 g (57%) of the desired product as colorless oil.

IR 2973, 2883, 1687, 1488, 1401, 1242, 1036, 701 cm^{-1}
^{1}H NMR (500 MHz, $CDCl_3$) δ 6.72 (d, *J* = 7.9 Hz, 1H), 6.66 (d, *J* = 1.7 Hz, 1H), 6.62 (dd, *J* = 7.9, 1.7 Hz, 1H), 5.92 (s, 2H), 3.63 – 3.24 (m, 4H), 3.15 – 3.01 (m, 1H), 2.88 – 2.67 (m, 4H), 2.17 – 1.83 (m, 2H), 1.45 (s, 9H).

^{1}H NMR (400 MHz, DMSO-d_6, VT NMR conducted at 80 °C) δ 6.77 (d, J = 6.8 Hz, 2H), 6.66 (d, J = 8.0 Hz, 1H), 5.94 (s, 2H), 3.41 (t, J = 9.3 Hz, 1H), 3.36 – 3.30 (m, 2H), 3.24 (dq, J = 15.2, 7.5, 6.7 Hz, 2H), 3.08 (d, J = 3.8 Hz, 1H), 2.80 (t, J = 6.2 Hz, 2H), 2.75 (d, J = 6.3 Hz, 2H), 2.11 – 1.99 (m, 1H), 1.88 (dq, J = 14.6, 7.8 Hz, 1H), 1.42 (d, J = 2.2 Hz, 9H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 208.5, 154.3, 147.7, 145.9, 134.6, 121.1, 108.8, 108.3, 100.9, 79.4, 53.4, 50.6, 49.6, 47.0, 45.4, 45.2, 43.6, 29.4, 28.5, 28.0, 27.7.
HRMS (ESI) m/z calcd for $C_{19}H_{25}NO_5$ $[M+Na]^+$ 370.1625, found 370.1622.

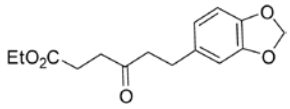
methyl 4-(3-(benzo[d][1,3]dioxol-5-yl)propanoyl)bicyclo[2.2.2]octane-1-carboxylate (25)



Following GP-5, pyridinium salt SI-2 (272 mg, 0.5 mmol, 1 equiv) and 4-(methoxycarbonyl)bicyclo[2.2.2]octane-1-carboxylic acid (106 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (10-30% ethyl acetate/hexane) to afford 0.124 g (72%) of the desired product as light yellow oil.

IR 2953, 2869, 1719, 1689, 1439, 1363, 1034, 752 cm^{-1}
^{1}H NMR (500 MHz, $CDCl_3$) δ 6.70 (d, J = 7.9 Hz, 1H), 6.65 (d, J = 1.7 Hz, 1H), 6.60 (dd, J = 7.9, 1.7 Hz, 1H), 5.91 (s, 2H), 3.64 (s, 3H), 2.81 – 2.73 (m, 2H), 2.72 – 2.66 (m, 2H), 1.80 (dd, J = 10.3, 5.4 Hz, 6H), 1.72 – 1.68 (m, 6H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 213.8, 177.7, 147.6, 145.8, 135.3, 121.1, 108.9, 108.2, 100.8, 51.8, 44.5, 39.4, 38.9, 29.4, 27.8, 27.0.
HRMS (ESI) m/z calcd for $C_{20}H_{24}O_5$ $[M+Na]^+$ 367.1516, found 367.1513.

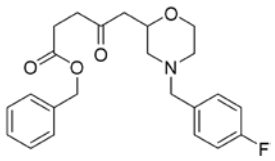
ethyl 6-(benzo[d][1,3]dioxol-5-yl)-4-oxohexanoate (26)



Following GP-5, pyridinium salt SI-2 (272 mg, 0.5 mmol, 1 equiv) and mono-ethyl succinate (73 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (0-10% ethyl acetate/hexane) to afford 0.096 g (69%) of the desired product as light yellow oil.

IR 2988, 2887, 1658, 1522, 1264, 905, 727 cm^{-1}
^{1}H NMR (500 MHz, $CDCl_3$) δ 6.71 (d, J = 7.9 Hz, 1H), 6.67 (d, J = 1.7 Hz, 1H), 6.62 (dd, J = 7.9, 1.8 Hz, 1H), 5.91 (s, 2H), 4.13 (q, J = 7.1 Hz, 2H), 2.83 (t, J = 7.1 Hz, 2H), 2.77 – 2.71 (m, 2H), 2.69 (t, J = 6.5 Hz, 2H), 2.57 (t, J = 6.5 Hz, 2H), 1.25 (t, J = 7.2 Hz, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 208.0, 172.8, 147.7, 145.8, 134.8, 121.1, 108.8, 108.3, 100.8, 60.7, 44.5, 37.3, 29.4, 28.0, 14.2.
HRMS (ESI) m/z calcd for $C_{15}H_{18}O_5$ $[M+H]^+$ 279.1227, found 279.1224.

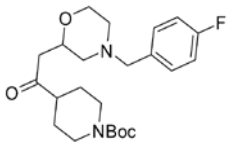
benzyl 5-(4-(4-fluorobenzyl)morpholin-2-yl)-4-oxopentanoate (27)



Following GP-5, pyridinium salt SI-8 (301 mg, 0.5 mmol, 1 equiv) and 4-(benzyloxy)-4-oxobutanoic acid (104 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (20-50% ethyl acetate/hexane) to afford 0.084 g (42%) of the desired product as light yellow oil.

IR 2928, 1716, 1508, 1264, 1109, 733 cm^{-1}
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 – 7.31 (m, 5H), 7.30 – 7.26 (m, 2H), 7.05 – 6.96 (m, 2H), 5.10 (s, 2H), 3.99 (dddd, *J* = 10.0, 7.5, 4.7, 2.3 Hz, 1H), 3.79 (ddd, *J* = 11.4, 3.3, 1.8 Hz, 1H), 3.64 (td, *J* = 11.3, 2.5 Hz, 1H), 3.44 (d, *J* = 1.7 Hz, 2H), 2.78 (td, *J* = 6.6, 2.0 Hz, 2H), 2.71 (dt, *J* = 10.7, 1.9 Hz, 1H), 2.69 – 2.63 (m, 2H), 2.63 – 2.58 (m, 2H), 2.42 (dd, *J* = 15.7, 4.7 Hz, 1H), 2.13 (td, *J* = 11.3, 3.3 Hz, 1H), 1.87 (dd, *J* = 11.2, 9.9 Hz, 1H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 206.4, 172.6, 162.1 (d, $J_{C\text{-}F}$ = 245.2 Hz), 135.9, 133.4 (d, $J_{C\text{-}F}$ = 3.2 Hz), 130.5 (d, $J_{C\text{-}F}$ = 7.9 Hz), 128.6, 128.2, 128.2, 115.1 (d, $J_{C\text{-}F}$ = 21.2 Hz), 72.0, 66.8, 66.5, 62.3, 58.0, 52.7, 46.7, 38.0, 27.9.
^{19}F NMR (377 MHz, $CDCl_3$) δ -116.06.
HRMS (ESI) m/z calcd for $C_{23}H_{26}FNO_4$ $[M+H]^+$ 400.1919, found 400.1918.

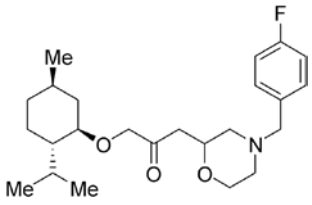
tert-butyl 4-(2-(4-(4-fluorobenzyl)morpholin-2-yl)acetyl)piperidine-1-carboxylate (28)



Following GP-4, pyridinium salt SI-8 (301 mg, 0.5 mmol, 1 equiv) and SII-2 (115 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (20-50% ethyl acetate/hexane) to afford 0.193 g (92%) of the desired product as yellow oil.

IR 2976, 1735, 1684, 1420, 1161, 729 cm^{-1}
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 – 7.24 (m, 2H), 7.04 – 6.95 (m, 2H), 4.22 – 3.93 (m, 3H), 3.79 (ddd, *J* = 11.4, 3.3, 1.8 Hz, 1H), 3.64 (td, *J* = 11.3, 2.6 Hz, 1H), 3.45 (s, 2H), 2.81 – 2.66 (m, 4H), 2.61 (dq, *J* = 11.5, 2.1 Hz, 1H), 2.47 (tt, *J* = 11.4, 3.7 Hz, 1H), 2.39 (dd, *J* = 16.0, 4.9 Hz, 1H), 2.13 (td, *J* = 11.3, 3.5 Hz, 1H), 1.87 (t, *J* = 10.5 Hz, 1H), 1.80 (s, 2H), 1.45 (d, *J* = 5.0 Hz, 11H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 209.7, 162.1 (d, $J_{C\text{-}F}$ = 245.7 Hz), 154.7, 133.3 (d, $J_{C\text{-}F}$ = 3.1 Hz), 130.3 (d, $J_{C\text{-}F}$ = 8.0 Hz), 115.2 (d, $J_{C\text{-}F}$ = 21.6 Hz), 79.6, 72.0, 66.7, 62.3, 58.0, 52.7, 49.1, 44.4, 28.4, 27.3, 27.1.
^{19}F NMR (377 MHz, $CDCl_3$) δ -115.62.
HRMS (ESI) m/z calcd for $C_{23}H_{33}FN_2O_4$ $[M+H]^+$ 421.2497, found 421.2494.

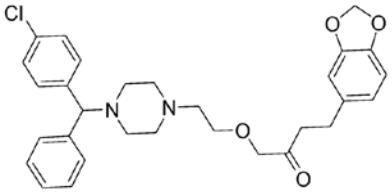
1-(4-(4-fluorobenzyl)morpholin-2-yl)-3-(((1R,2S,5R)-2-isopropyl-5-methylcyclohexyl)oxy)propan-2-one (29)



Following GP-5, L2 (11 mg, 0.005 mmol, 0.1 equiv), pyridinium salt SI-8 (301 mg, 0.5 mmol, 1 equiv) and (-)-Menthyloxyacetic acid (107 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (0-30% ethyl acetate/hexane) to afford 0.144 g (71%) of the desired product as a mixture of two diastereomers (d.r. = 1:1), as light yellow oil.

IR 2953, 2868, 1719, 1689, 1239, 1034, 732 cm^{-1}
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 – 7.23 (m, 2H), 7.04 – 6.95 (m, 2H), 4.15 (dd, *J* = 16.9, 10.5 Hz, 1H), 4.05 – 3.92 (m, 2H), 3.80 (ddt, *J* = 11.3, 3.3, 1.6 Hz, 1H), 3.65 (td, *J* = 11.3, 2.5 Hz, 1H), 3.45 (s, 2H), 3.10 (tdd, *J* = 10.6, 4.2, 2.0 Hz, 1H), 2.77 – 2.67 (m, 2H), 2.61 (dq, *J* = 11.5, 2.1 Hz, 1H), 2.48 (ddd, *J* = 16.0, 5.1, 1.8 Hz, 1H), 2.23 (hd, *J* = 7.0, 2.7 Hz, 1H), 2.14 (tdd, *J* = 11.3, 3.4, 1.9 Hz, 1H), 2.02 (dp, *J* = 12.4, 3.4 Hz, 1H), 1.88 (ddd, *J* = 11.5, 9.9, 2.1 Hz, 1H), 1.63 (tdd, *J* = 12.2, 5.6, 2.7 Hz, 2H), 1.31 (dtdd, *J* = 25.5, 12.8, 6.8, 3.6 Hz, 2H), 1.02 – 0.81 (m, 9H), 0.77 (d, *J* = 6.9 Hz, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 207.2, 207.0, 162.1 (d, $J_{C\text{-}F}$ = 246.2 Hz), 133.5 (d, $J_{C\text{-}F}$ = 3.3 Hz),133.4 (d, $J_{C\text{-}F}$ = 3.3 Hz), 133.5 (d, $J_{C\text{-}F}$ = 7.9 Hz), 115.1 (d, $J_{C\text{-}F}$ = 21.2 Hz), 80.2, 80.2, 74.5, 74.4, 71.8, 71.7, 66.76, 66.75, 62.3, 58.1, 58.1, 52.7, 52.7, 48.11, 48.09, 43.2, 39.99, 39.96, 34.4, 31.5, 25.59, 25.58, 23.22, 23.21, 22.3, 21.0, 16.2.
^{19}F NMR (377 MHz, $CDCl_3$) δ -115.72, -115.74.
HRMS (ESI) m/z calcd for $C_{24}H_{36}FNO_3$ $[M+H]^+$ 406.2752, found 406.2748.

4-(benzo[d][1,3]dioxol-5-yl)-1-(2-(4-((4-chlorophenyl)(phenyl)methyl)piperazin-1-yl)ethoxy)butan-2-one (30)

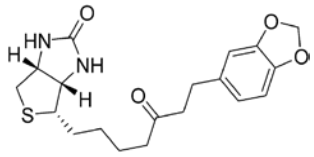


Following GP-5, L2 (11 mg, 0.005 mmol, 0.1 equiv), pyridinium salt SI-2 (272 mg, 0.5 mmol, 1 equiv) and cetirizine (194 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (25-100% ethyl acetate/hexane) to afford 0.088 g (34%) of the desired product as light yellow oil.

IR 2808, 1717, 1487, 1243, 1037, 1008, 757 cm^{-1}

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.36 (m, 4H), 7.32 – 7.24 (m, 4H), 7.23 – 7.18 (m, 1H), 6.72 (d, *J* = 7.9 Hz, 1H), 6.69 (d, *J* = 1.7 Hz, 1H), 6.63 (dd, *J* = 7.9, 1.7 Hz, 1H), 5.92 (s, 2H), 4.22 (s, 1H), 4.03 (s, 2H), 3.60 (t, *J* = 5.7 Hz, 2H), 2.88 – 2.80 (m, 2H), 2.78 – 2.70 (m, 2H), 2.63 (t, *J* = 5.7 Hz, 2H), 2.55 (s, 3H), 2.43 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 208.2, 147.7, 145.9, 142.2, 141.4, 134.7, 132.5, 129.2, 128.7, 128.6, 127.9, 127.2, 121.1, 108.9, 108.3, 100.8, 76.4, 75.5, 69.3, 57.8, 53.8, 51.7, 40.7, 29.1.
HRMS (ESI) m/z calcd for $C_{30}H_{33}ClN_2O_4$ $[M+H]^+$ 521.2202, found 521.2204.

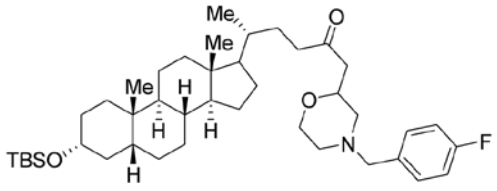
(3aS,4S,6aR)-4-(7-(benzo[d][1,3]dioxol-5-yl)-5-oxoheptyl)tetrahydro-1H-thieno[3,4-d]imidazol-2(3H)-one (31)



Following GP-5, pyridinium salt SI-2 (272 mg, 0.5 mmol, 1 equiv) and biotin (122 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (0-7% MeOH/DCM) to afford 0.041 g (22%) of the desired product as light yellow oil.

IR 3209, 2923, 1695, 1501, 1488, 1242, 1035, 762 cm^{-1}
^{1}H NMR (400 MHz, $CDCl_3$) δ 6.65 (d, *J* = 7.9 Hz, 1H), 6.60 (d, *J* = 1.7 Hz, 1H), 6.55 (dd, *J* = 7.9, 1.7 Hz, 1H), 5.84 (s, 2H), 5.69 (s, 1H), 5.29 (s, 1H), 4.43 (dd, *J* = 7.8, 5.0 Hz, 1H), 4.22 (ddd, *J* = 7.7, 4.6, 1.6 Hz, 1H), 3.07 (ddd, *J* = 8.4, 6.5, 4.6 Hz, 1H), 2.83 (dd, *J* = 12.8, 5.0 Hz, 1H), 2.73 (t, *J* = 7.5 Hz, 2H), 2.70 – 2.57 (m, 2H), 2.33 (t, *J* = 7.3 Hz, 2H), 1.68 – 1.42 (m, 4H), 1.40 – 1.23 (m, 2H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 210.1, 163.5, 147.6, 145.8, 134.9, 121.1, 108.8, 108.3, 100.8, 62.0, 60.1, 55.4, 44.5, 42.6, 40.6, 29.5, 28.41, 28.39, 23.5.
HRMS (ESI) m/z calcd for $C_{19}H_{24}N_2O_4S$ $[M+H]^+$ 377.1530, found 377.1527.

(5R)-5-((3R,5R,8R,9S,10S,13R,14S)-3-((tert-butyldimethylsilyl)oxy)-10,13-dimethylhexadecahydro-1H-cyclopenta[a]phenanthren-17-yl)-1-(4-(4-fluorobenzyl)morpholin-2-yl)hexan-2-one (32)



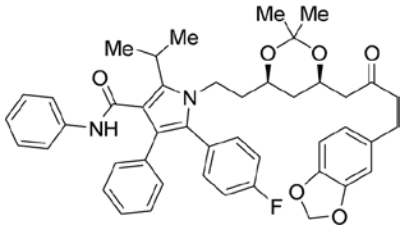
Lithocholic acid was protected as the TBS ether following the literature procedure.[11] Following GP-5, pyridinium salt SI-8 (301 mg, 0.5 mmol, 1 equiv) and TBS protected lithocholic acid (245 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material

[11] D. M. Scott, A. T. McPhail, N. A. Porter, *J. Org. Chem*. **1993**, *58*, 1178 - 1186.

was purified by chromatography (10-30% ethyl acetate/hexane) to afford 0.167 g (49%) of the desired product as a mixture of two diastereomers (d.r. = 1:1), as yellow oil.

IR 2927, 2855, 1715, 1508, 1221, 1092, 834, 772 cm^{-1}
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25 – 7.19 (m, 2H), 6.97 – 6.90 (m, 2H), 3.98 – 3.89 (m, 1H), 3.75 (ddd, *J* = 11.4, 3.4, 1.8 Hz, 1H), 3.60 (td, *J* = 11.3, 2.6 Hz, 1H), 3.52 (ddt, *J* = 15.6, 10.6, 4.6 Hz, 1H), 3.39 (d, *J* = 2.0 Hz, 2H), 2.66 (dq, *J* = 11.0, 1.7 Hz, 1H), 2.62 – 2.52 (m, 2H), 2.39 (m, *J* = 15.3, 10.3, 5.1, 2.0 Hz, 1H), 2.30 (m, *J* = 16.4, 14.2, 6.4, 4.1 Hz, 2H), 2.08 (td, *J* = 11.4, 3.4 Hz, 1H), 1.90 – 1.60 (m, 8H), 1.52 – 1.45 (m, 2H), 1.43 – 1.25 (m, 9H), 1.24 – 1.11 (m, 6H), 1.10 – 0.92 (m, 6H), 0.89 – 0.76 (m, 17), 0.56 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 209.1, 162.1 (d, $J_{C\text{-}F}$ = 244.9 Hz), 133.4 (d, $J_{C\text{-}F}$ = 3.18 Hz), 130.5 (d, $J_{C\text{-}F}$ = 7.8 Hz), 115.1 (d, $J_{C\text{-}F}$ = 21.3 Hz), 72.8, 72.09, 72.07, 66.7, 62.3, 58.12, 58.09, 56.4, 56.0, 52.7, 46.63, 46.60, 42.7, 42.3, 40.6, 40.5, 40.22, 40.15, 36.9, 35.9, 35.6, 35.2, 34.6, 31.0, 29.5, 28.21, 28.19, 27.3, 26.4, 26.0, 24.2, 23.4, 20.8, 18.4, 18.3, 12.0, -4.6.
^{19}F NMR (377 MHz, $CDCl_3$) δ -115.72.
HRMS (ESI) m/z calcd for $C_{42}H_{68}FNO_3Si$ $[M+H]^+$ 682.5025, found 682.5028.

1-(2-((4R,6R)-6-(4-(benzo[d][1,3]dioxol-5-yl)-2-oxobutyl)-2,2-dimethyl-1,3-dioxan-4-yl)ethyl)-5-(4-fluorophenyl)-2-isopropyl-N,4-diphenyl-1H-pyrrole-3-carboxamide (33)



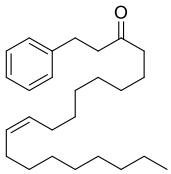
Atorvastatin was protected following literature procedure.[12] Following GP-5, pyridinium salt SI-2 (97 mg, 0.178 mmol, 1 equiv) and protected Atorvastatin (107 mg, 0.178 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (10-50% ethyl acetate/hexane) to afford 0.043 g (33%) of the desired product as white foam.

IR 3408, 2981, 1713, 1666, 1507, 1311, 1155, 1038,735 cm^{-1}
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (dtp, *J* = 9.6, 6.3, 2.8 Hz, 11H), 7.09 (d, *J* = 8.0 Hz, 2H), 7.01 (td, *J* = 8.0, 7.4, 4.1 Hz, 4H), 6.89 (s, 1H), 6.73 (d, *J* = 7.9 Hz, 1H), 6.67 (d, *J* = 1.7 Hz, 1H), 6.63 (dd, *J* = 7.9, 1.7 Hz, 1H), 5.93 (s, 2H), 4.25 (dtd, *J* = 12.0, 6.3, 2.3 Hz, 1H), 4.20 – 4.03 (m, 2H), 3.84 (ddd, *J* = 14.5, 10.0, 6.4 Hz, 1H), 3.75 – 3.65 (m, 1H), 3.58 (h, *J* = 7.1 Hz, 1H), 2.88 – 2.76 (m, 2H), 2.72 (td, *J* = 8.8, 7.8, 5.6 Hz, 2H), 2.64 (dd, *J* = 16.1, 6.8 Hz, 1H), 2.35 (dd, *J* = 16.1, 5.7 Hz, 1H), 2.07 (s, 1H), 1.75 – 1.63 (m, 2H), 1.55 (d, *J* = 7.1 Hz, 6H), 1.36 (s, 3H), 1.30 (s, 3H), 1.05 – 0.90 (m, 1H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 207.8, 171.1, 164.8, 162.3 (d, $J_{C\text{-}F}$ = 247.6 Hz), 147.6, 145.8, 141.5, 138.4, 134.73, 134.66, 133.2 (d, $J_{C\text{-}F}$ = 8.7 Hz), 130.5, 128.8, 128.7, 128.4, 128.3 (d, $J_{C\text{-}F}$ = 3.8 Hz),

[12] T. Qin, J. Cornella, C. Li, L. R. Malins, J. T. Edwards, S. Kawamura, B. D. Maxwell, M. D. Eastgate, P. S. Baran, *Science* **2016**, *352*, 801 - 805.

126.6, 123.5, 121.8, 121.1, 119.6, 115.4 (d, $J_{C\text{-}F}$ = 21.7 Hz), 108.8, 108.2, 100.8, 98.7, 66.4, 65.4, 60.4, 49.2, 45.6, 40.8, 38.1, 36.2, 29.9, 29.2, 26.1, 21.8, 21.6, 19.7.
^{19}F NMR (377 MHz, $CDCl_3$) δ -113.71.
HRMS (ESI) m/z calcd for $C_{45}H_{47}FN_2O_6$ $[M+H]^+$ 731.3491, found 731.3496.

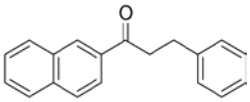
(Z)-1-phenylicos-11-en-3-one (34)



Following GP-5, pyridinium salt SI-1 (250 mg, 0.5 mmol, 1 equiv) and oleic acid (141 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (0-30% toluene/hexane) to afford 0.124 g (67%) of the desired product as colorless oil.

IR 3382, 2923, 2853, 1715, 1604, 1454, 905 cm^{-1}
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.23 (m, 2H), 7.18 (d, *J* = 7.5 Hz, 3H), 5.35 (d, *J* = 6.0 Hz, 2H), 2.89 (t, *J* = 7.7 Hz, 2H), 2.72 (t, *J* = 7.6 Hz, 2H), 2.37 (t, *J* = 7.4 Hz, 2H), 2.00 (q, *J* = 6.6 Hz, 4H), 1.55 (p, *J* = 7.3 Hz, 2H), 1.28 (d, *J* = 12.1 Hz, 20H), 0.88 (t, *J* = 6.6 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 210.4, 141.2, 130.0, 129.8, 128.5, 128.4, 126.8, 44.3, 43.1, 31.9, 29.81, 29.79, 29.7, 29.6, 29.4, 29.3, 29.2, 29.1, 27.24, 27.19, 23.8, 22.7, 14.2.
Note: one of the ^{13}C resonances in the aliphatic region is missing, presumably due to two of the resonances overlapping. The ^{1}H NMR and HRMS data are consistent with this hypothesis.
HRMS (ESI) m/z calcd for $C_{26}H_{42}O$ $[M+H]^+$ 371.3308, found 371.3308.

1-(naphthalen-2-yl)-3-phenylpropan-1-one (35)



Following GP-4, using **L1** (13 mg, 0.05 mmol) as ligand, pyridinium salt SI-1 (375 mg, 0.75 mmol, 1.5 equiv) and acyl fluoride SII-4 (87 mg, 0.5 mmol, 1 equiv) were used. After 24 h, the reaction was quenched, and the crude material was purified by chromatography (0-5% diethyl ether/hexane) to afford 0.044 g (41%) of the desired product as light yellow oil. The Spectra matches the previous report.[13]

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.44 (d, *J* = 1.7 Hz, 1H), 8.06 – 7.97 (m, 1H), 7.91 (d, *J* = 8.1 Hz, 1H), 7.86 (t, *J* = 8.6 Hz, 2H), 7.63 – 7.47 (m, 2H), 7.35 – 7.25 (m, 4H), 7.25 – 7.16 (m, 1H), 3.42 (t, *J* = 7.7 Hz, 2H), 3.12 (t, *J* = 7.7 Hz, 2H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 199.1, 141.4, 135.6, 134.2, 132.5, 129.7, 129.5, 128.5, 128.46, 128.44, 128.42, 127.8, 126.8, 126.2, 123.8, 40.5, 30.3.

[13] F. Li, J. Ma, N. Wang, *J. Org. Chem*., **2014**, *79*, 10447.

7. Cyclic Voltammetry measurement for alkyl pyridinium salt (SI-1) and acyl fluoride (SII-1)

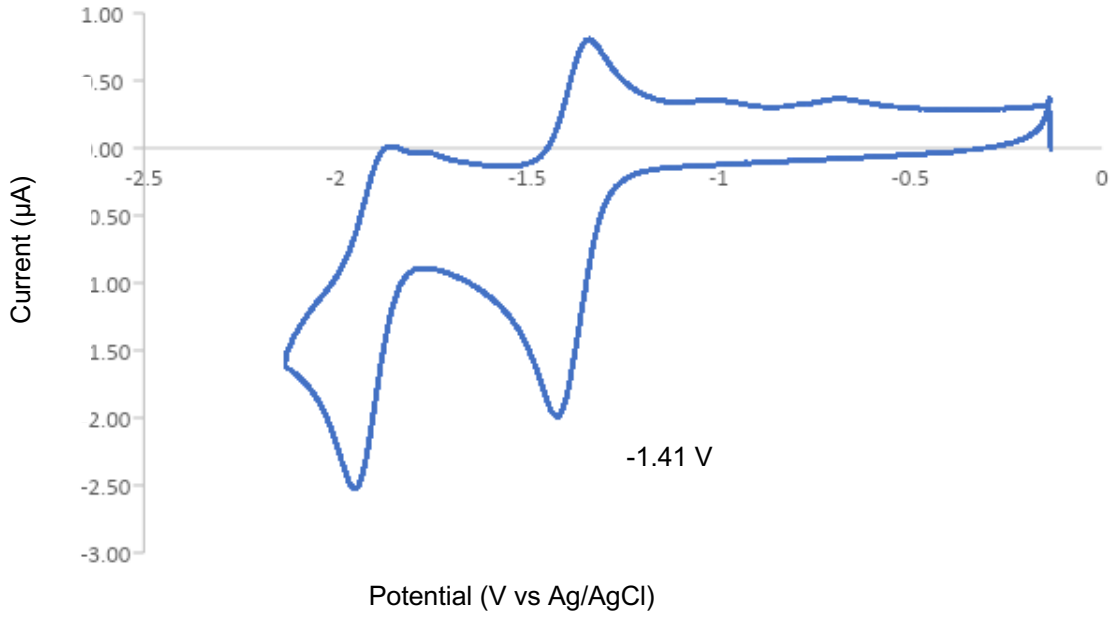
Cyclic Voltammetry in NMP:

Cyclic voltammetry experiments were performed using a WaveNowXV potentiostat at a sweep rate of 50 mV/s. The electrochemical cell was equipped with a glassy carbon disk working electrode (3 mm diameter) and a Pt wire counter electrode. Redox potentials are reported versus a Ag/AgCl reference electrode. Solutions were made to contain 10 mM of the analyte and 100 mM NBu_4PF_6 in DMF and were sparged with nitrogen for 5 min before analysis.

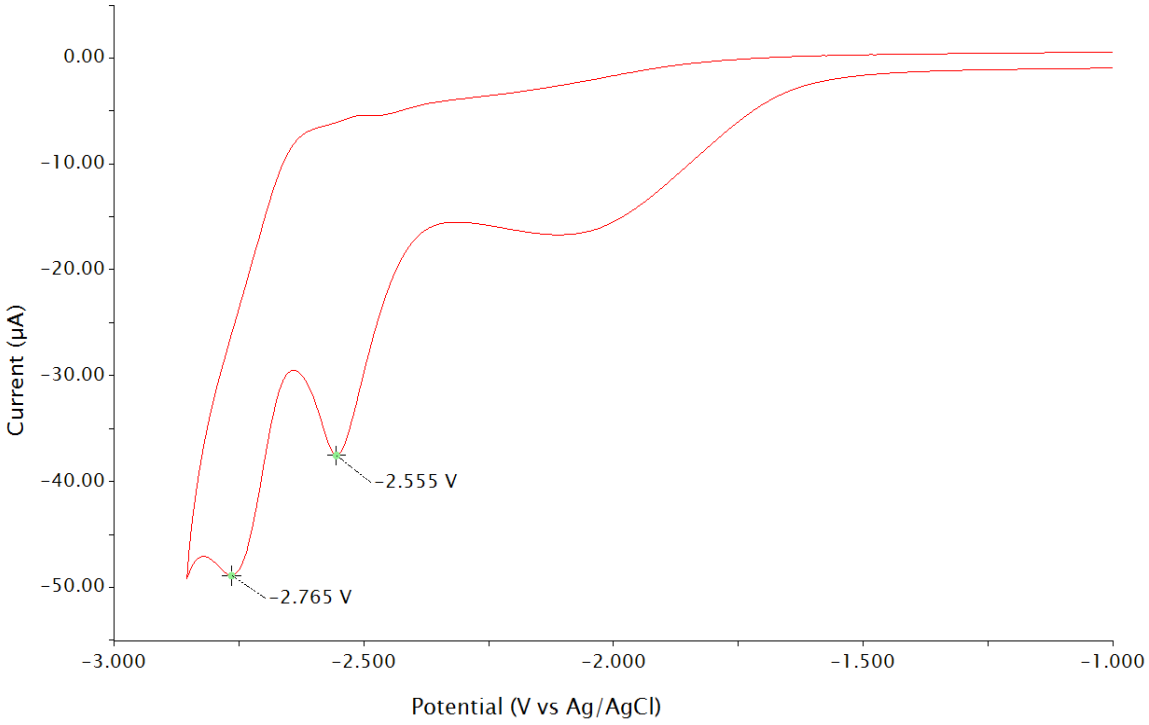
According to our cyclic voltammetry measurement, the reduction peak potential for alkyl pyridinium salt **SI-1** is: E_p = -1.41 V vs. Ag/AgCl. While our previous Suzuki[14] and Negishi[15] couplings have shown that nickel(0) could certainly reduce the pyridinium salts via a single electron reduction process, the reduction potential of Mn (Mn/ Mn^{2+} = -1.4 V vs. Ag/AgCl) is also within the range to directly reduce **SI-1**. Hence, we could not rule out the possibility of Mn directly reducing these pyridinium salts to the corresponding alkyl radicals. Meanwhile, the reduction potential of acyl fluoride SII-1 was measured to be E_p = -2.56 V, which could be hard for a single electron reduction of Mn or nickel (0)[16] , and thus a two electron oxidative addition of nickel (0) to acyl fluoride is more likely in this case.

[14] C. H. Basch, J. Liao, J. Xu, J. J. Piane, M. P. Watson, *J. Am. Chem. Soc*. **2017**, *139*, 5313-5316.
[15] S. Plunkett, C. H. Basch, S. O. Santana, M. P. Watson, *J. Am. Chem. Soc*. **2019**, *141*, 2257-2262.
[16] R. J. Perkins, A. J. Hughes, D. J. Weix, E. C. Hansen, *Org. Process Res. Dev*. **2019**, *23*, 1746-1751.

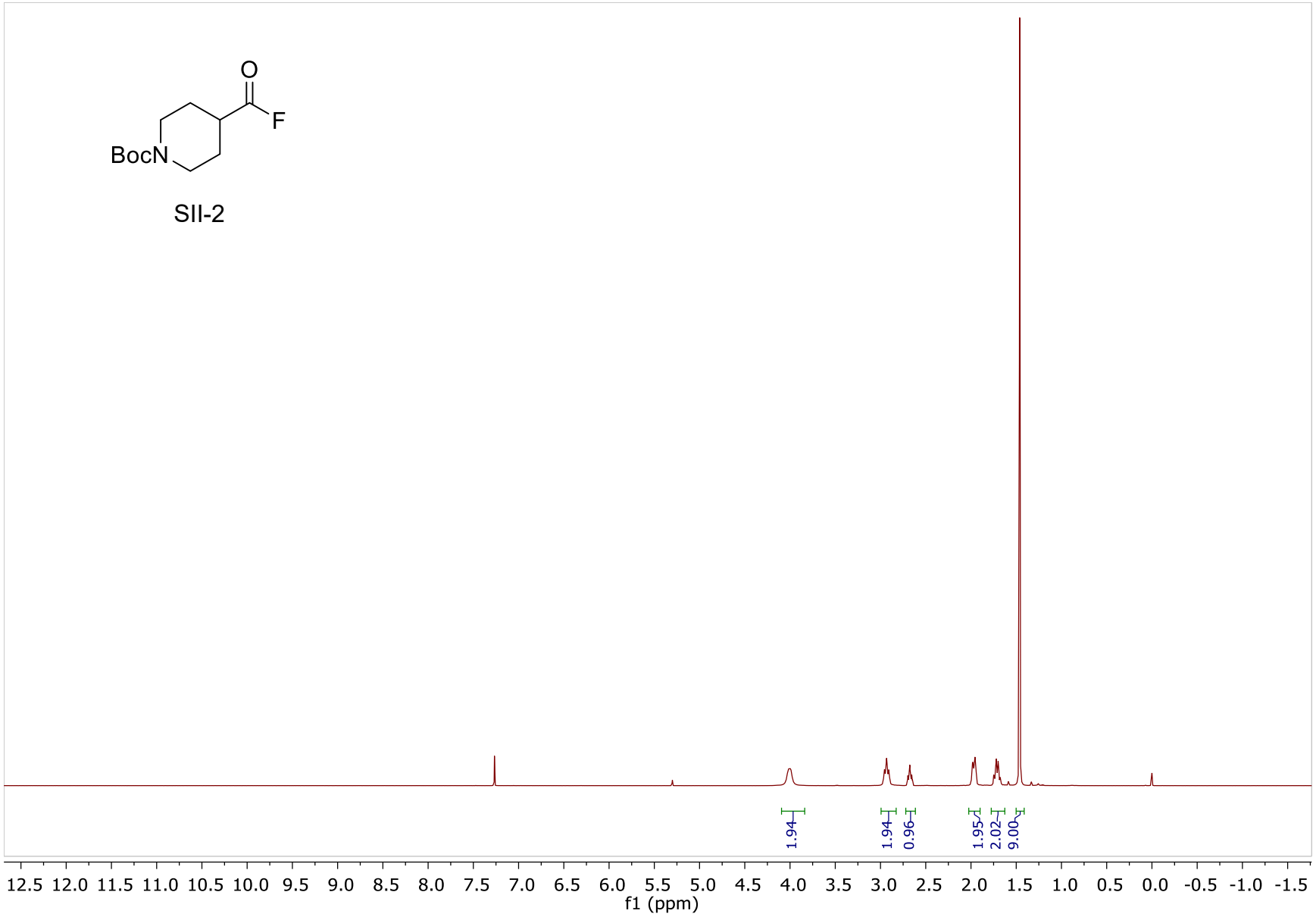
Cyclic Voltammetry of SI-1

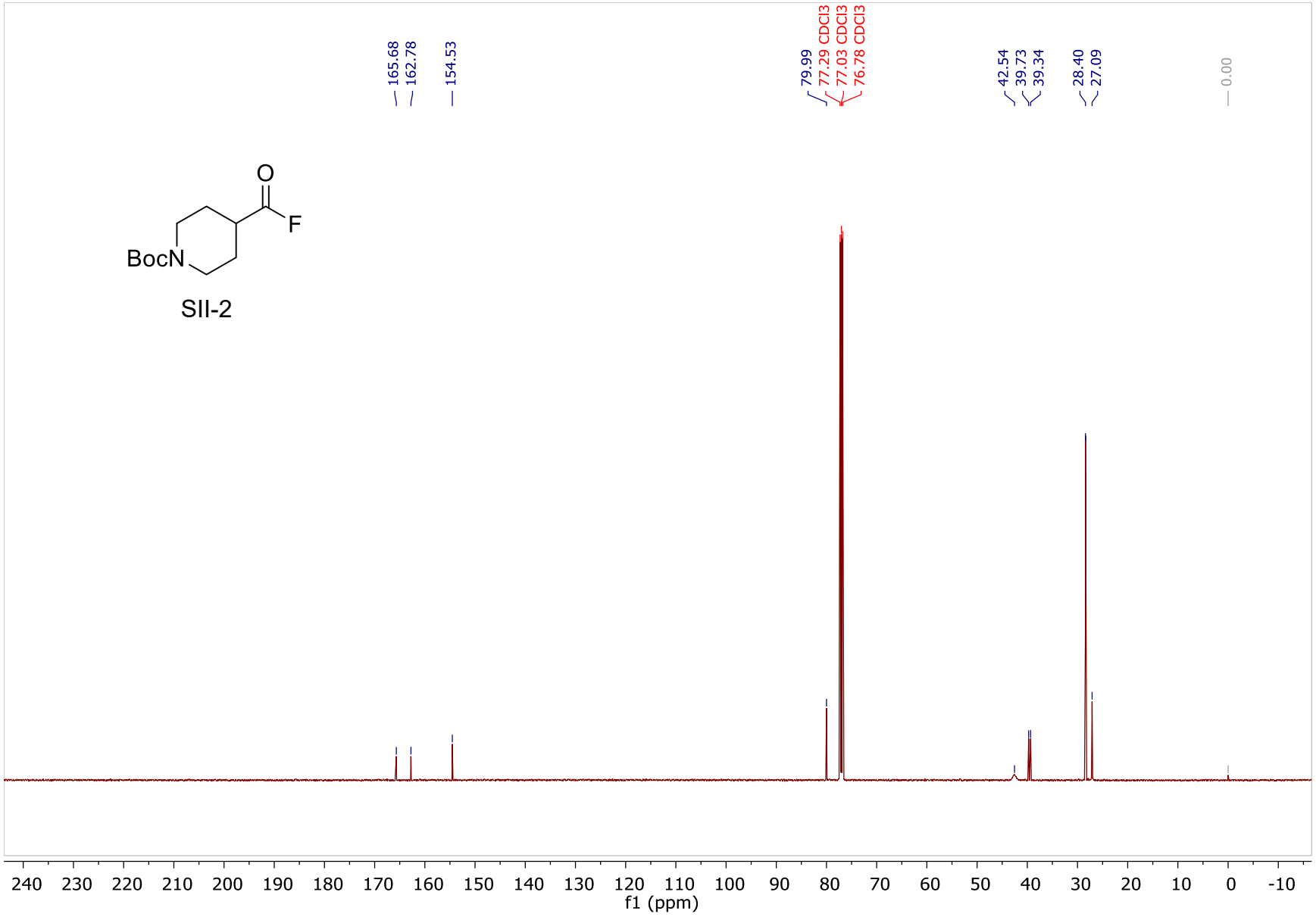


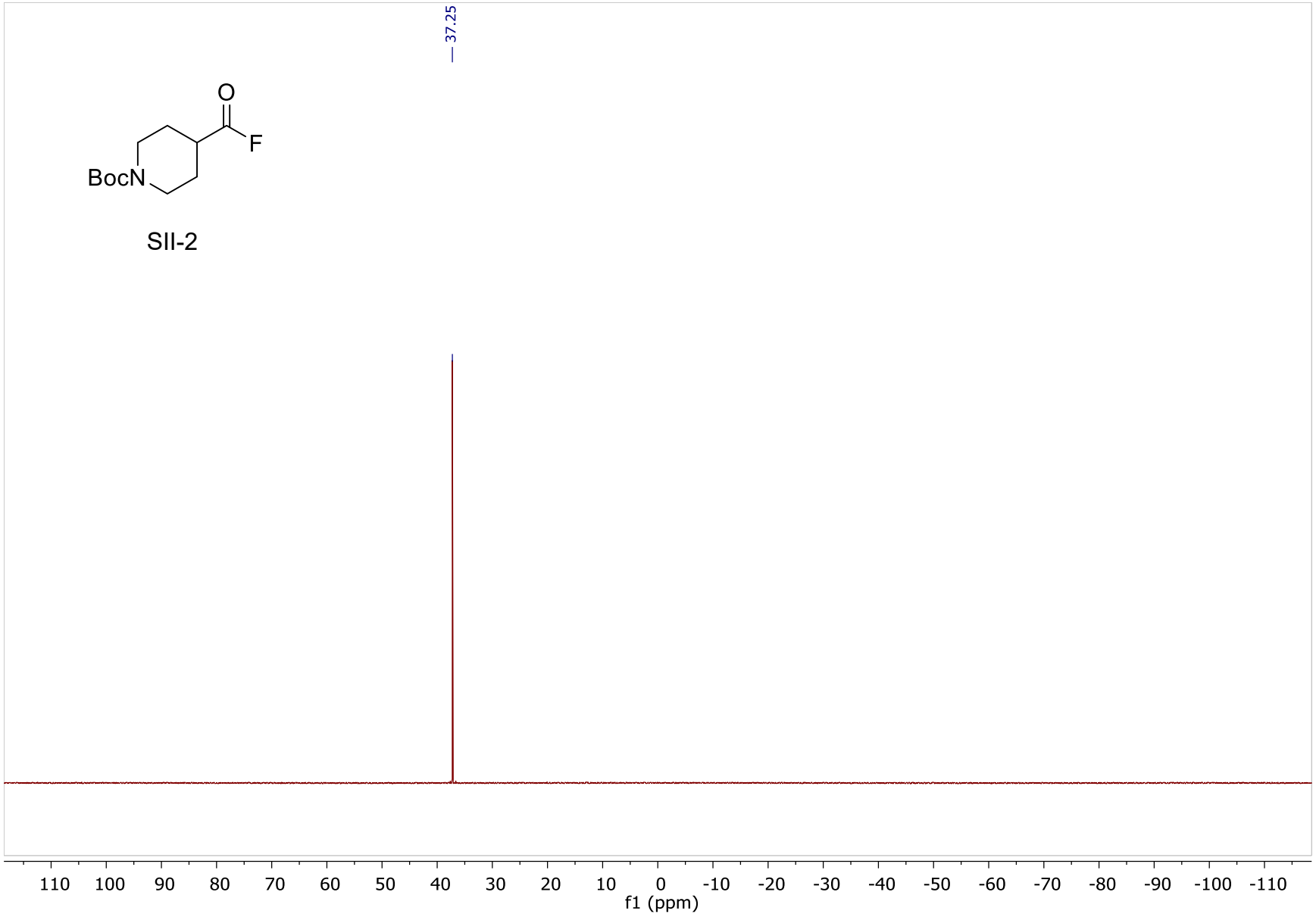
Cyclic Voltammogram of SII-1

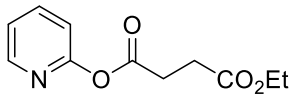


8. NMR Spectra Copies

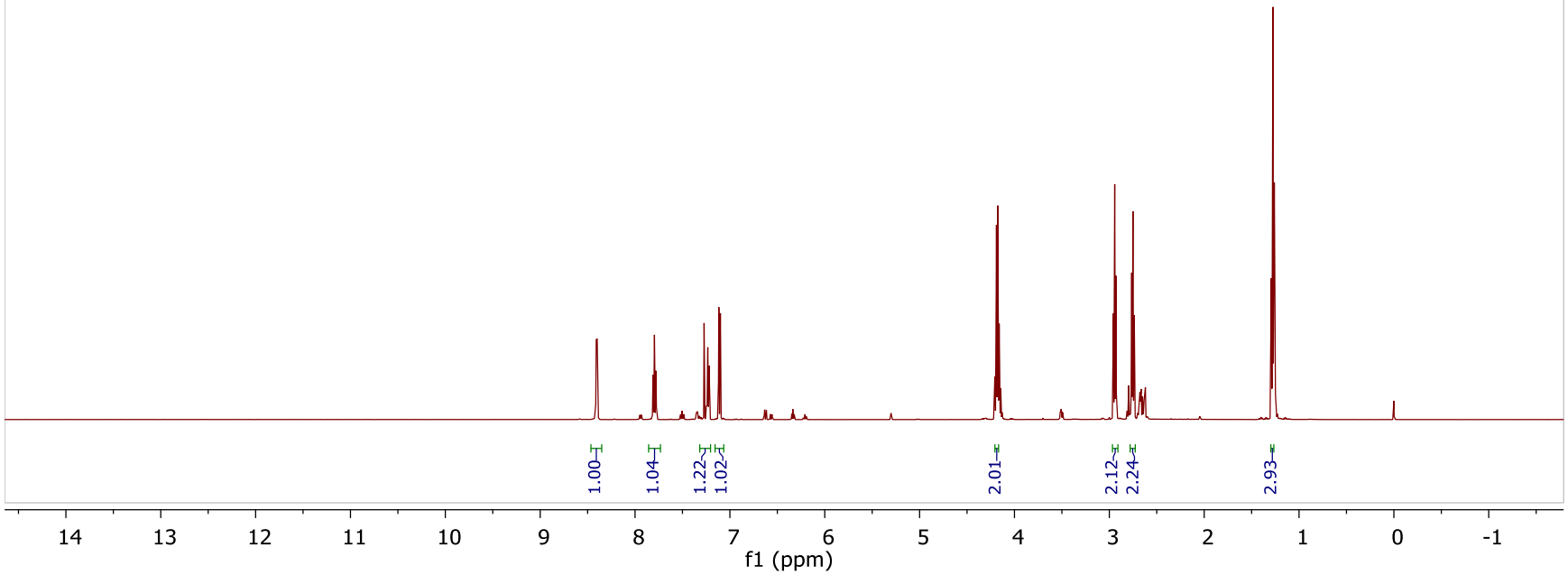

O
F
BocN
SII-2
1.94
1.94
0.96
1.95
2.02
9.00
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

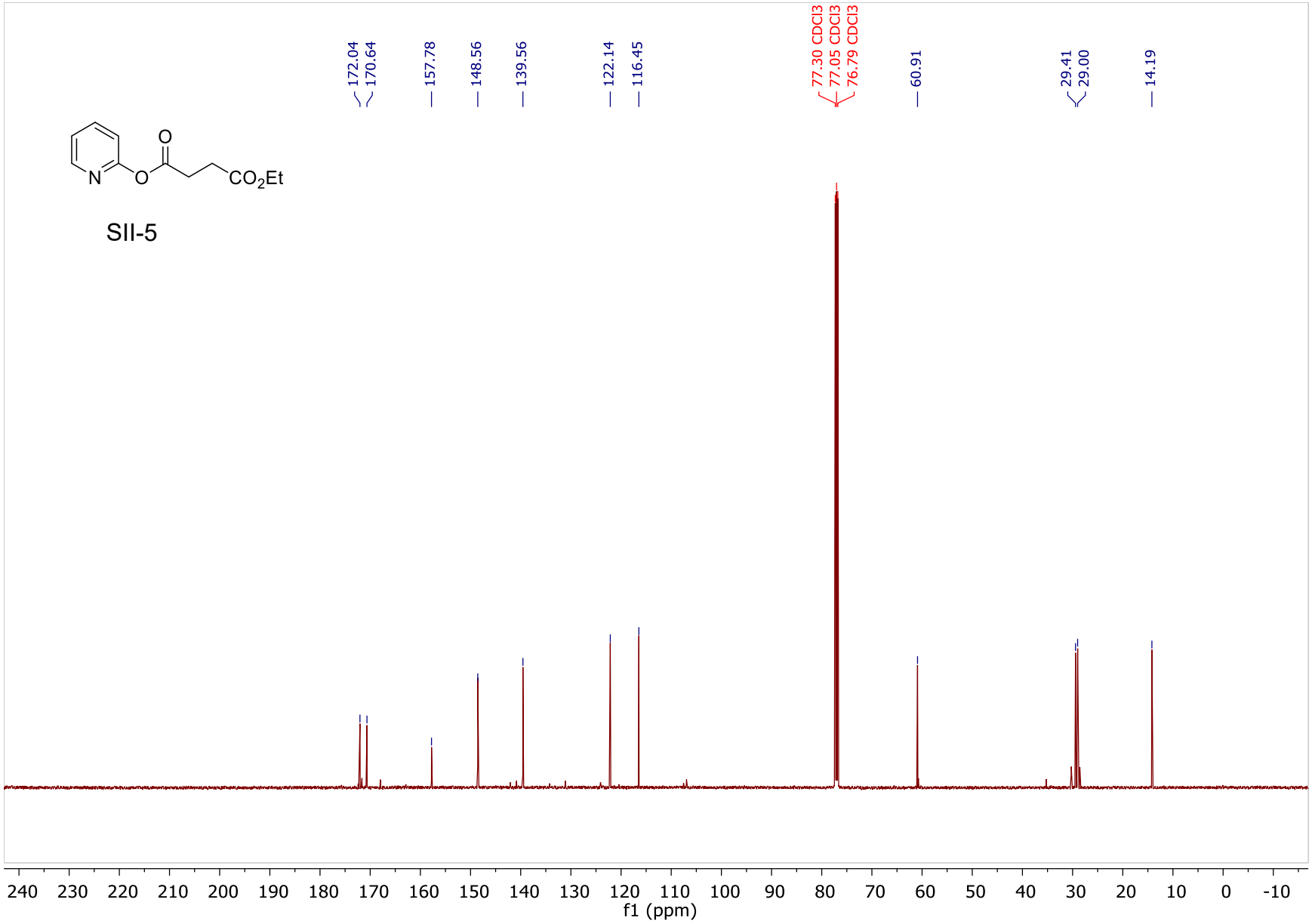

165.68
162.78
154.53
79.99
77.29 CDCl3
77.03 CDCl3
76.78 CDCl3
42.54
39.73
39.34
28.40
27.09
0.00
O
F
BocN
SII-2
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

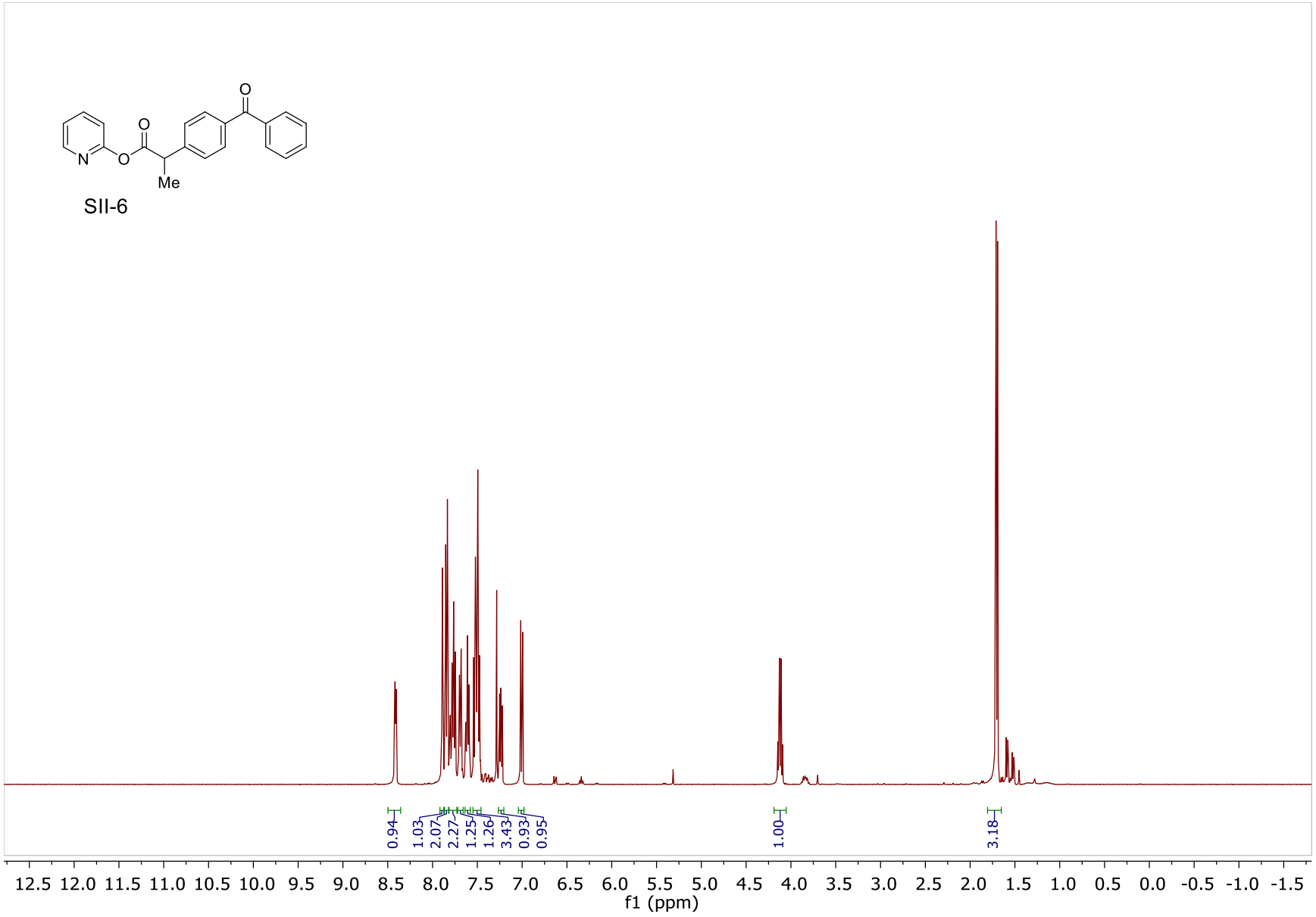

37.25
O
F
BocN
SII-2
110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110
f1 (ppm)

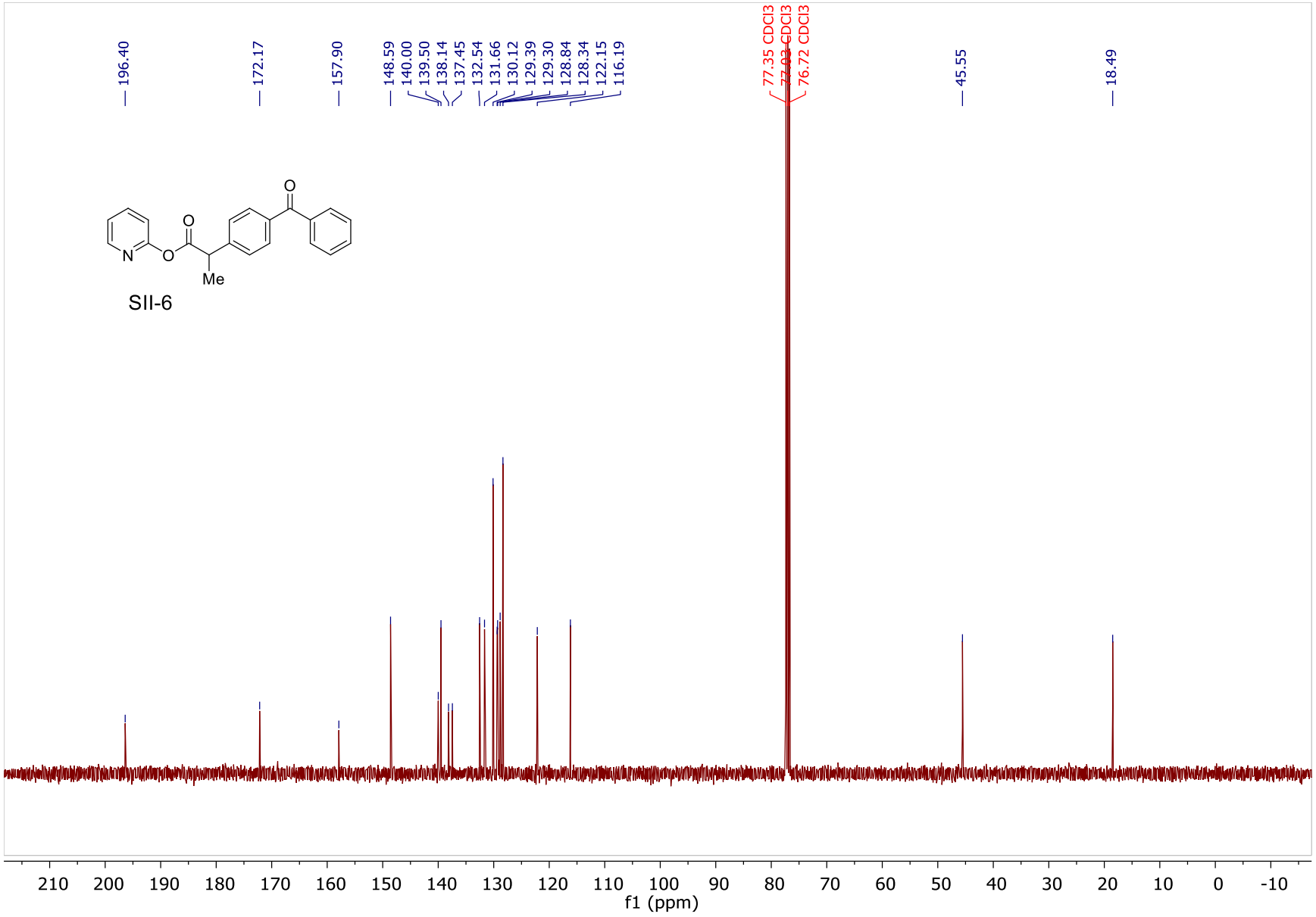

O
N
O
CO2Et

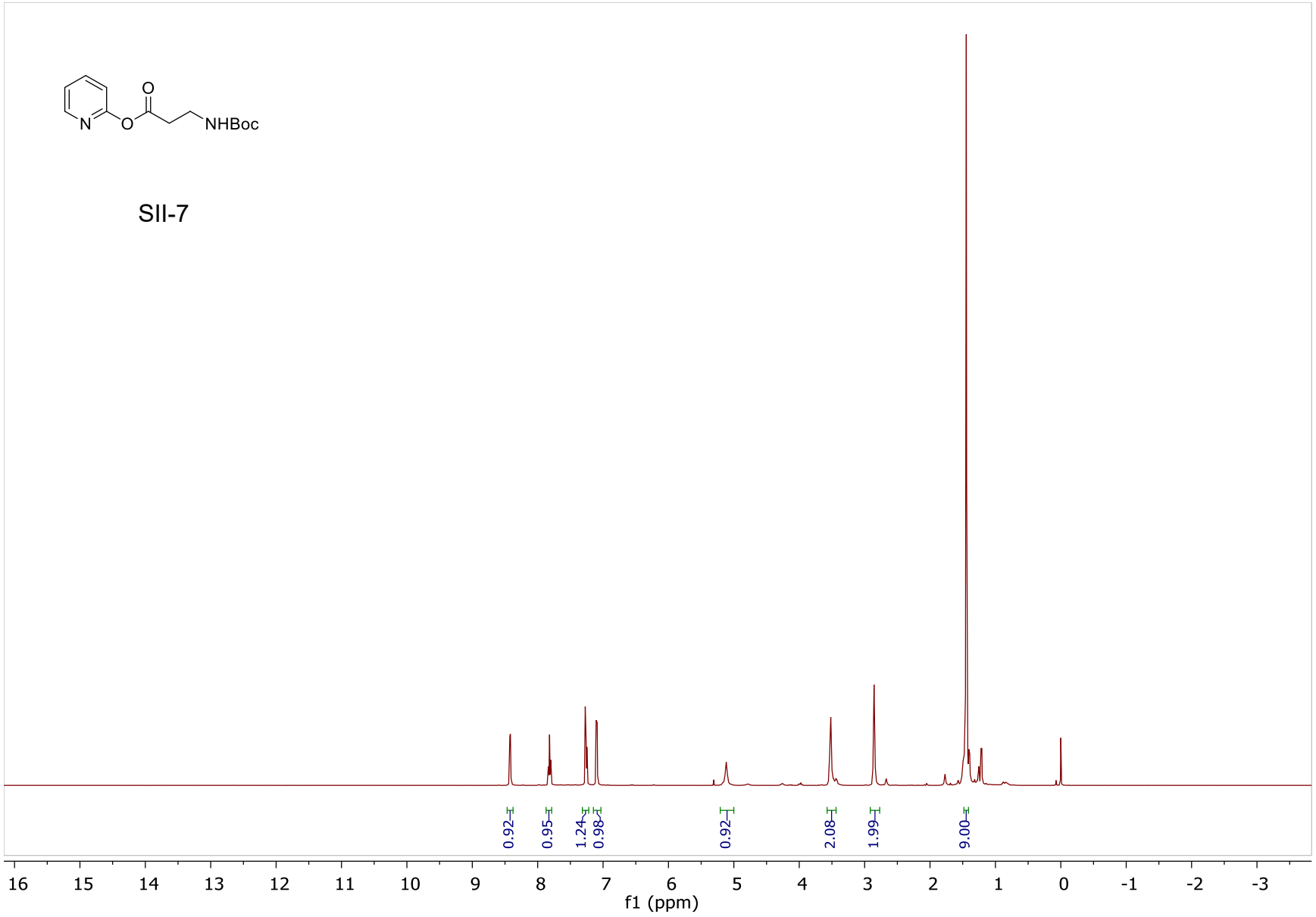
SII-5

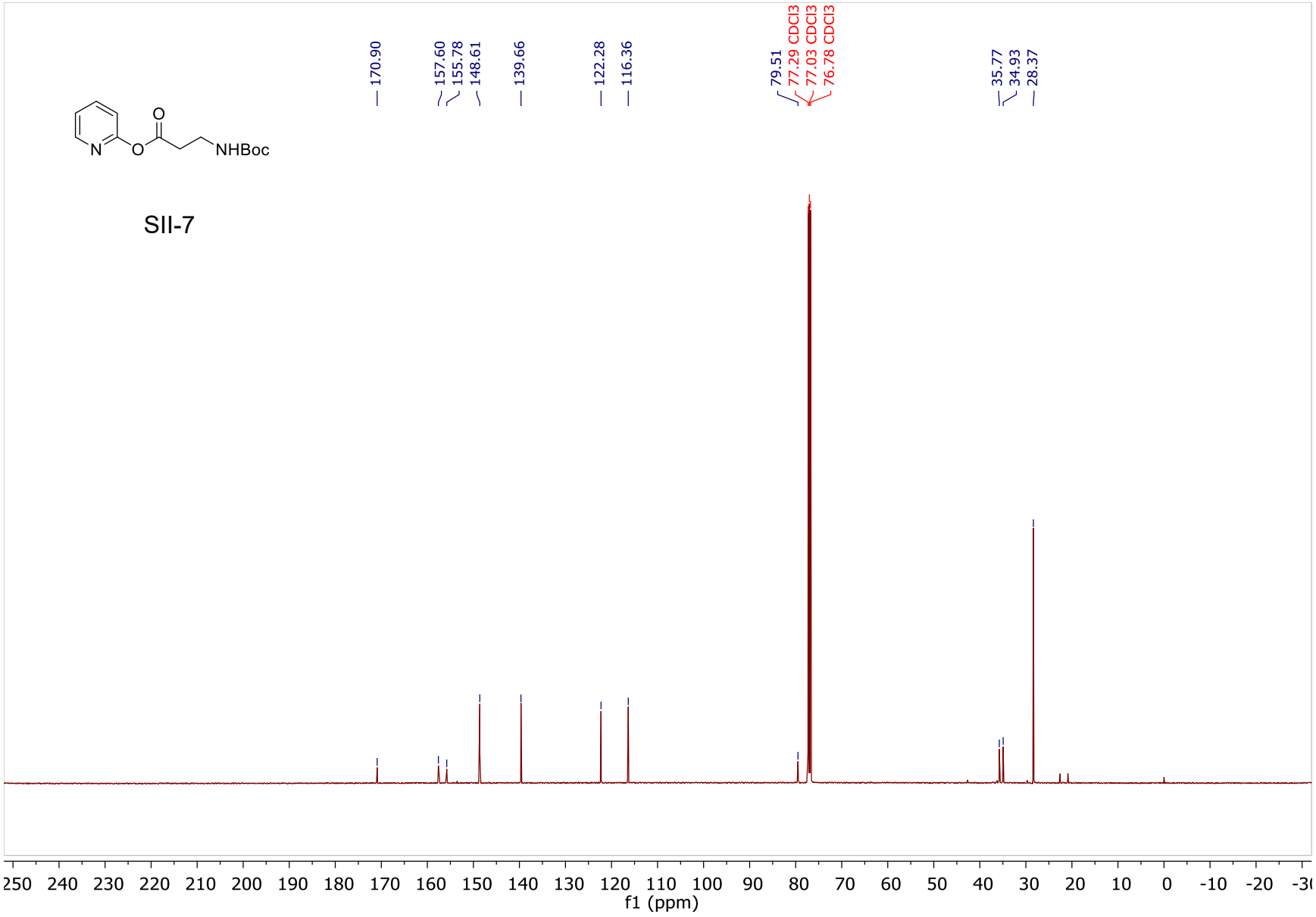

1.00
1.04
1.22
1.02
2.01
2.12
2.24
2.93
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

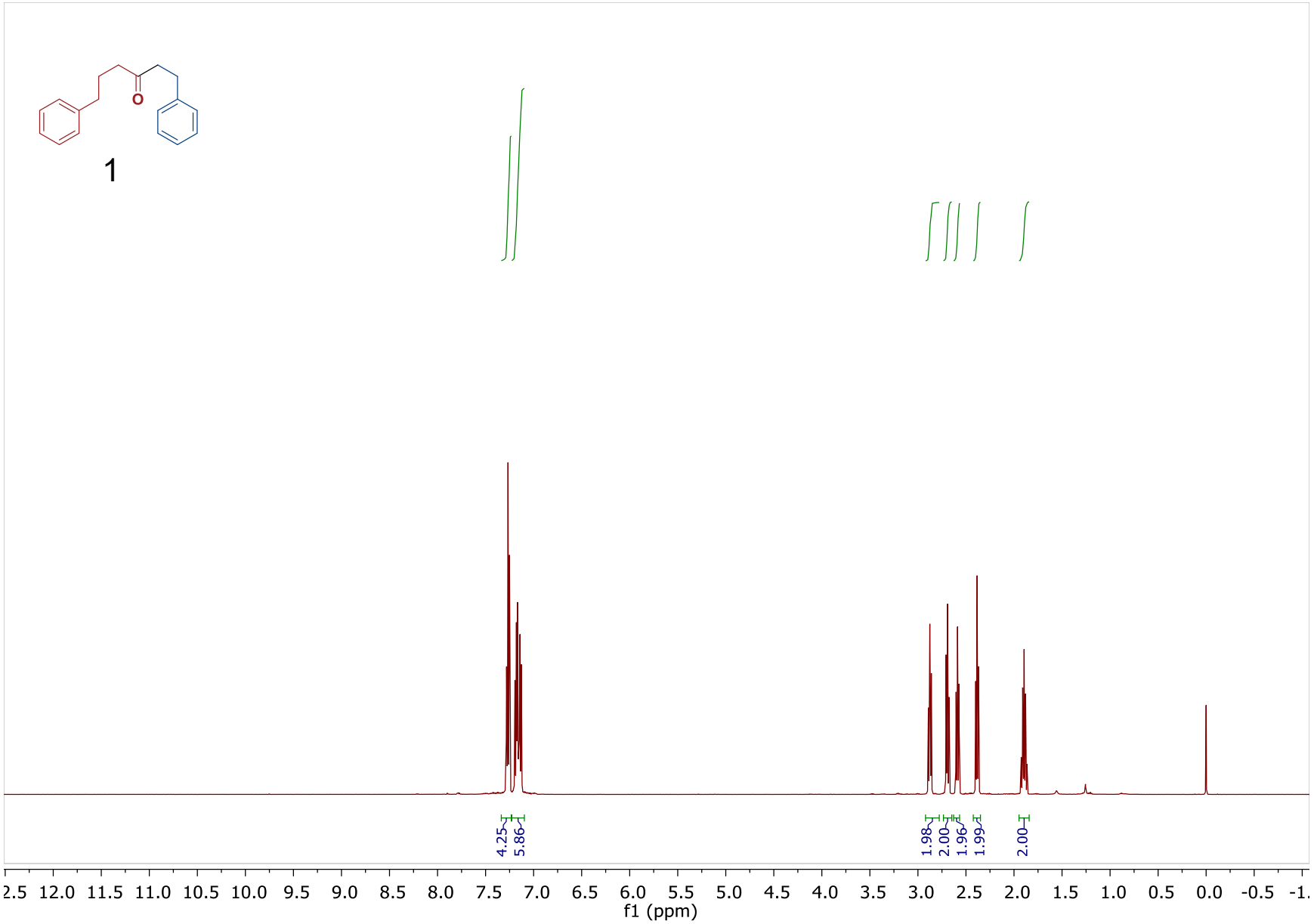

172.04
170.64
157.78
148.56
139.56
122.14
116.45
77.30 CDCl3
77.05 CDCl3
76.79 CDCl3
60.91
29.41
29.00
14.19
O
N
O
CO2Et
SII-5
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

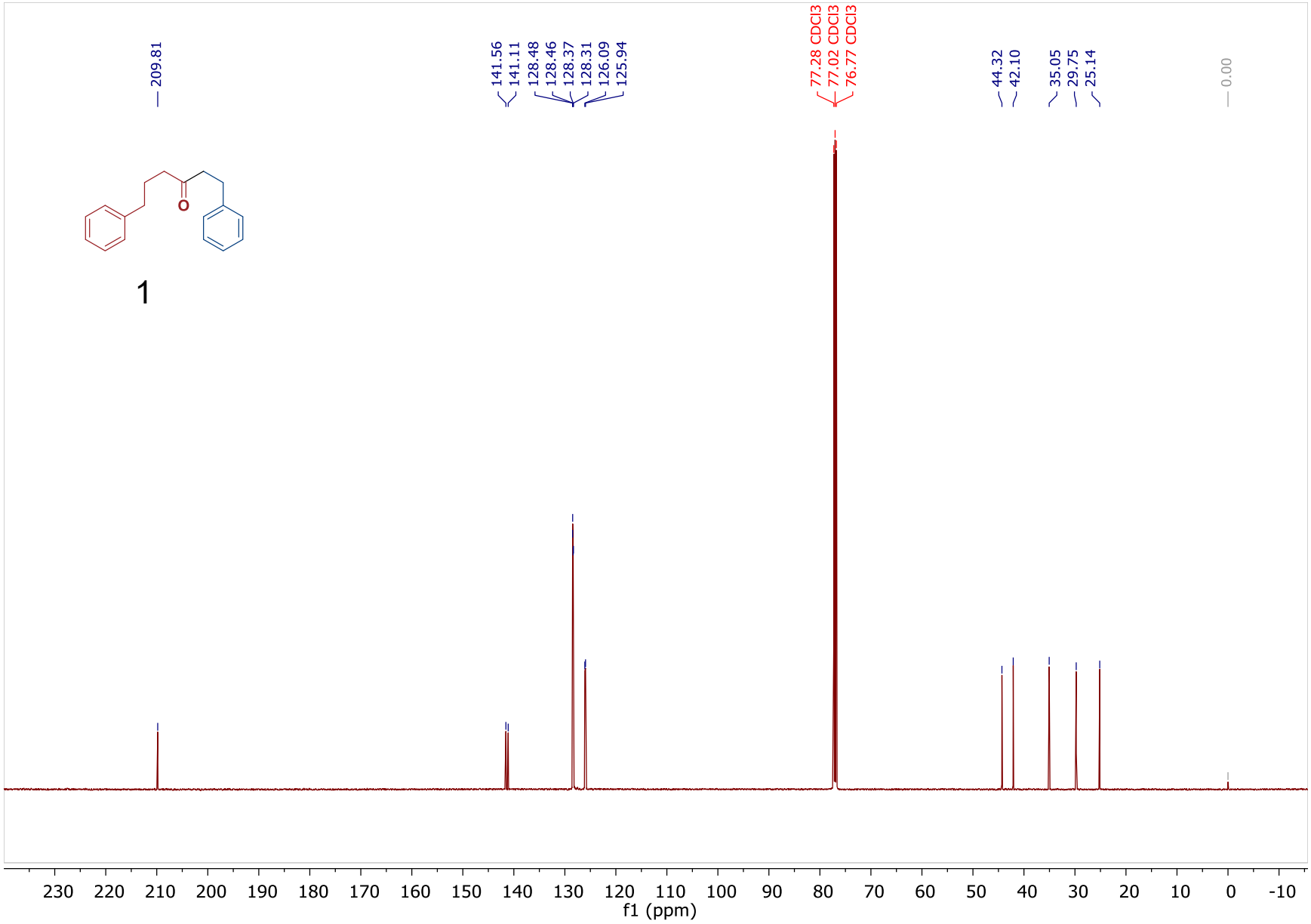

O
O
N
O
Me
SII-6
0.94
1.03
2.07
2.27
1.25
1.26
3.43
0.93
0.95
1.00
3.18
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

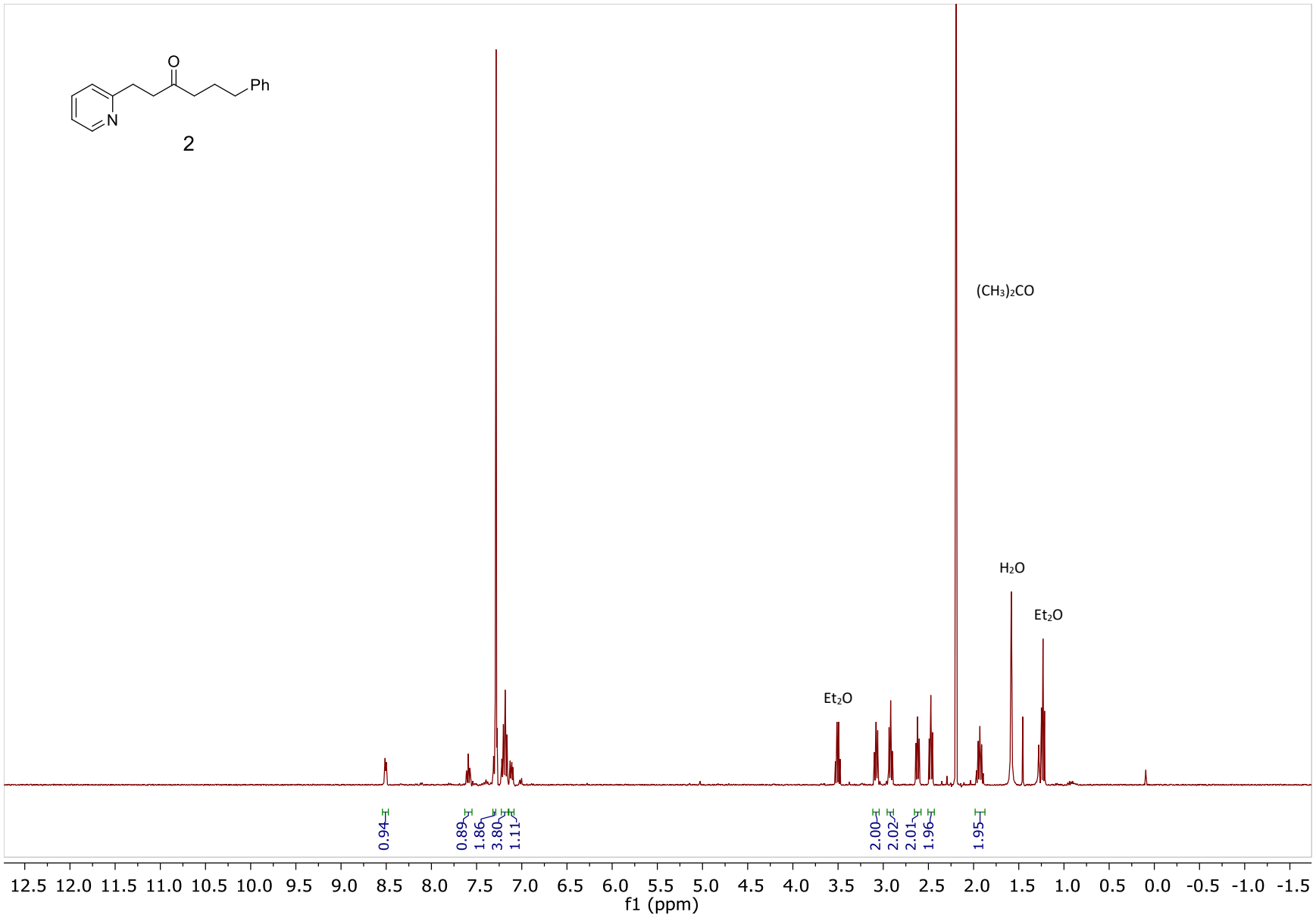

196.40
172.17
157.90
148.59
140.00
139.50
138.14
137.45
132.54
131.66
130.12
129.39
129.30
128.84
128.34
122.15
116.19
77.35 CDCl3
77.03 CDCl3
76.72 CDCl3
45.55
18.49
O
O
N
O
Me
SII-6
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

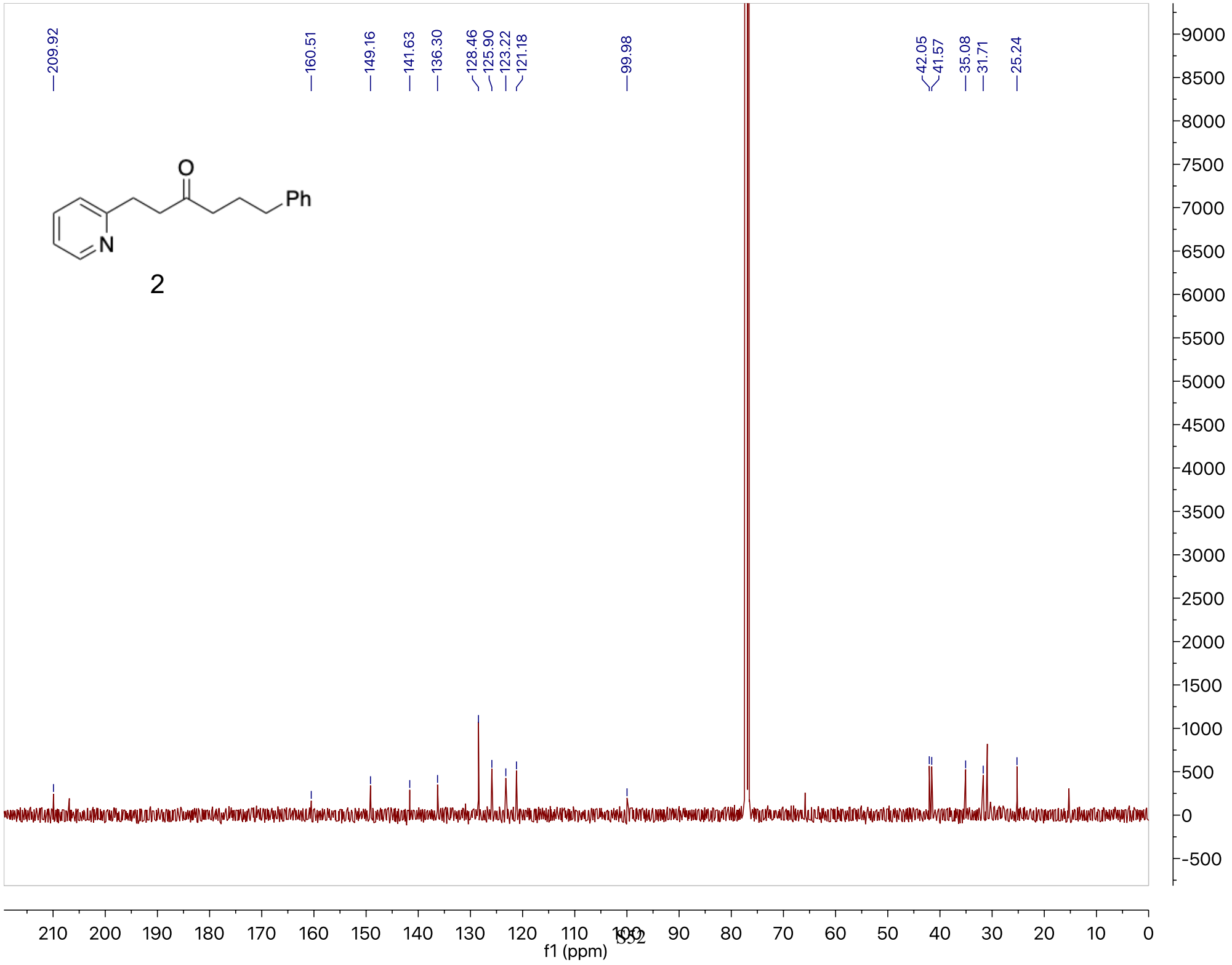

O
N
O
NHBoc
SII-7
0.92
0.95
1.24
0.98
0.92
2.08
1.99
9.00
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)


170.90
157.60
155.78
148.61
139.66
122.28
116.36
79.51
77.29 CDCl3
77.03 CDCl3
76.78 CDCl3
35.77
34.93
28.37
O
N
O
NHBoc
SII-7
250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30
f1 (ppm)


O
1
4.25
5.86
1.98
2.00
1.96
1.99
2.00
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.
f1 (ppm)


209.81
141.56
141.11
128.48
128.46
128.37
128.31
126.09
125.94
77.28 CDCl3
77.02 CDCl3
76.77 CDCl3
44.32
42.10
35.05
29.75
25.14
0.00
O
1
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)


O
Ph
N
2
(CH₃)₂CO
H₂O
Et₂O
Et₂O
0.94
0.89
1.86
3.80
1.11
2.00
2.02
2.01
1.96
1.95
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)


209.92
160.51
149.16
141.63
136.30
128.46
125.90
123.22
121.18
99.98
42.05
41.57
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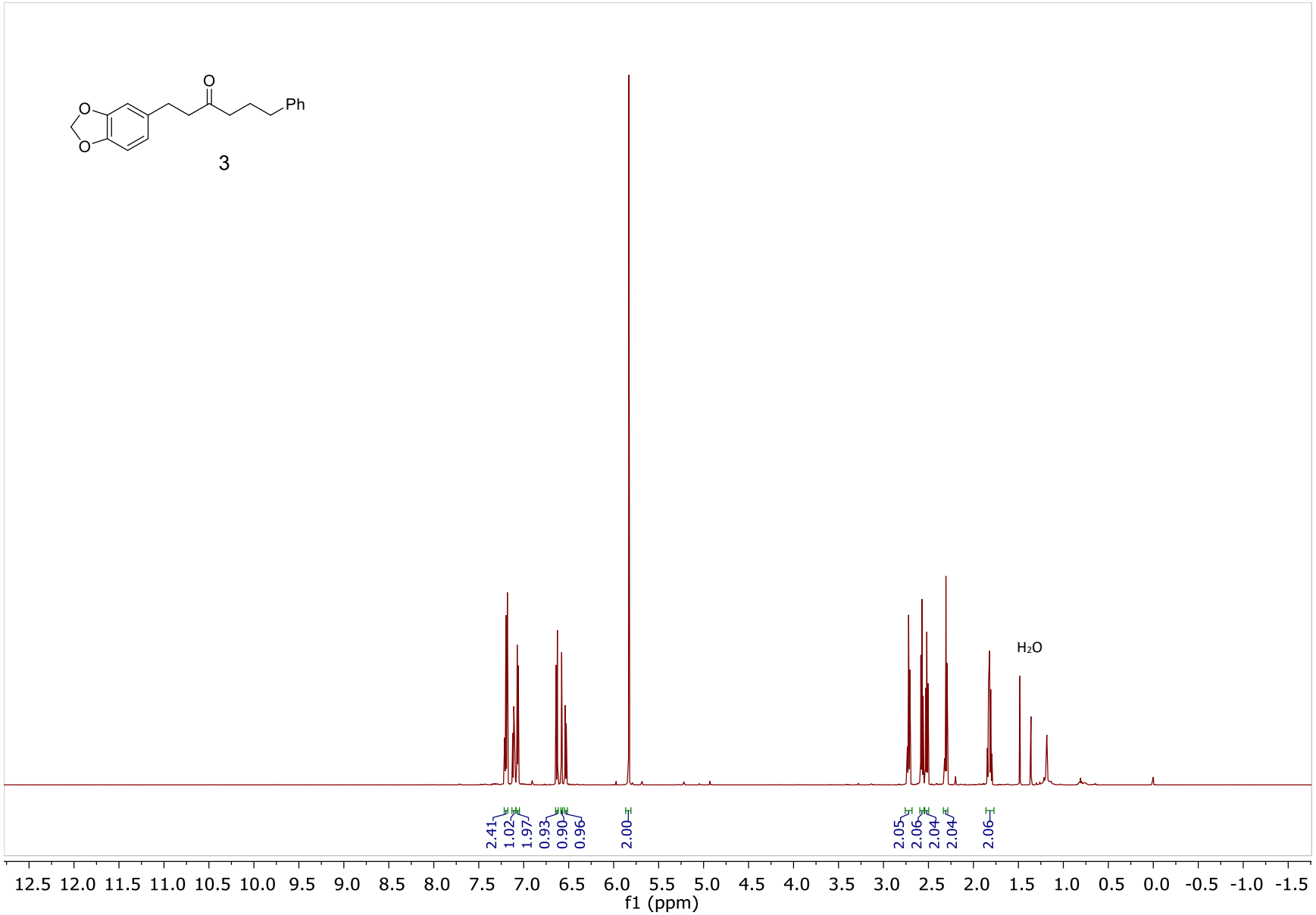
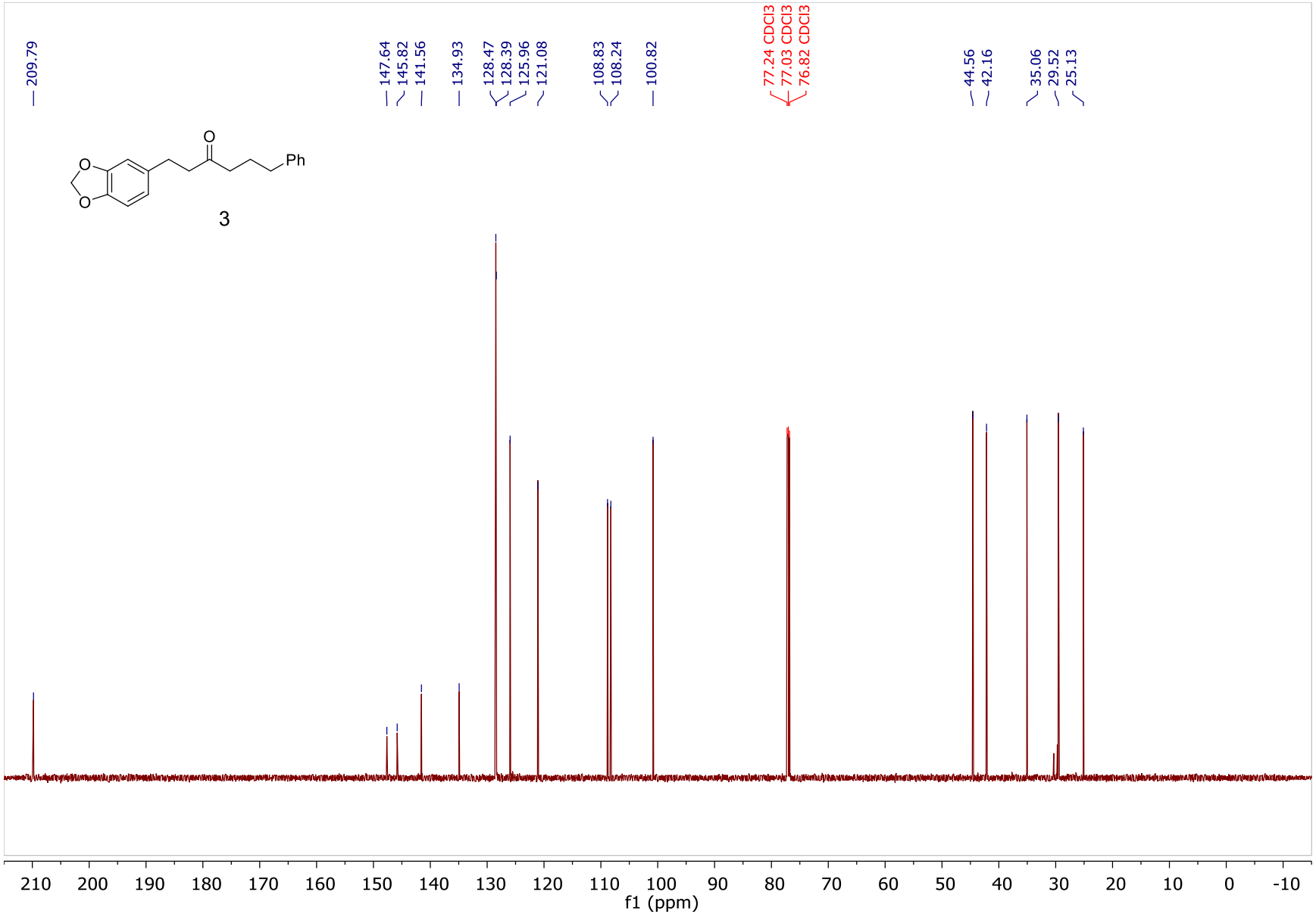
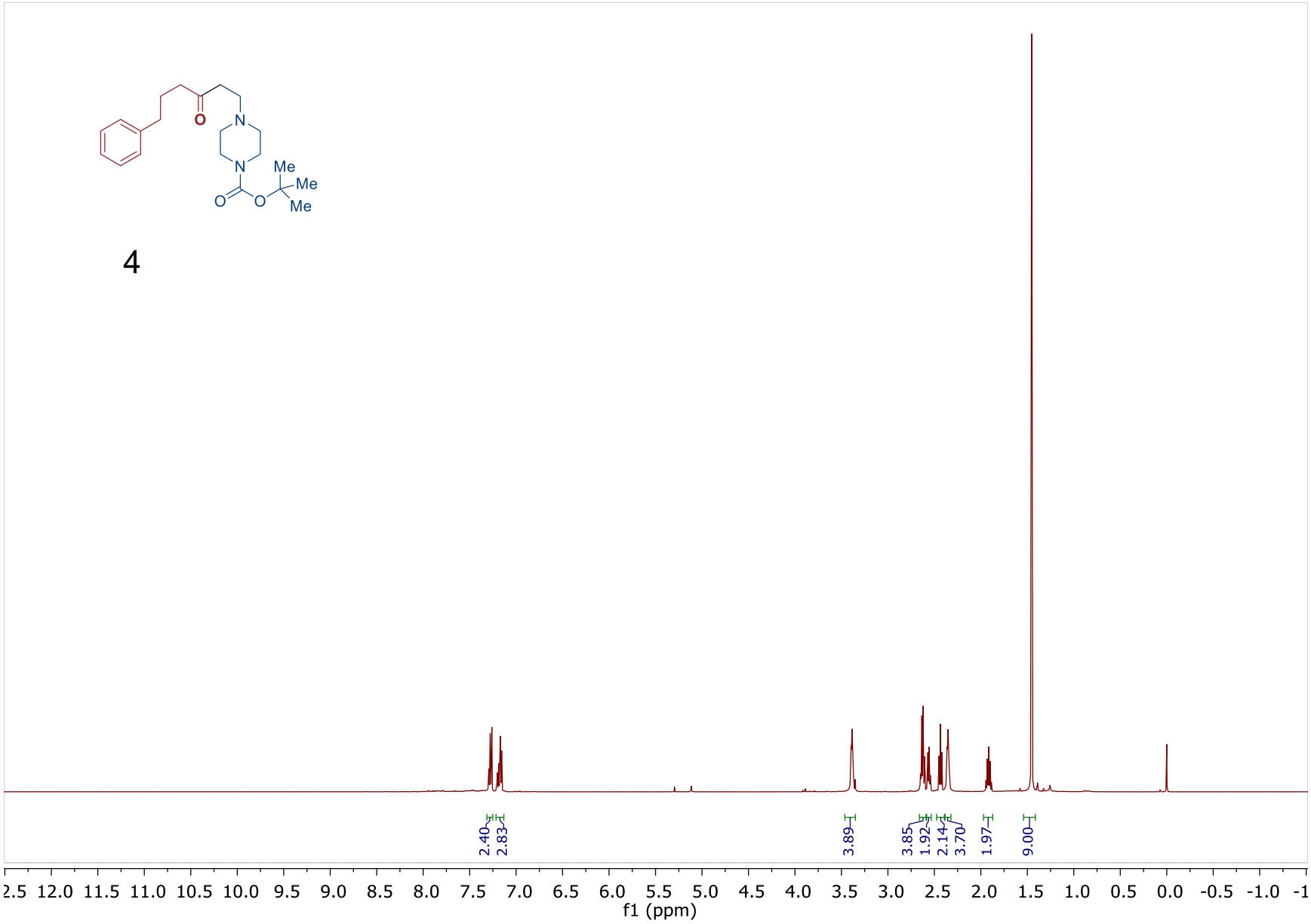
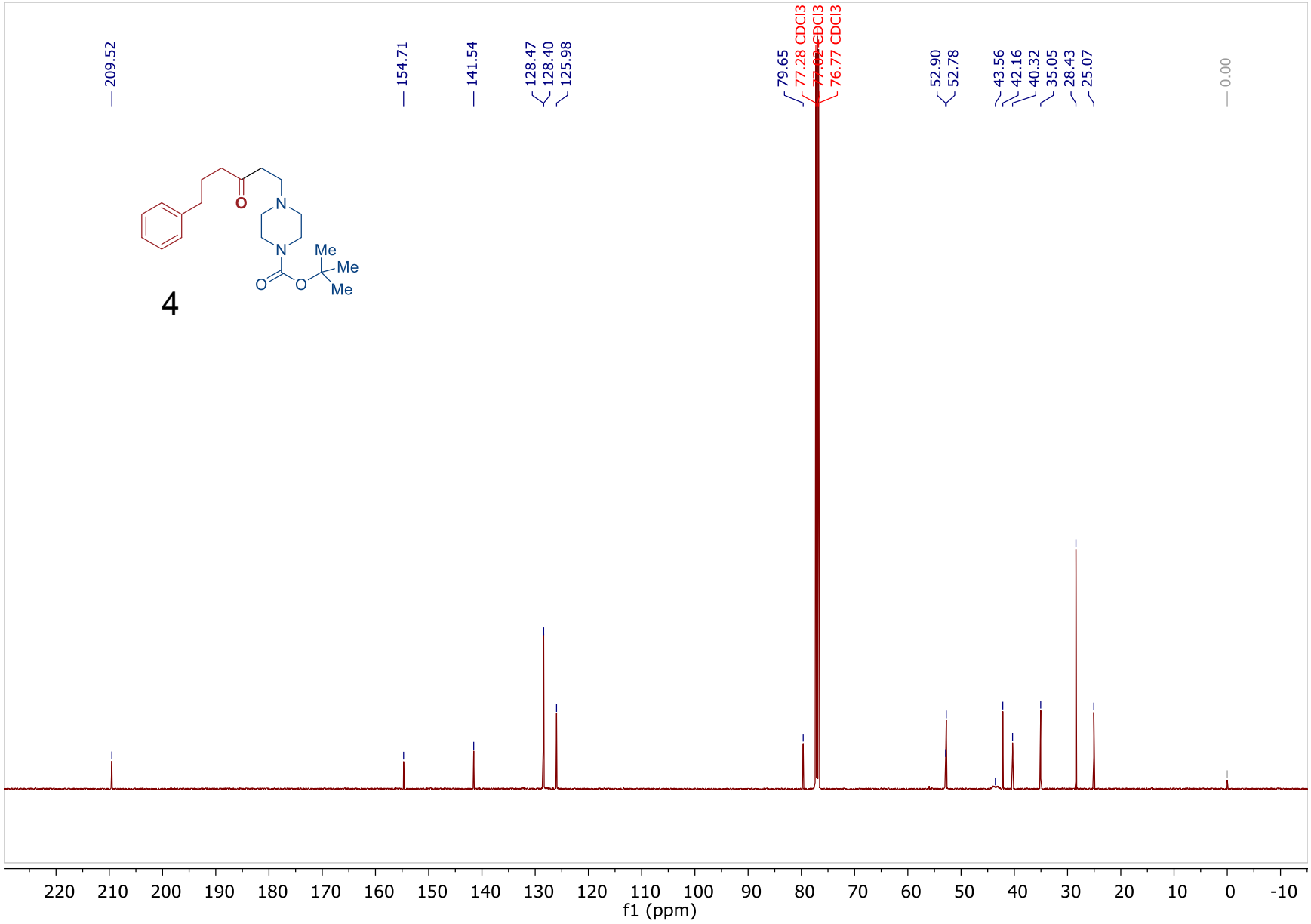
31.71
25.24
O
Ph
N
2
f1 (ppm)


O
O
O
Ph
3
H2O
2.41
1.02
1.97
0.93
0.90
0.96
2.00
2.05
2.06
2.04
2.04
2.06
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)


209.79
147.64
145.82
141.56
134.93
128.47
128.39
125.96
121.08
108.83
108.24
100.82
77.24 CDCl3
77.03 CDCl3
76.82 CDCl3
44.56
42.16
35.06
29.52
25.13
O
O
O
Ph
3
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)


O
N
N
Me
Me
Me
O
O
4
2.40
2.83
3.89
3.85
1.92
2.14
3.70
1.97
9.00
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1
f1 (ppm)


209.52
154.71
141.54
128.47
128.40
125.98
79.65
77.28 CDCl3
77.02 CDCl3
76.77 CDCl3
52.90
52.78
43.56
42.16
40.32
35.05
28.43
25.07
0.00
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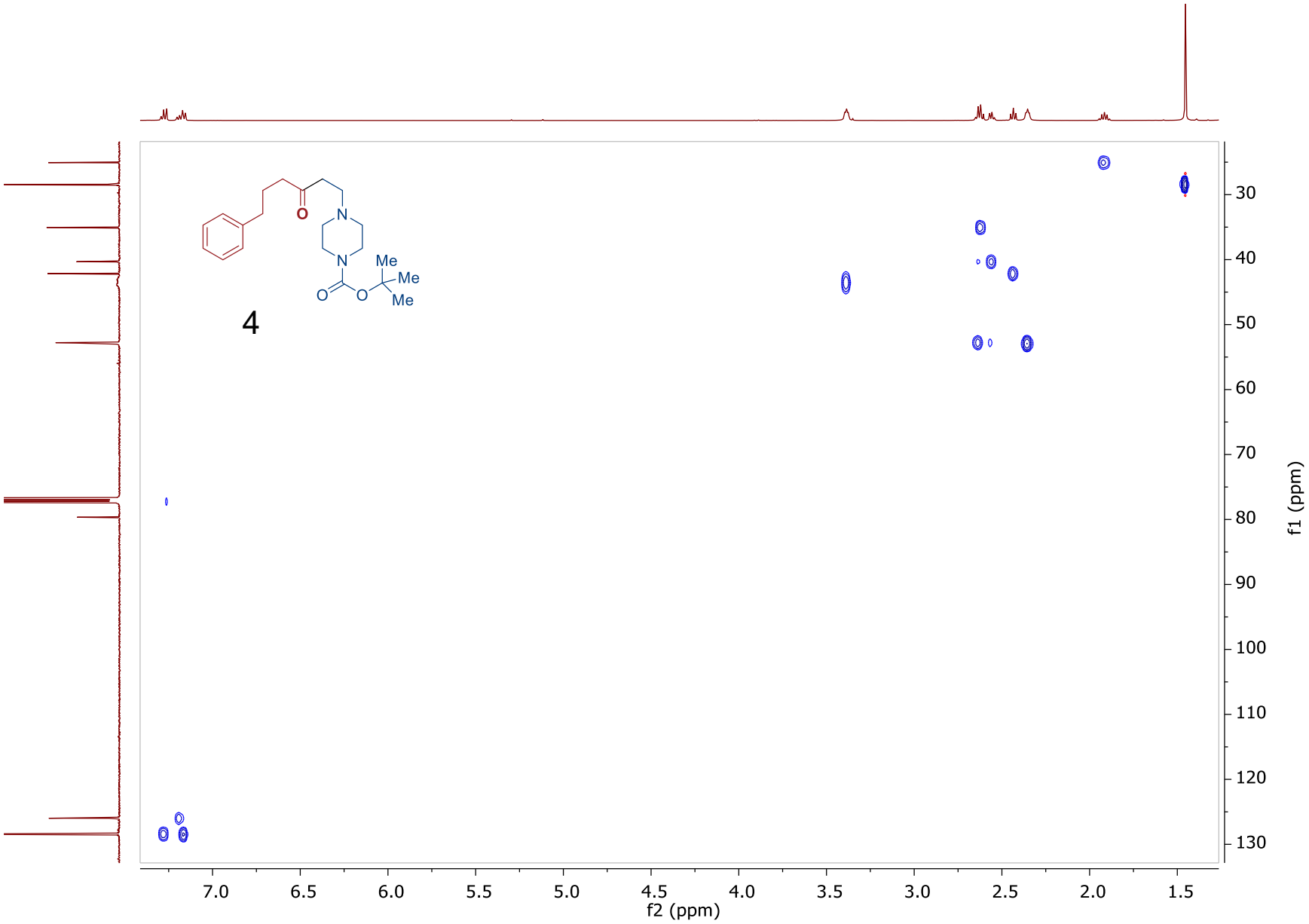
f1 (ppm)


O
N
N
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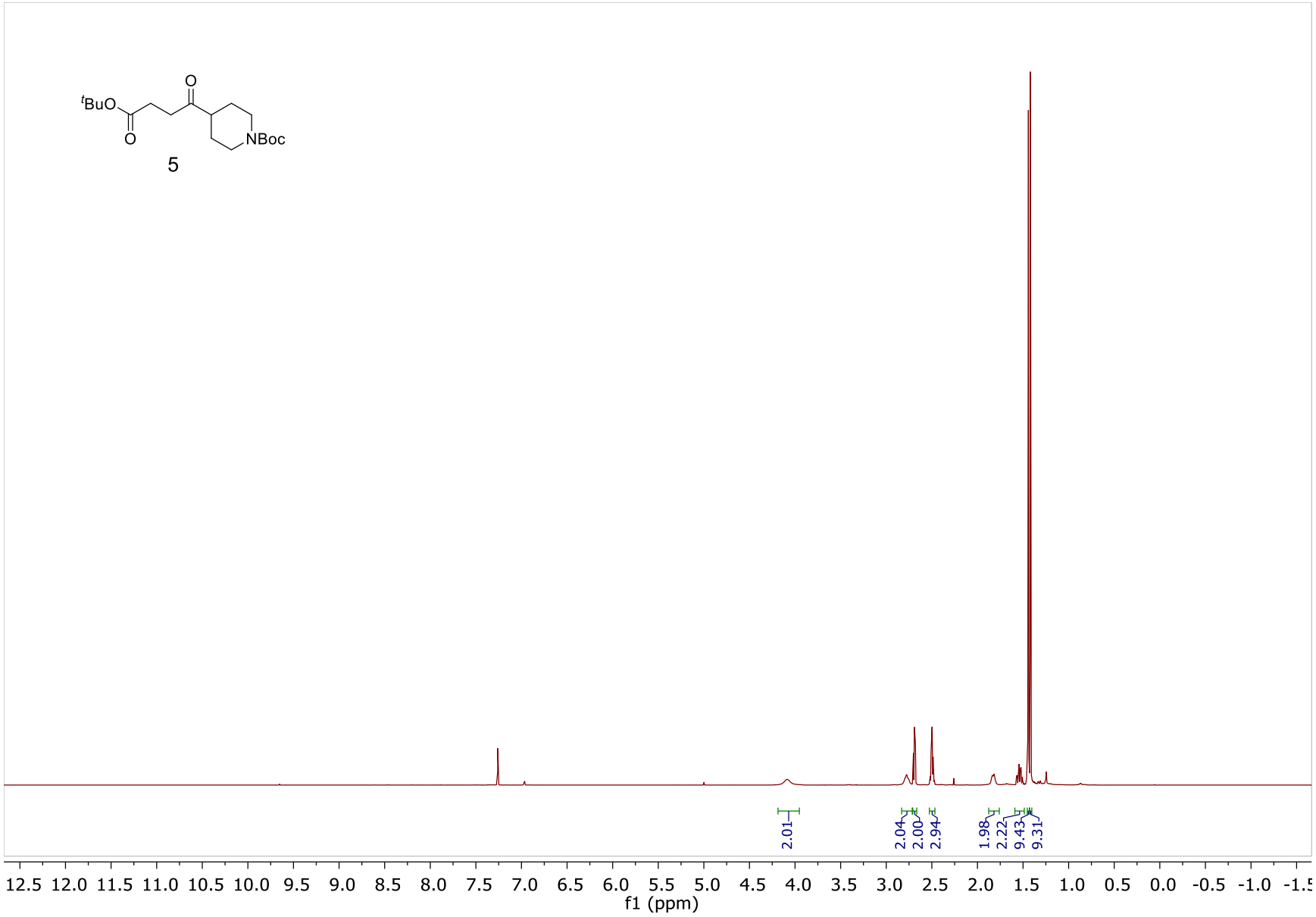
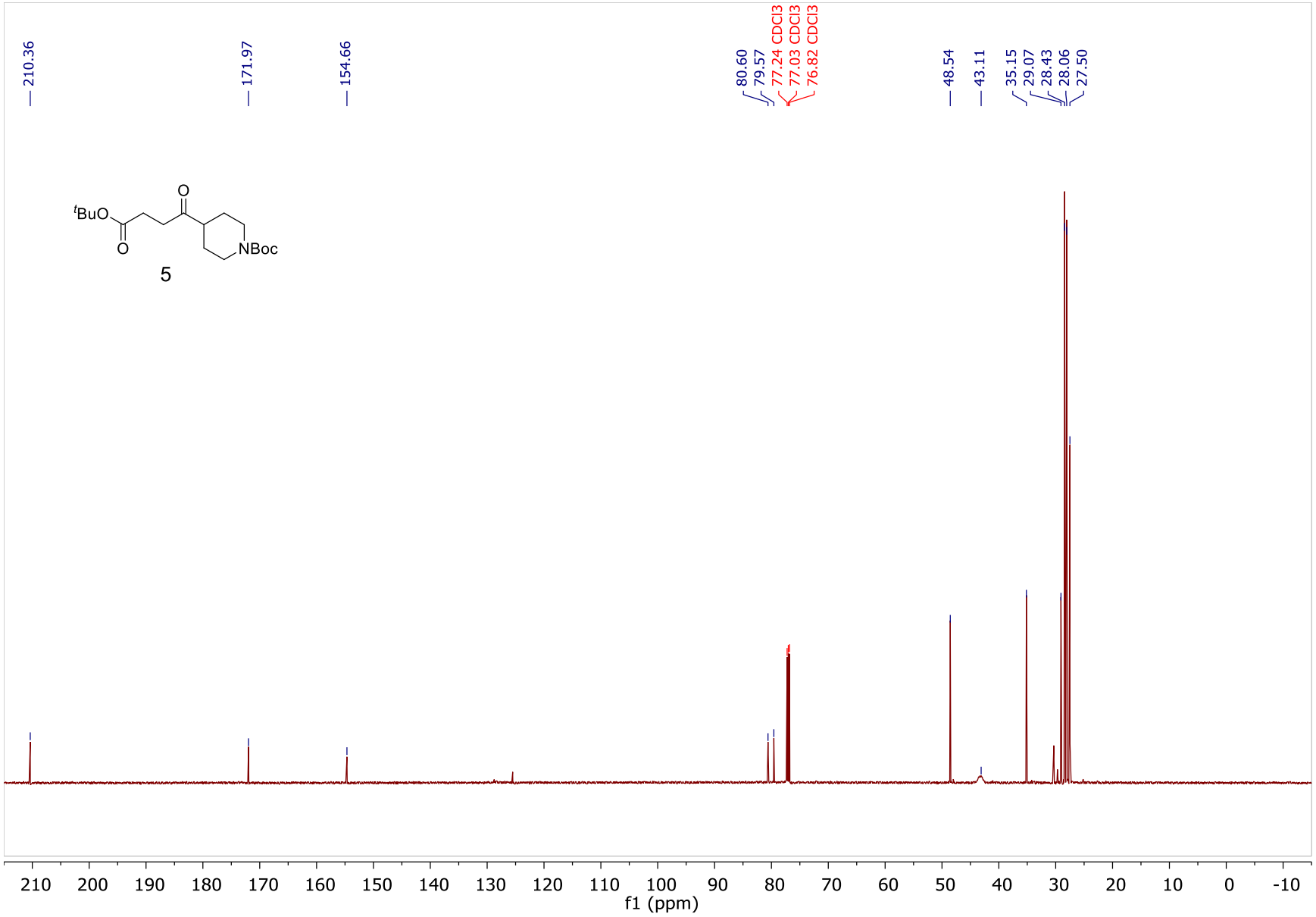
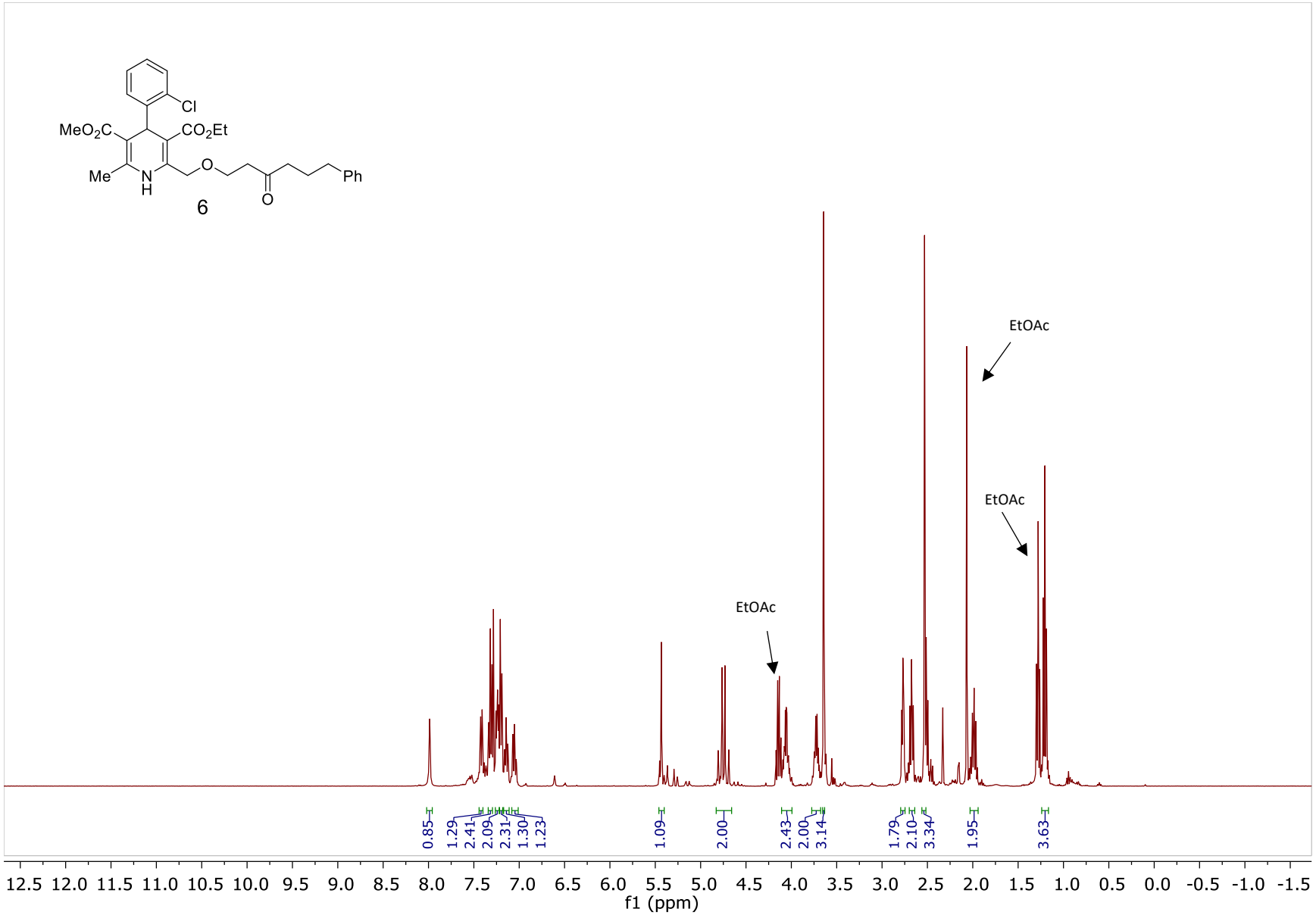
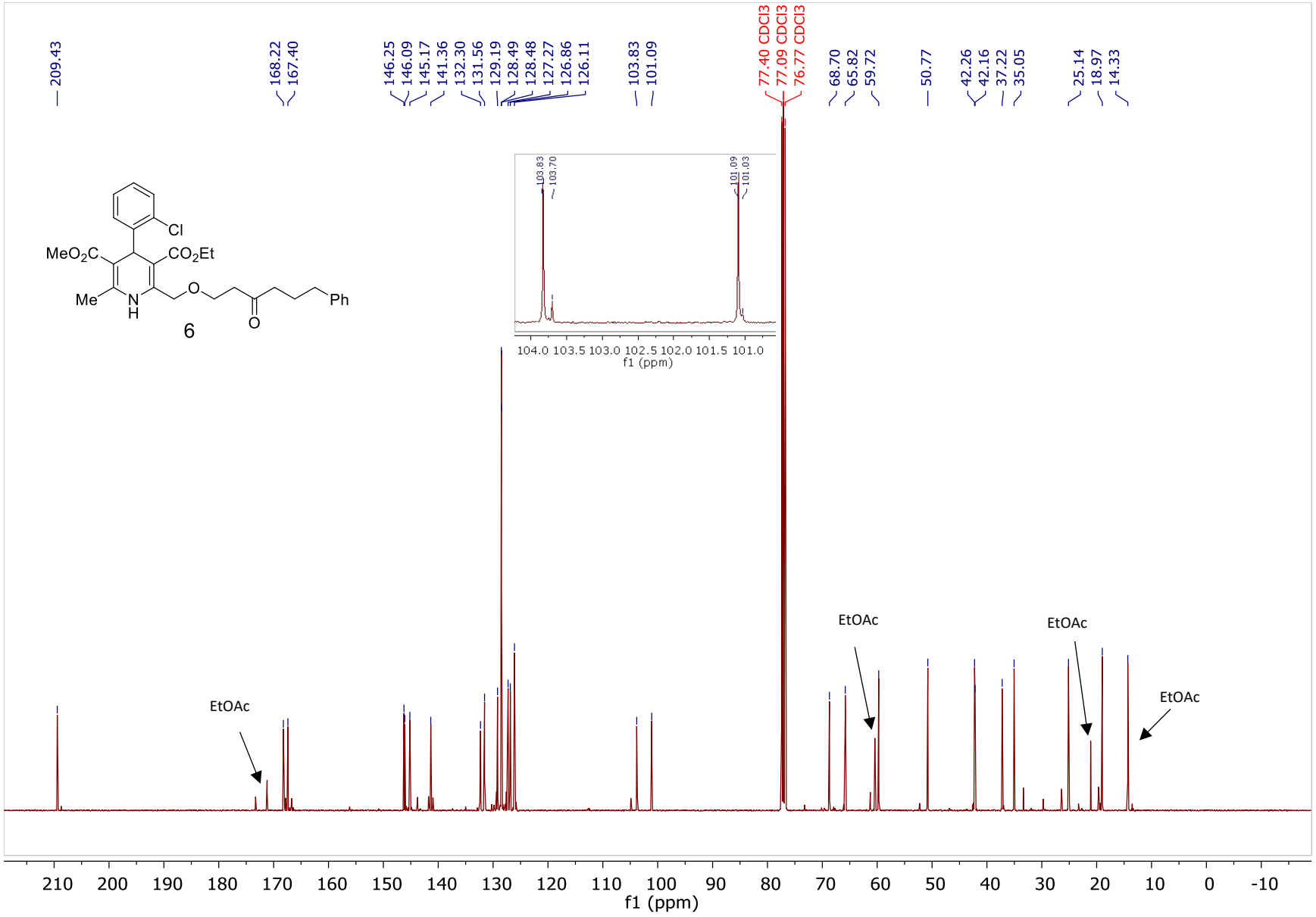
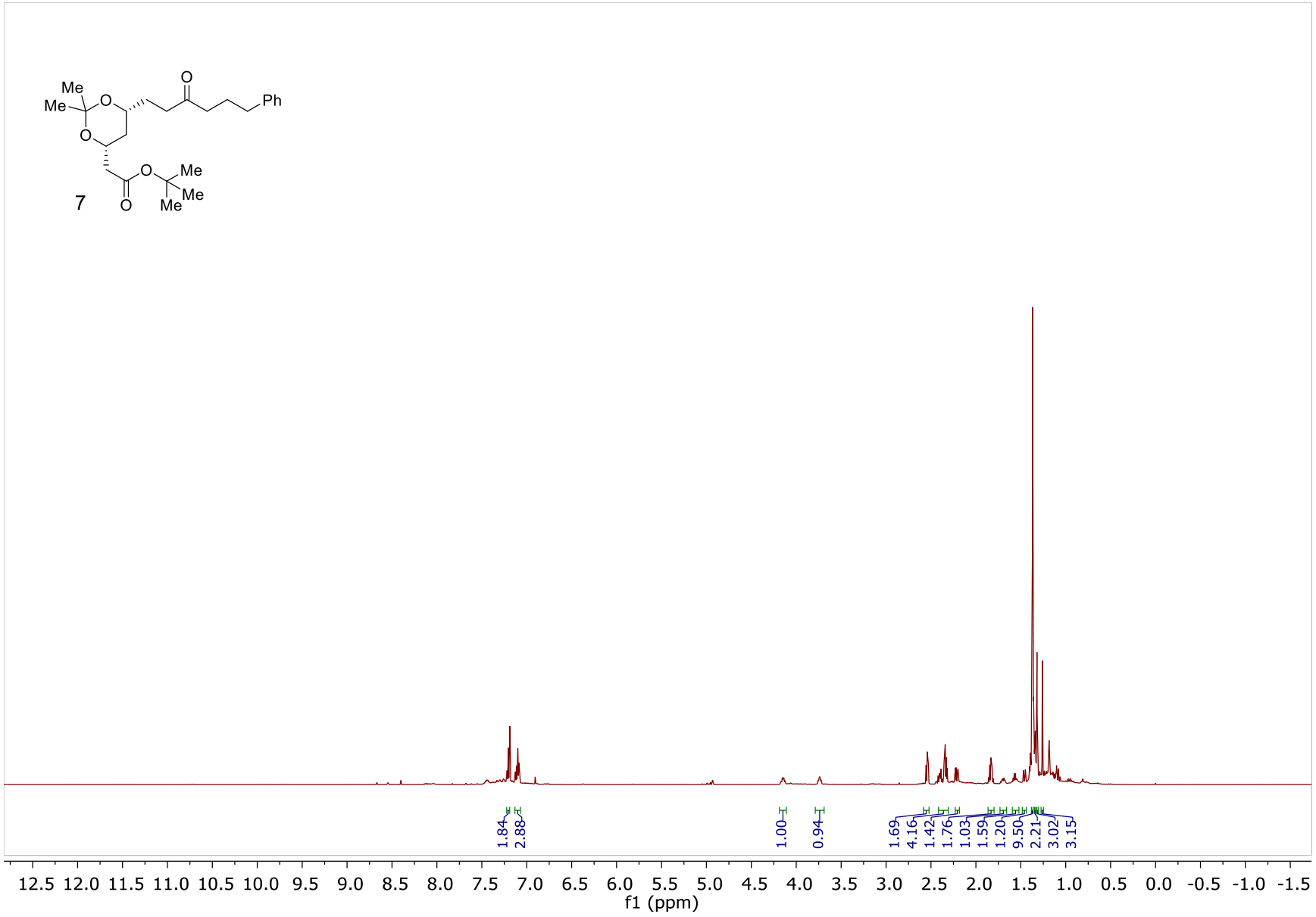
Me
Me
O
O
4
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
f2 (ppm)
30
40
50
60
70
80
90
100
110
120
130
f1 (ppm)

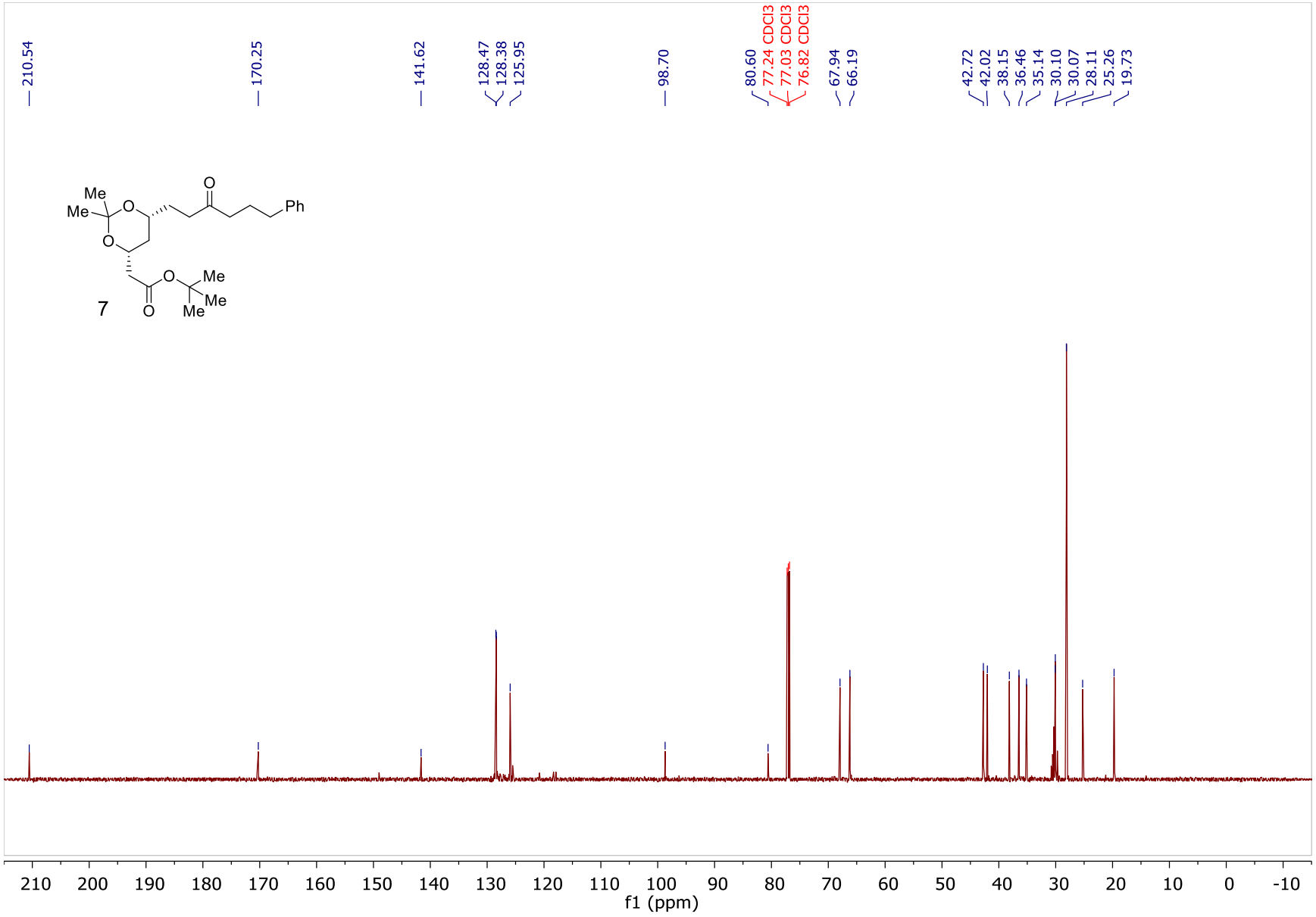

tBuO
O
O
NBoc
5
2.01
2.04
2.00
2.94
1.98
2.22
9.43
9.31
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

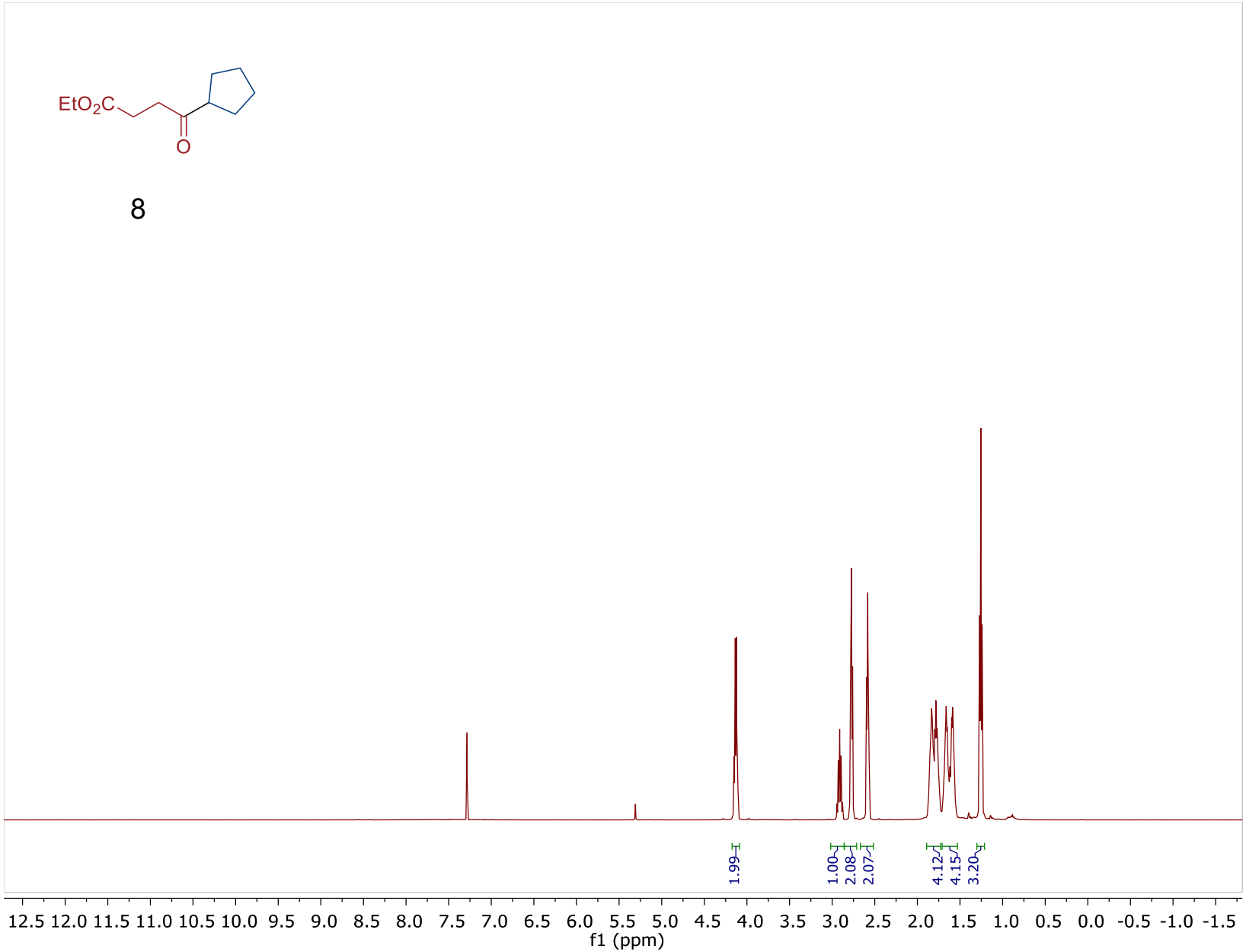

210.36
171.97
154.66
80.60
79.57
77.24 CDCl3
77.03 CDCl3
76.82 CDCl3
48.54
43.11
35.15
29.07
28.43
28.06
27.50
tBuO
O
O
O
NBoc
5
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

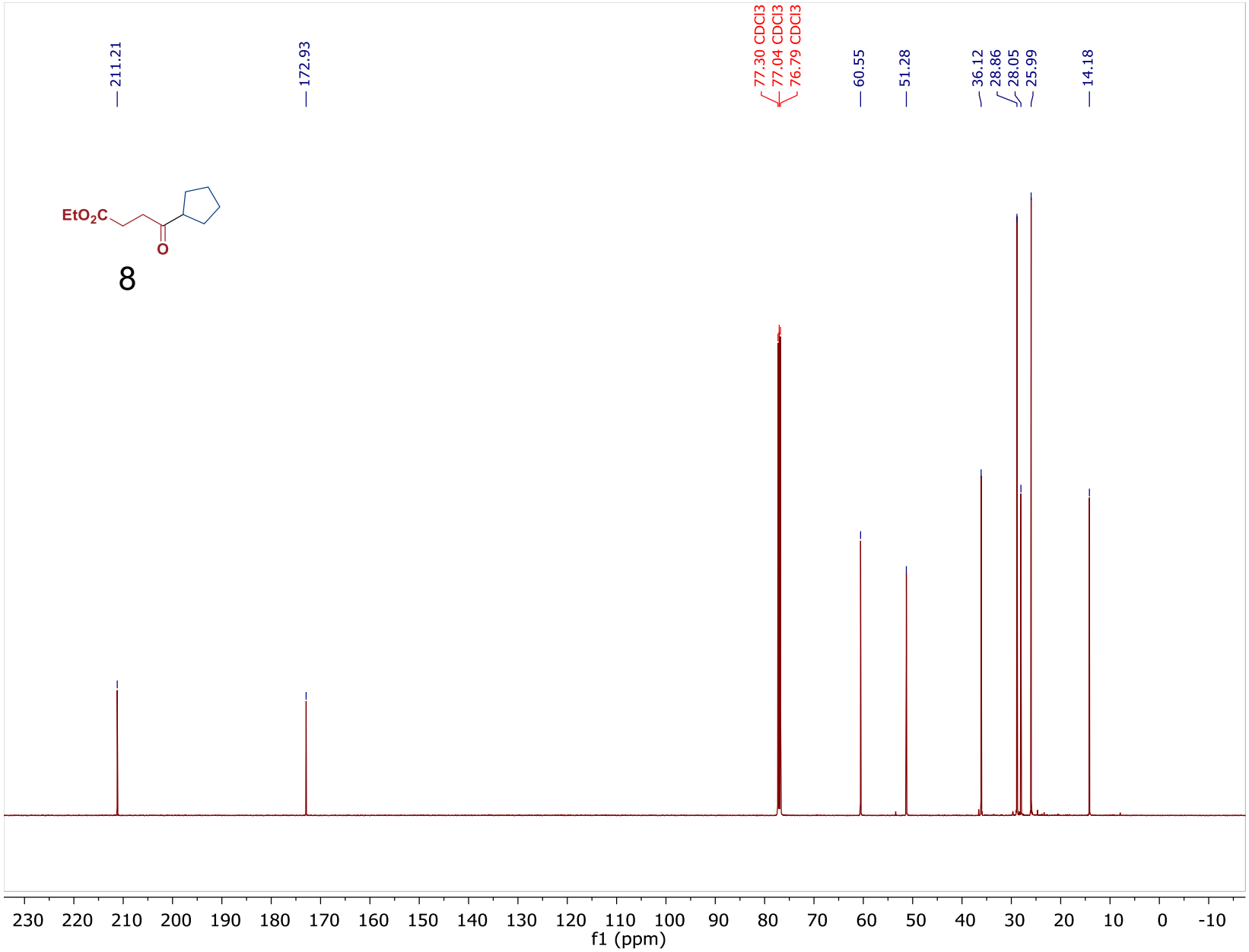

Cl
MeO2C
CO2Et
Me
N
H
O
O
Ph
6
EtOAc
EtOAc
EtOAc
0.85
1.29
2.41
2.09
2.31
1.30
1.23
1.09
2.00
2.43
2.00
3.14
1.79
2.10
3.34
1.95
3.63
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

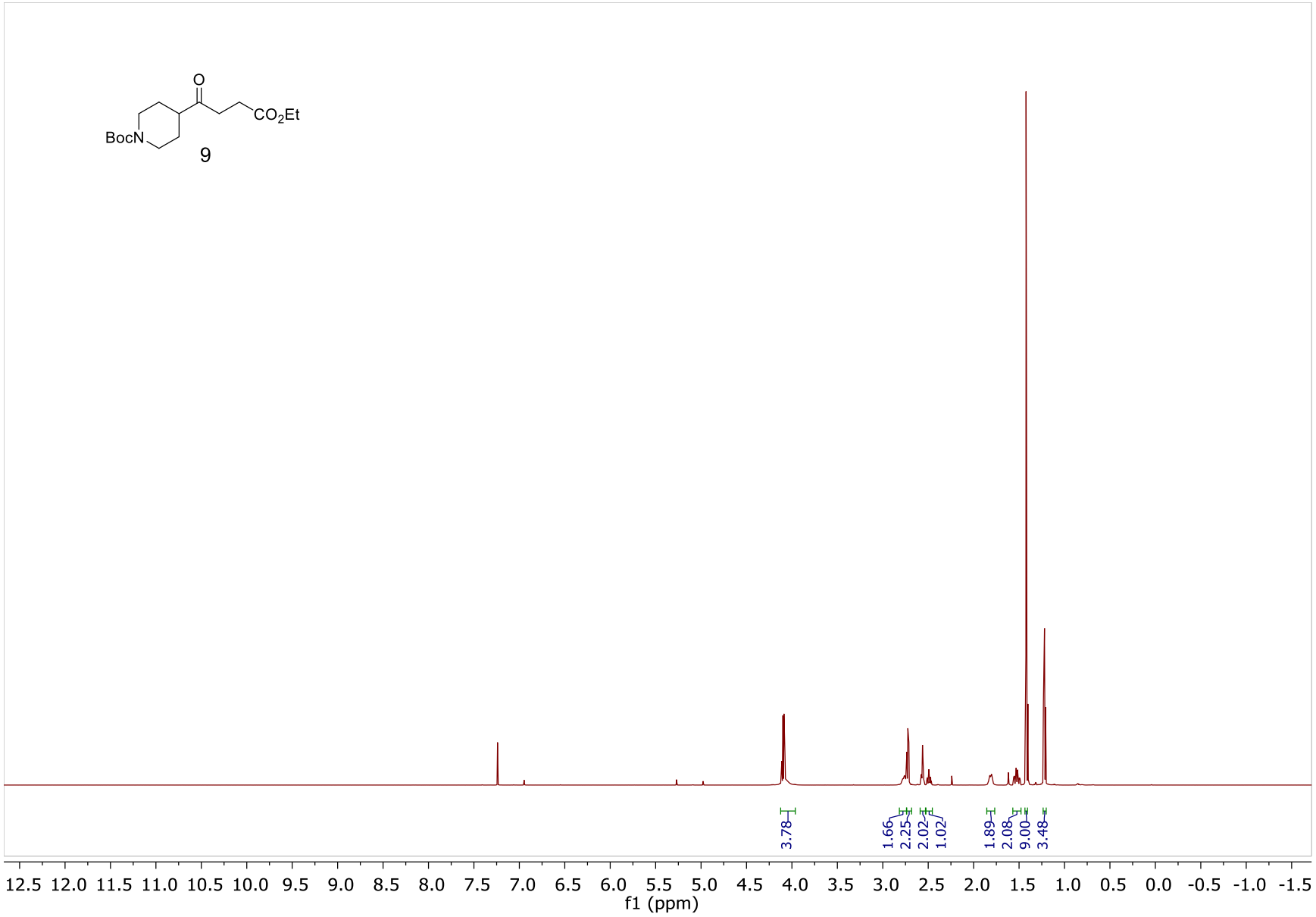

209.43
168.22
167.40
146.25
146.09
145.17
141.36
132.30
131.56
129.19
128.49
128.48
127.27
126.86
126.11
103.83
101.09
77.40 CDCl3
77.09 CDCl3
76.77 CDCl3
68.70
65.82
59.72
50.77
42.26
42.16
37.22
35.05
25.14
18.97
14.33
Cl
MeO2C
CO2Et
Me
N
H
O
Ph
O
6
103.83
103.70
101.09
101.03
104.0 103.5 103.0 102.5 102.0 101.5 101.0
f1 (ppm)
EtOAc
EtOAc
EtOAc
EtOAc
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)

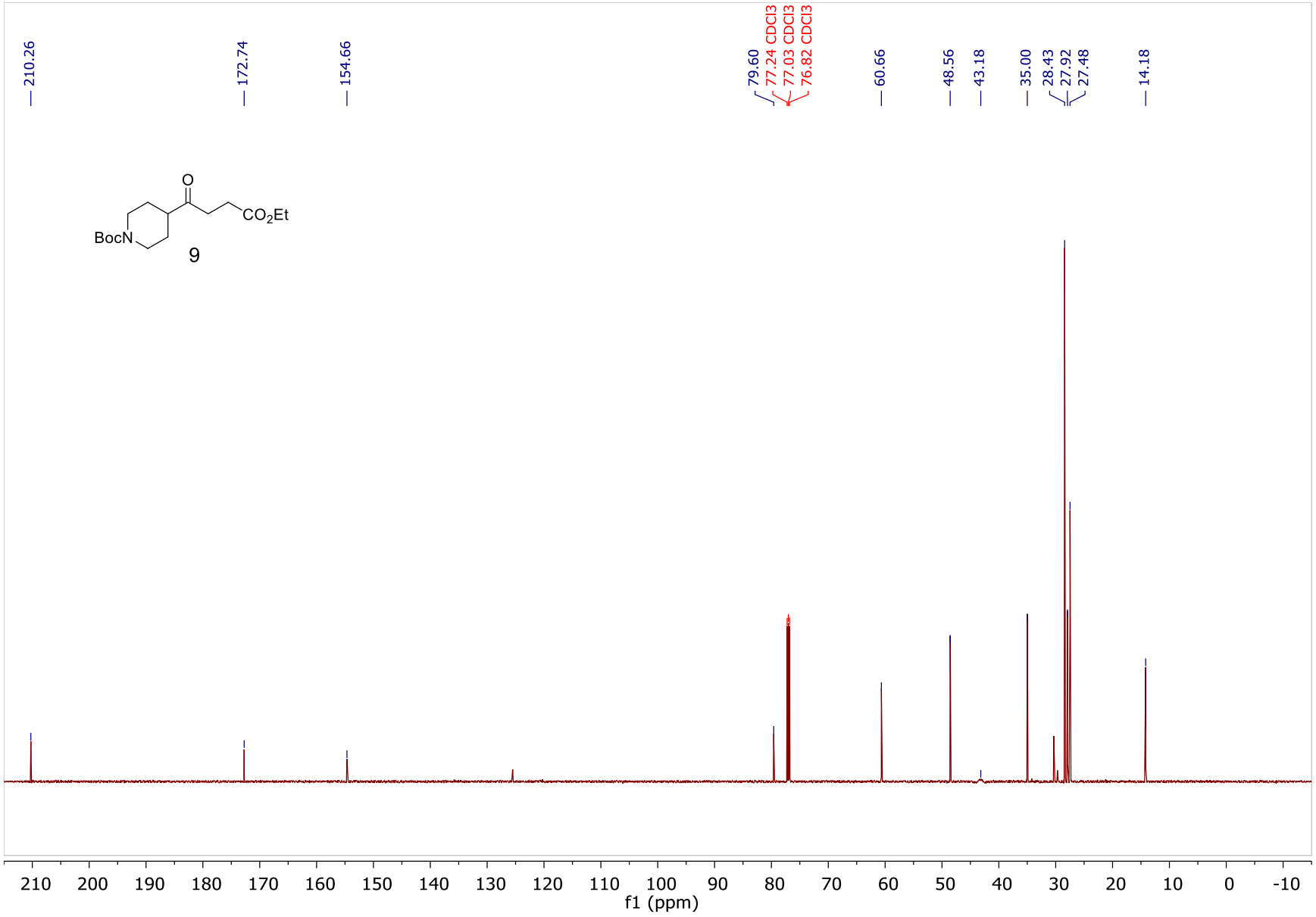

Me
Me
O
O
O
Ph
O
Me
Me
Me
O
7
1.84
2.88
1.00
0.94
1.69
4.16
1.42
1.76
1.03
1.59
1.20
9.50
2.21
3.02
3.15
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

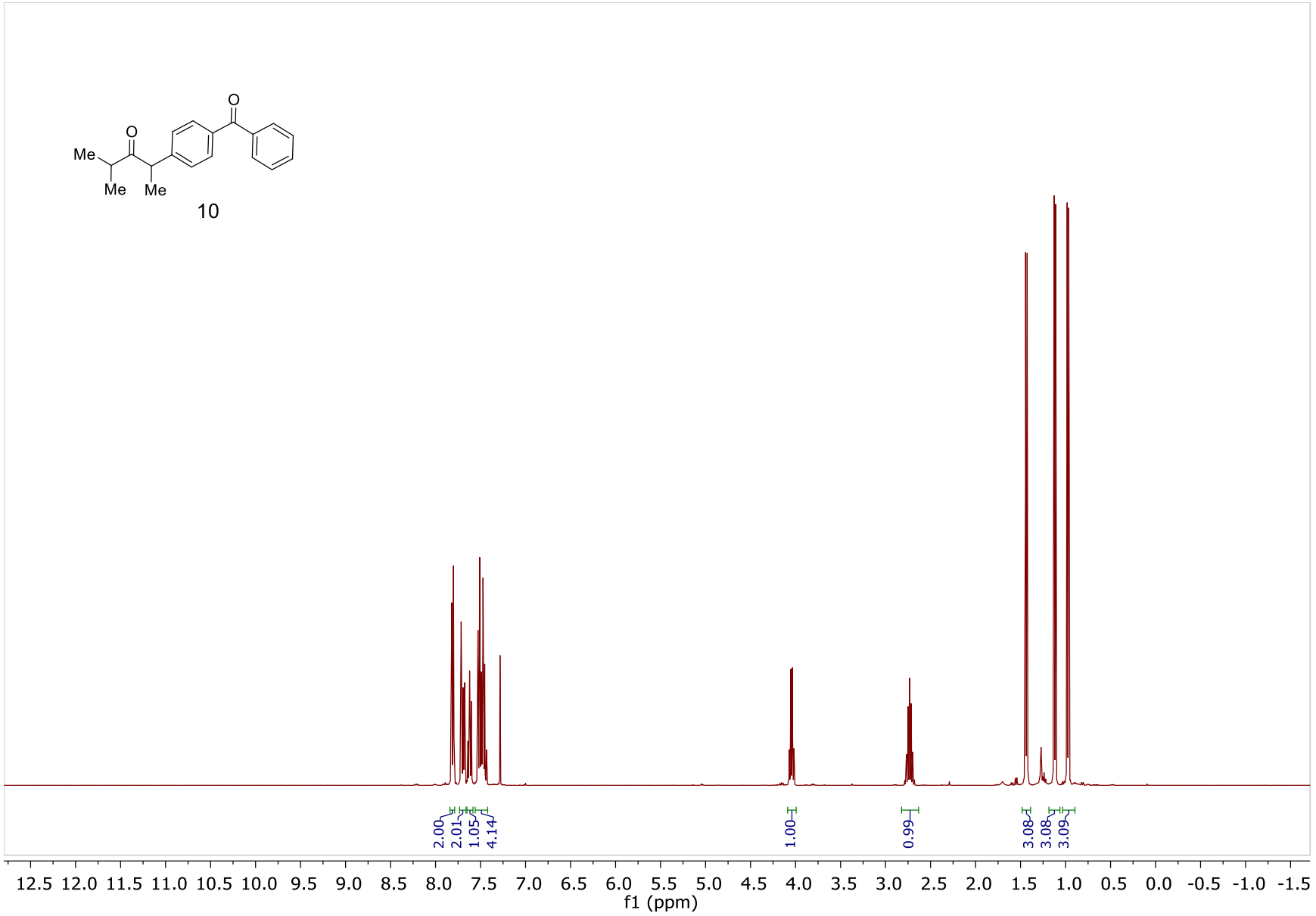

210.54
170.25
141.62
128.47
128.38
125.95
98.70
80.60
77.24 CDCl3
77.03 CDCl3
76.82 CDCl3
67.94
66.19
42.72
42.02
38.15
36.46
35.14
30.10
30.07
28.11
25.26
19.73
Me
Me
O
O
O
Ph
O
O
Me
Me
Me
7
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

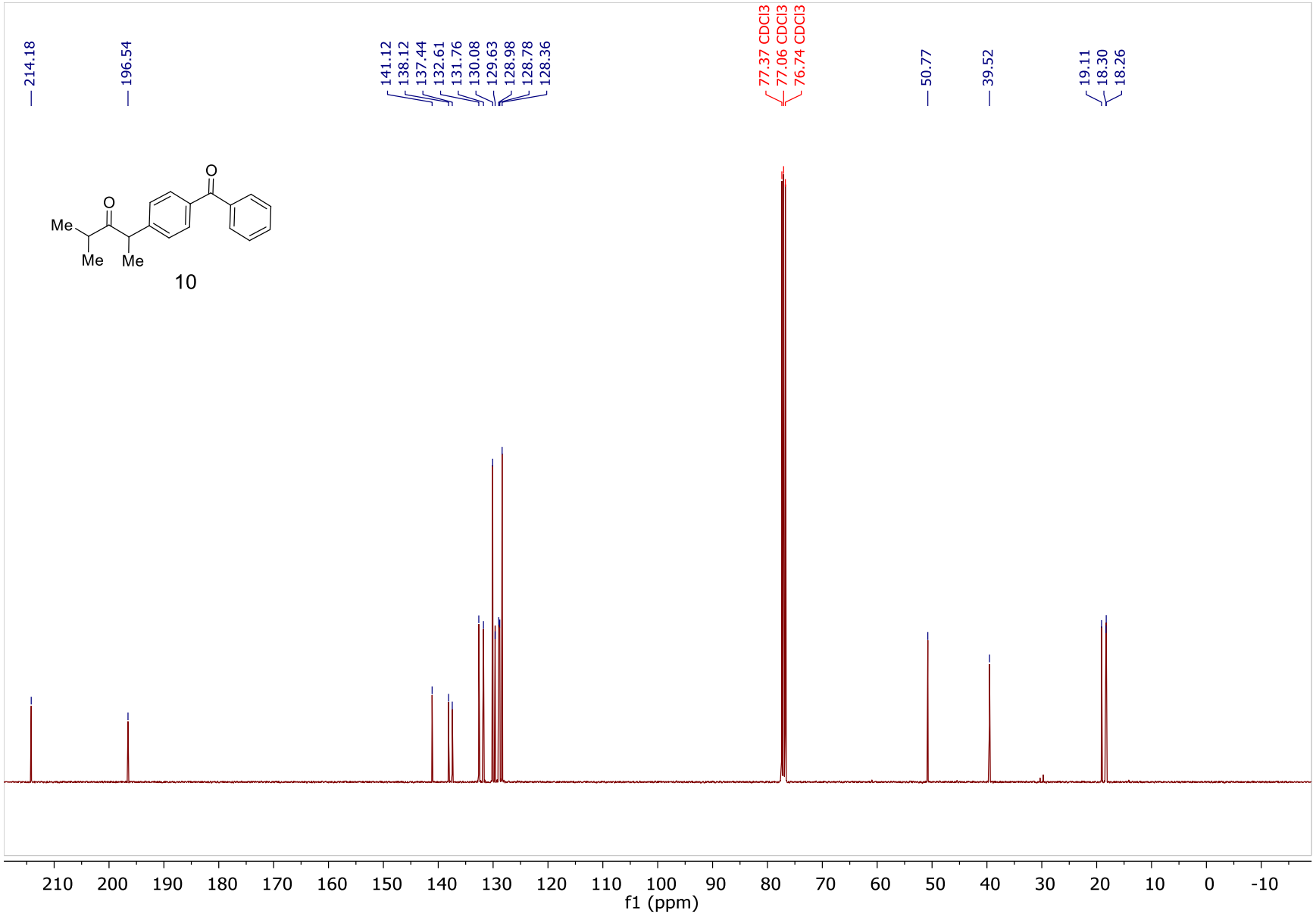

EtO2C
O
8
1.99
1.00
2.08
2.07
4.12
4.15
3.20
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

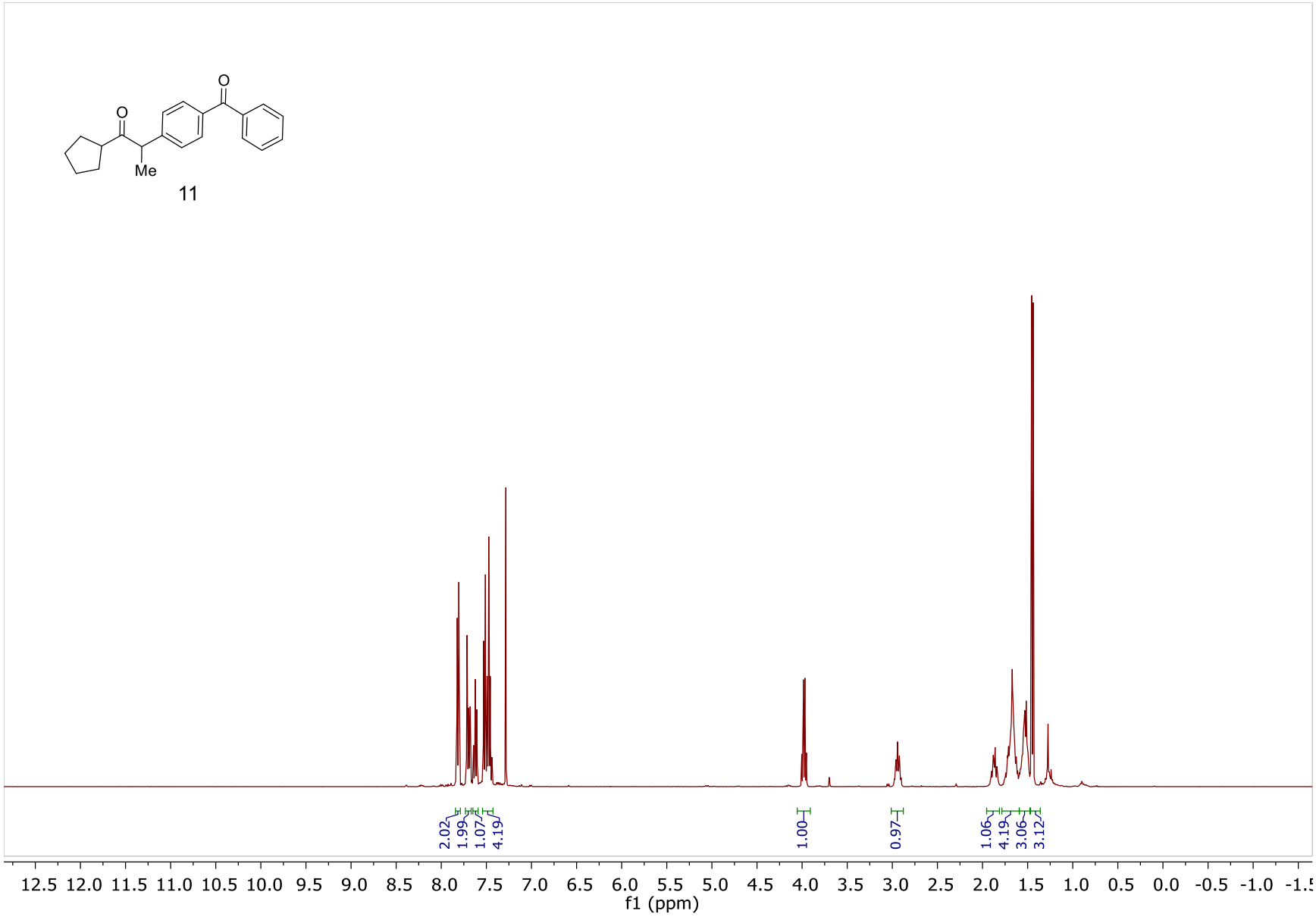

211.21
172.93
77.30 CDCl3
77.04 CDCl3
76.79 CDCl3
60.55
51.28
36.12
28.86
28.05
25.99
14.18
EtO2C
O
8
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

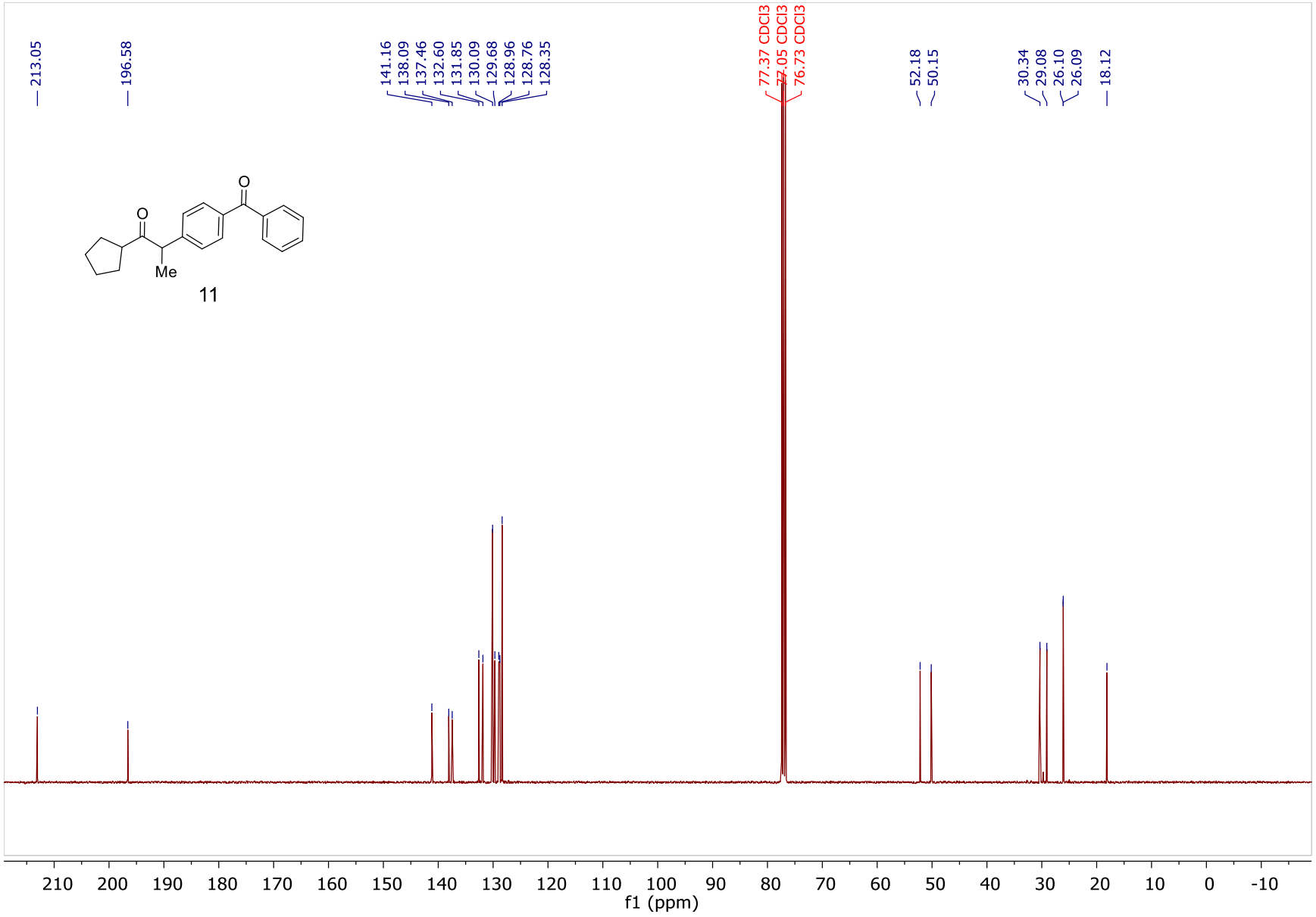

O
CO2Et
BocN
9
3.78
1.66
2.25
2.02
1.02
1.89
2.08
9.00
3.48
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

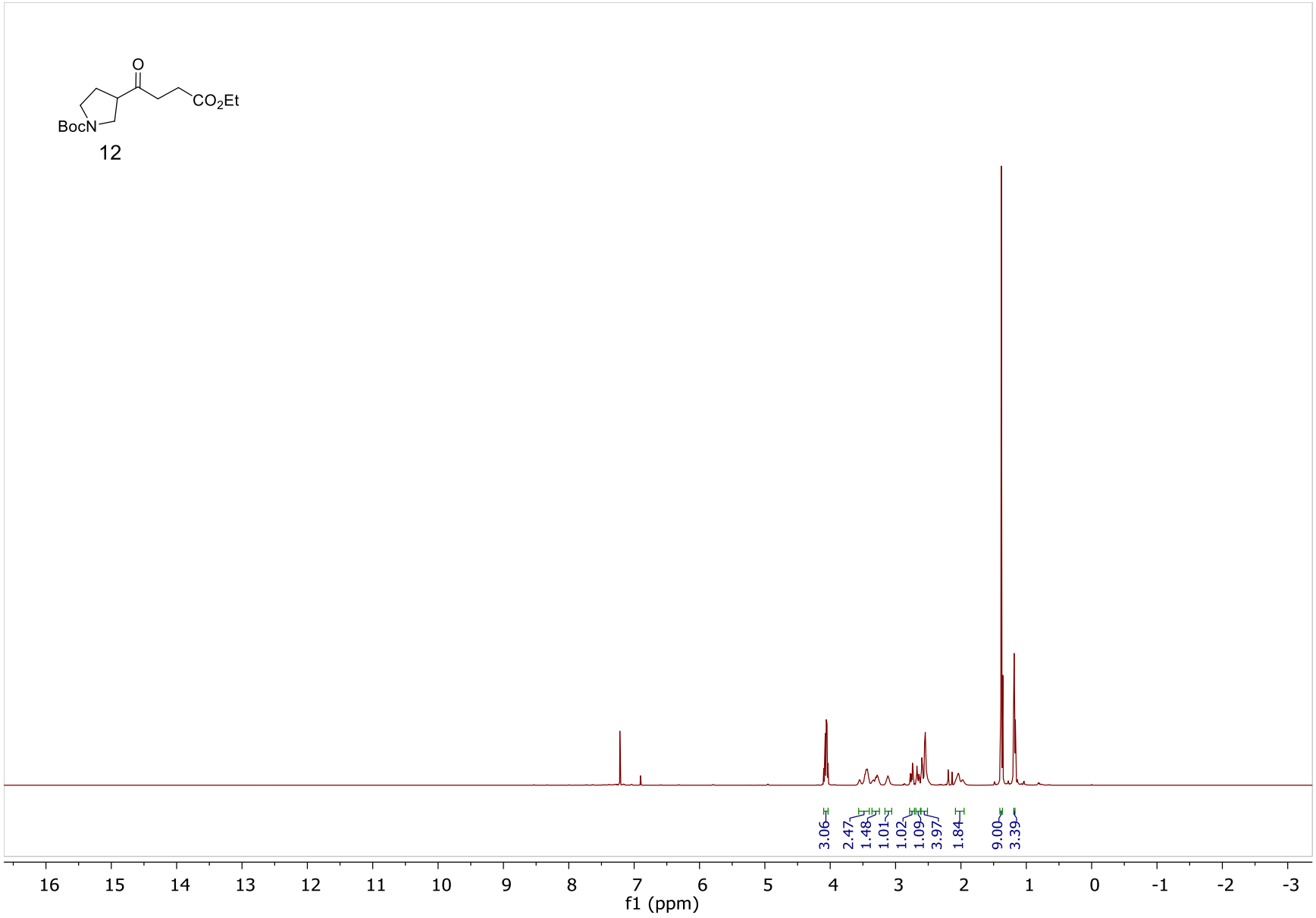

210.26
172.74
154.66
79.60
77.24 CDCl3
77.03 CDCl3
76.82 CDCl3
60.66
48.56
43.18
35.00
28.43
27.92
27.48
14.18
O
CO2Et
BocN
9
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

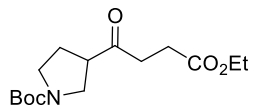

O
O
Me
Me
Me
10
2.00
2.01
1.05
4.14
1.00
0.99
3.08
3.08
3.09
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)


214.18
196.54
141.12
138.12
137.44
132.61
131.76
130.08
129.63
128.98
128.78
128.36
77.37 CDCl3
77.06 CDCl3
76.74 CDCl3
50.77
39.52
19.11
18.30
18.26
O
O
Me
Me
Me
10
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)


O
O
Me
11
2.02
1.99
1.07
4.19
1.00
0.97
1.06
4.19
3.06
3.12
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

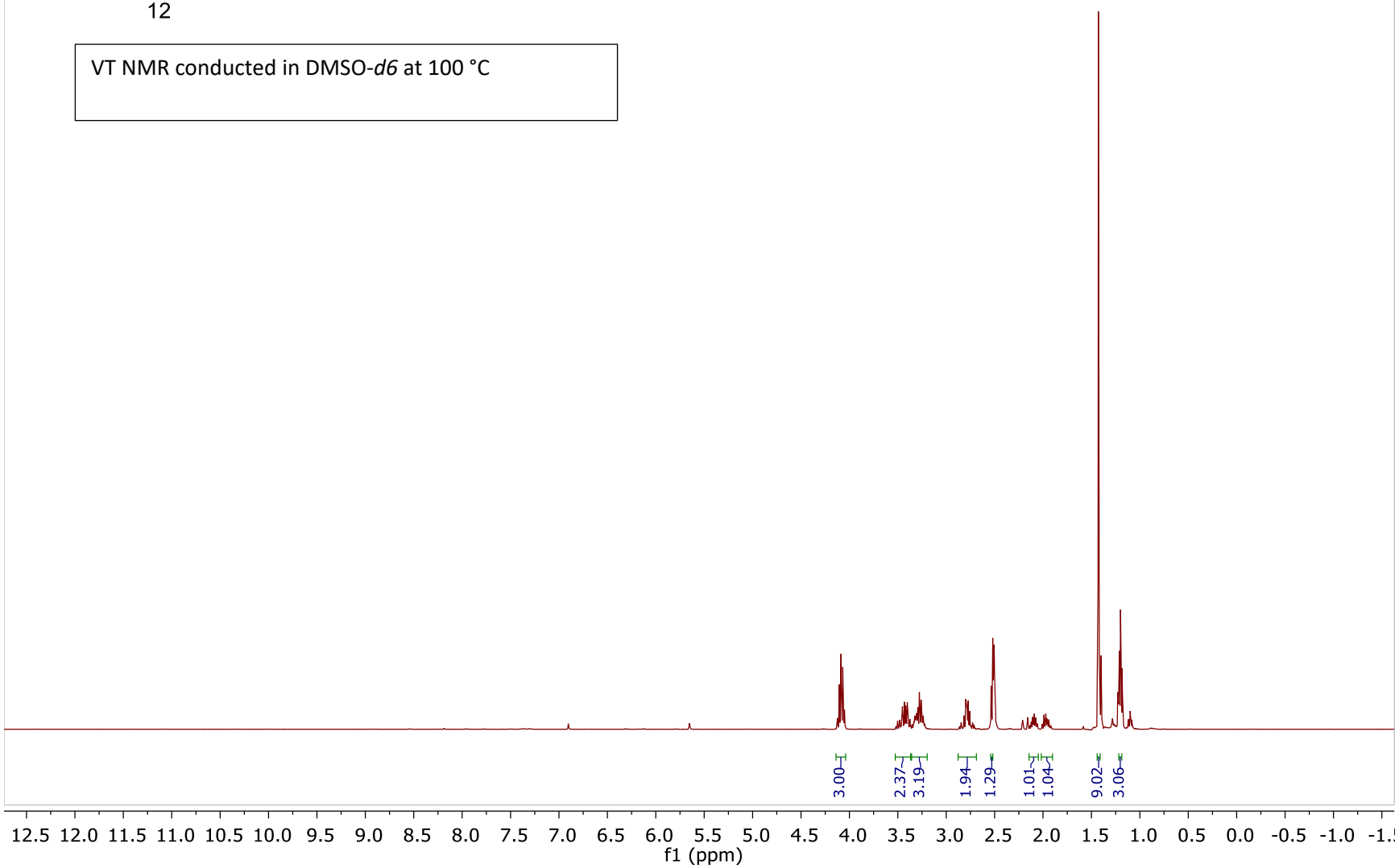

213.05
196.58
141.16
138.09
137.46
132.60
131.85
130.09
129.68
128.96
128.76
128.35
77.37 CDCl3
77.05 CDCl3
76.73 CDCl3
52.18
50.15
30.34
29.08
26.10
26.09
18.12
O
O
Me
11
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

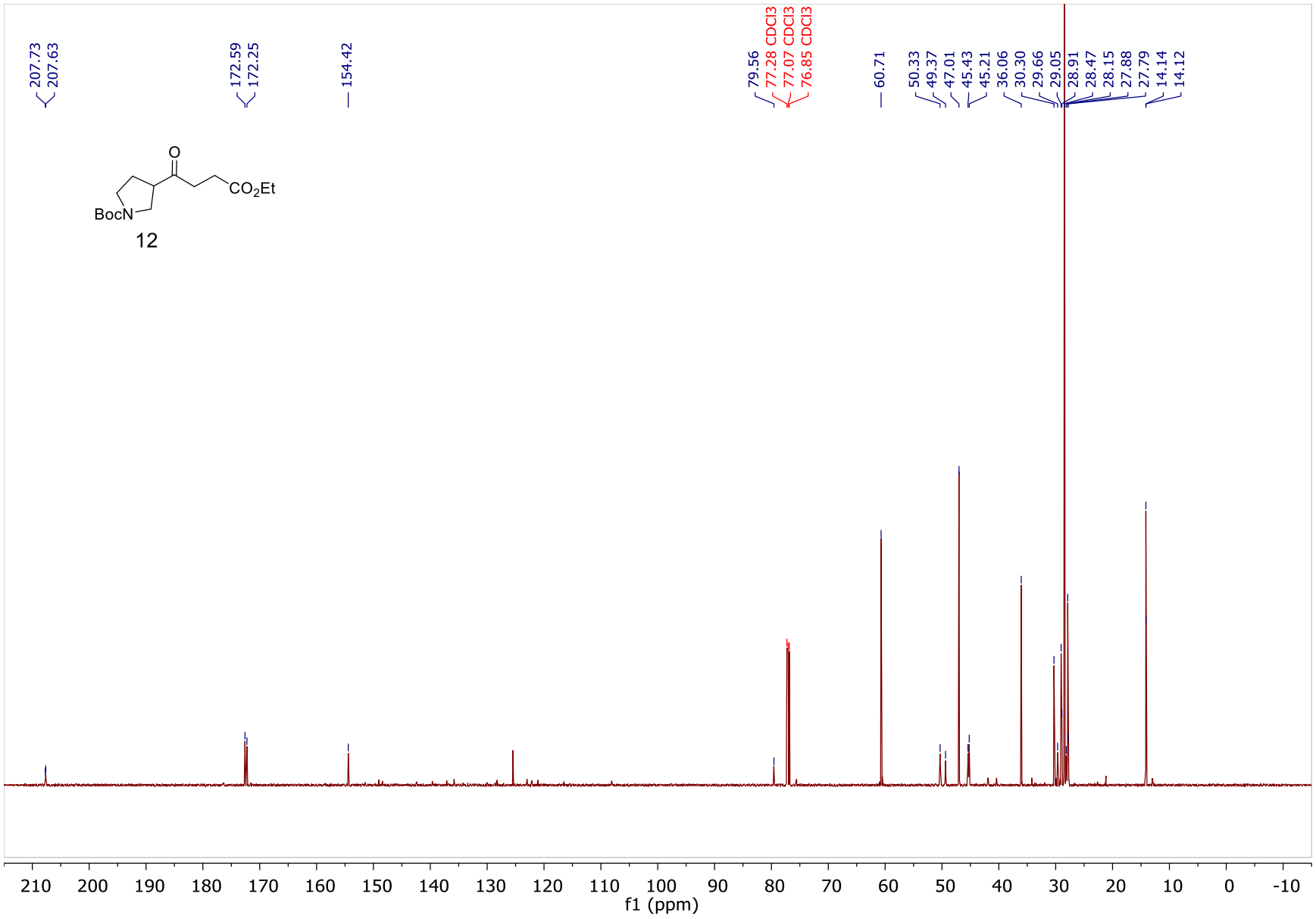

O
CO2Et
BocN
12
3.06
2.47
1.48
1.01
1.02
1.09
3.97
1.84
9.00
3.39
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

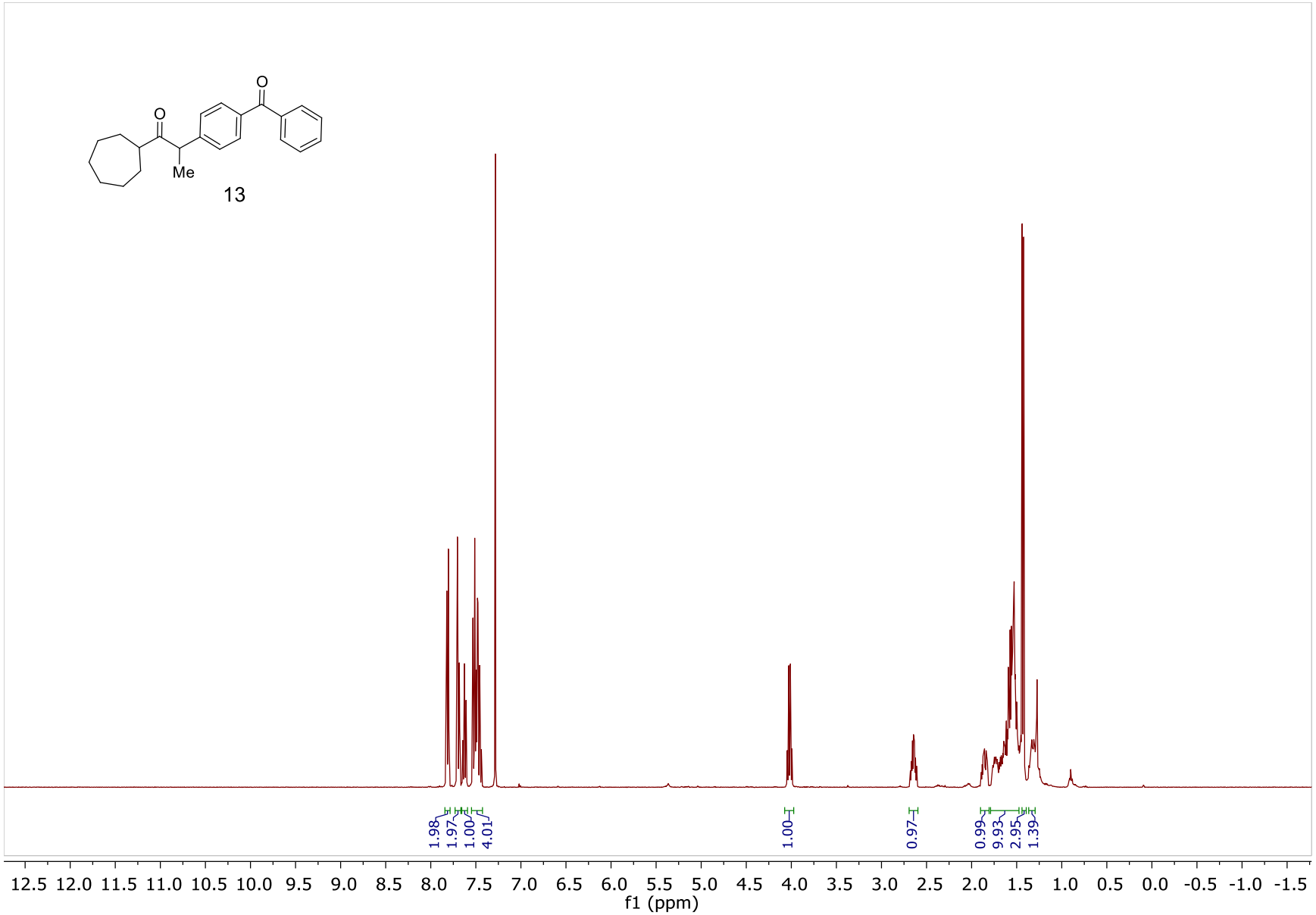


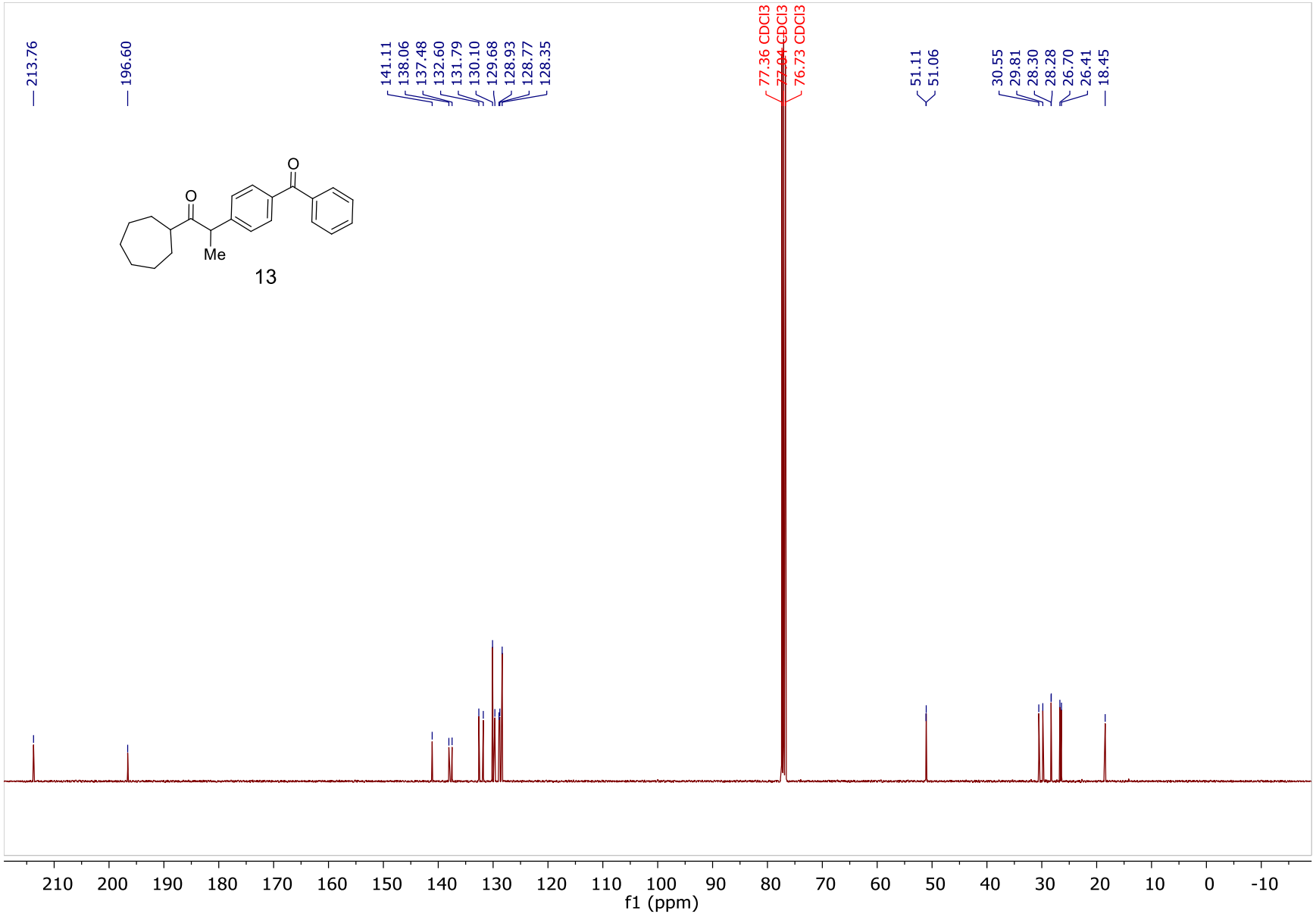
12

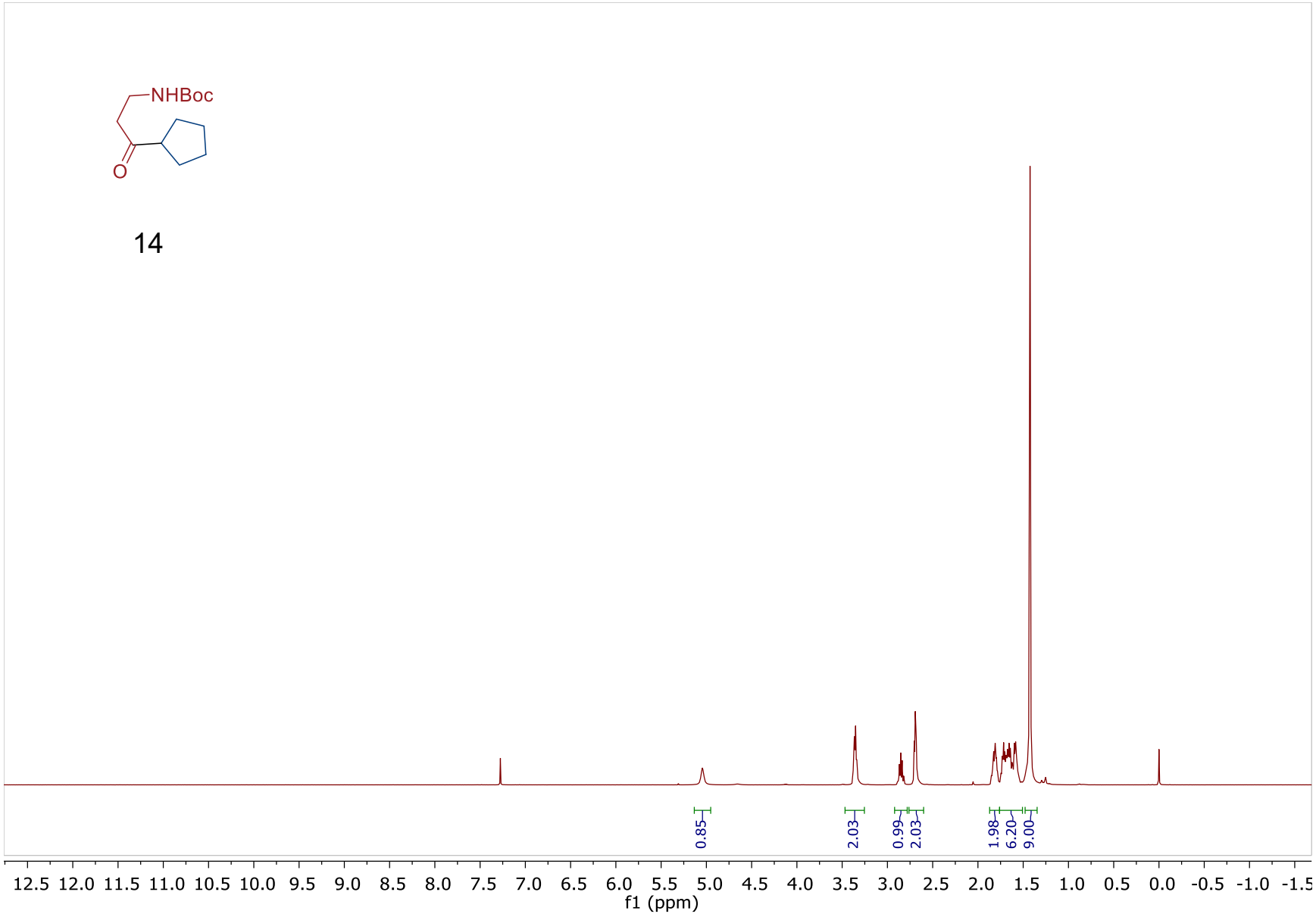
VT NMR conducted in DMSO-*d6* at 100 °C

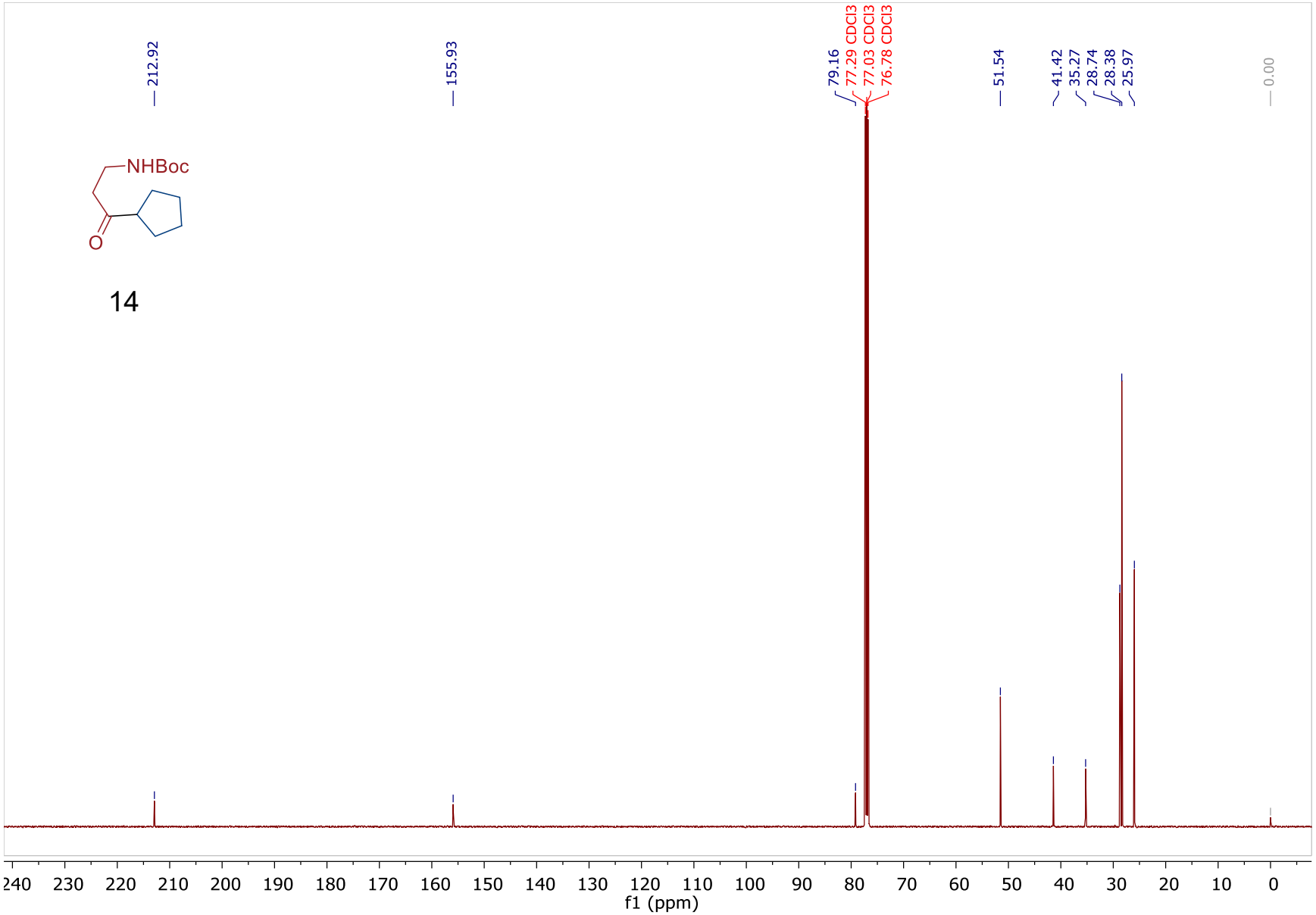


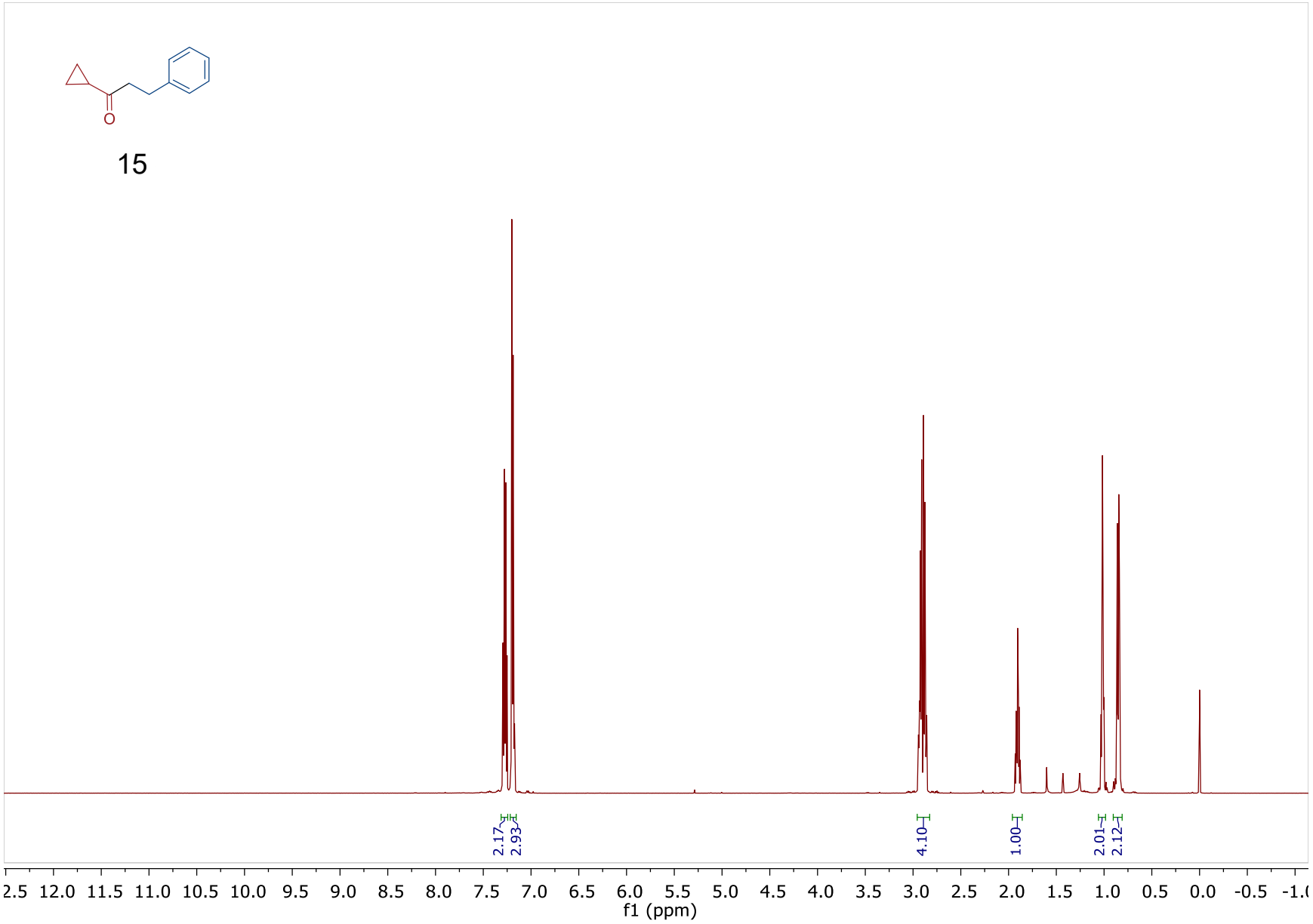

207.73
207.63
172.59
172.25
154.42
79.56
77.28 CDCl3
77.07 CDCl3
76.85 CDCl3
60.71
50.33
49.37
47.01
45.43
45.21
36.06
30.30
29.66
29.05
28.91
28.47
28.15
27.88
27.79
14.14
14.12
O
CO2Et
BocN
12
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

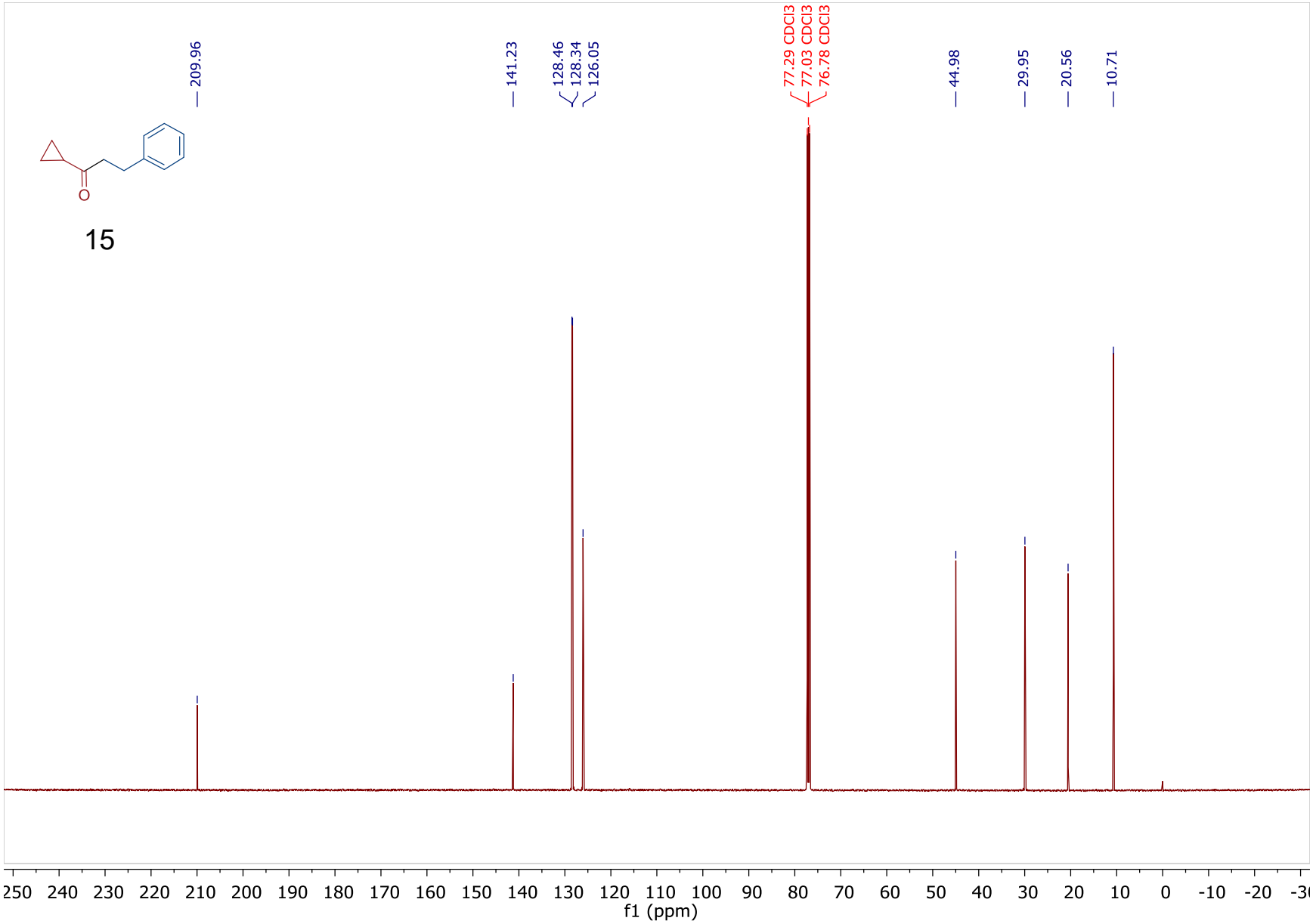

O
O
Me
13
1.98
1.97
1.00
4.01
1.00
0.97
0.99
9.93
2.95
1.39
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

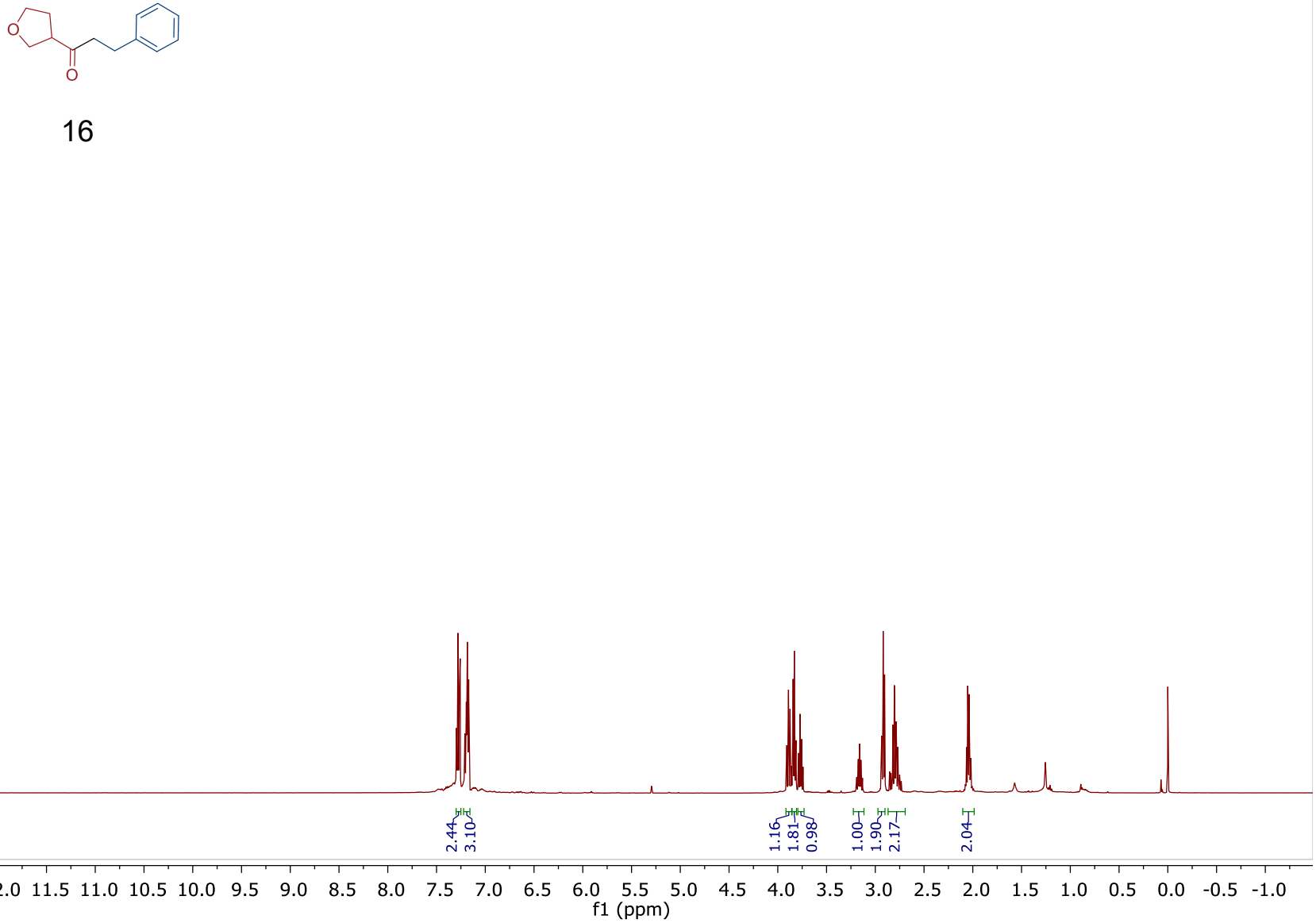

213.76
196.60
141.11
138.06
137.48
132.60
131.79
130.10
129.68
128.93
128.77
128.35
77.36 CDCl3
77.04 CDCl3
76.73 CDCl3
51.11
51.06
30.55
29.81
28.30
28.28
26.70
26.41
18.45
O
O
Me
13
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

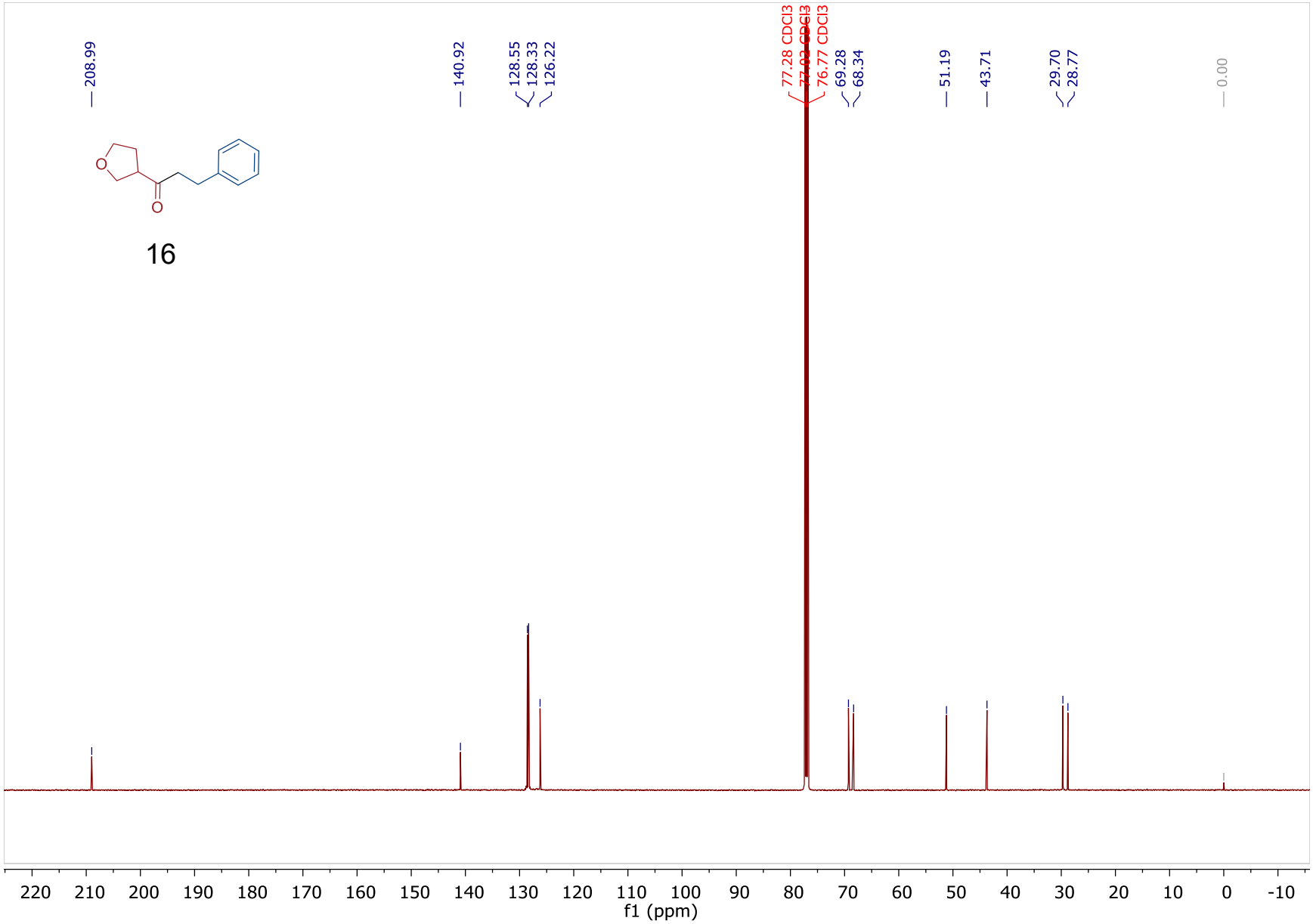

NHBoc
O
14
0.85
2.03
0.99
2.03
1.98
6.20
9.00
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

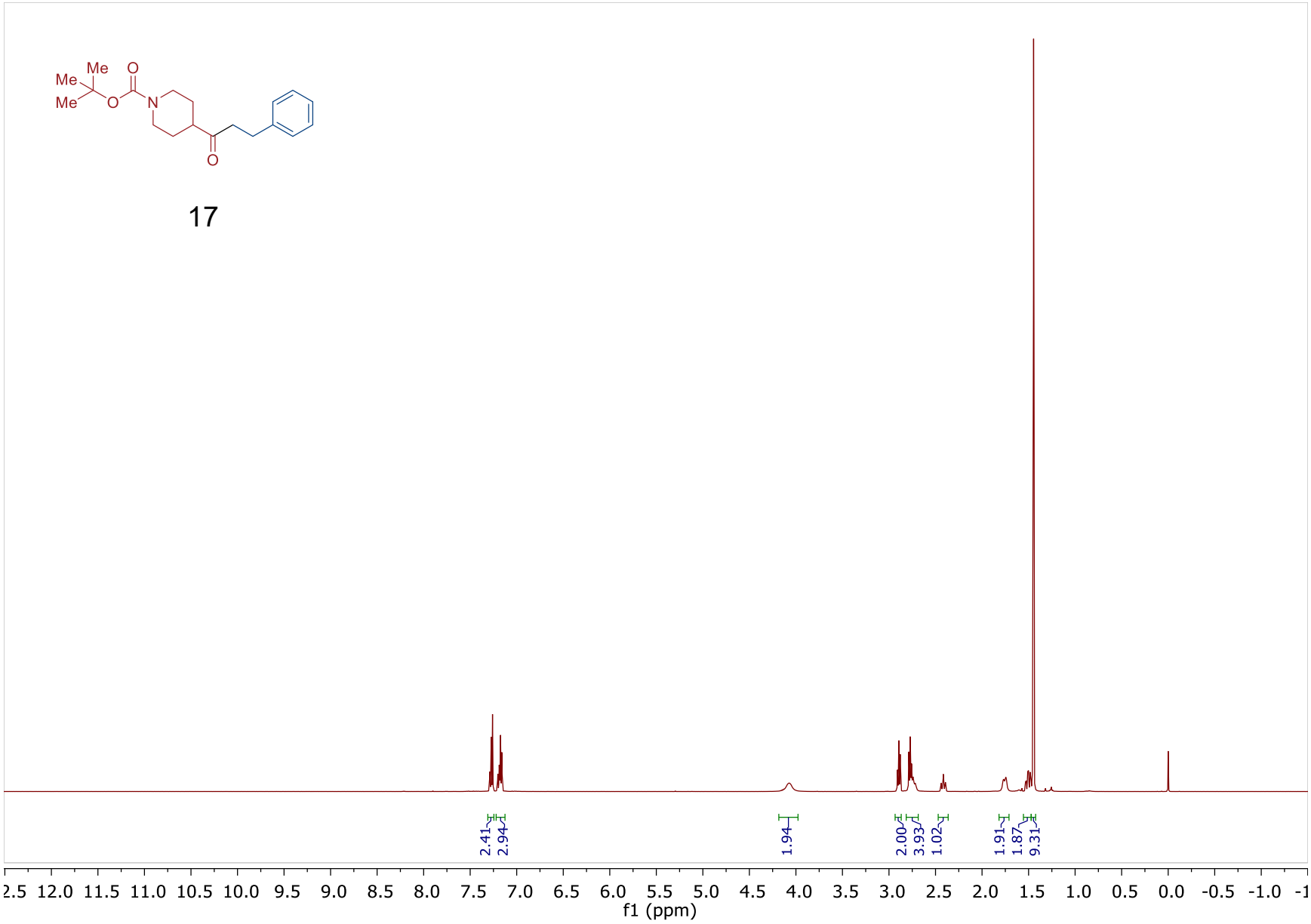

212.92
155.93
79.16
77.29 CDCl3
77.03 CDCl3
76.78 CDCl3
51.54
41.42
35.27
28.74
28.38
25.97
0.00
NHBoc
O
14
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

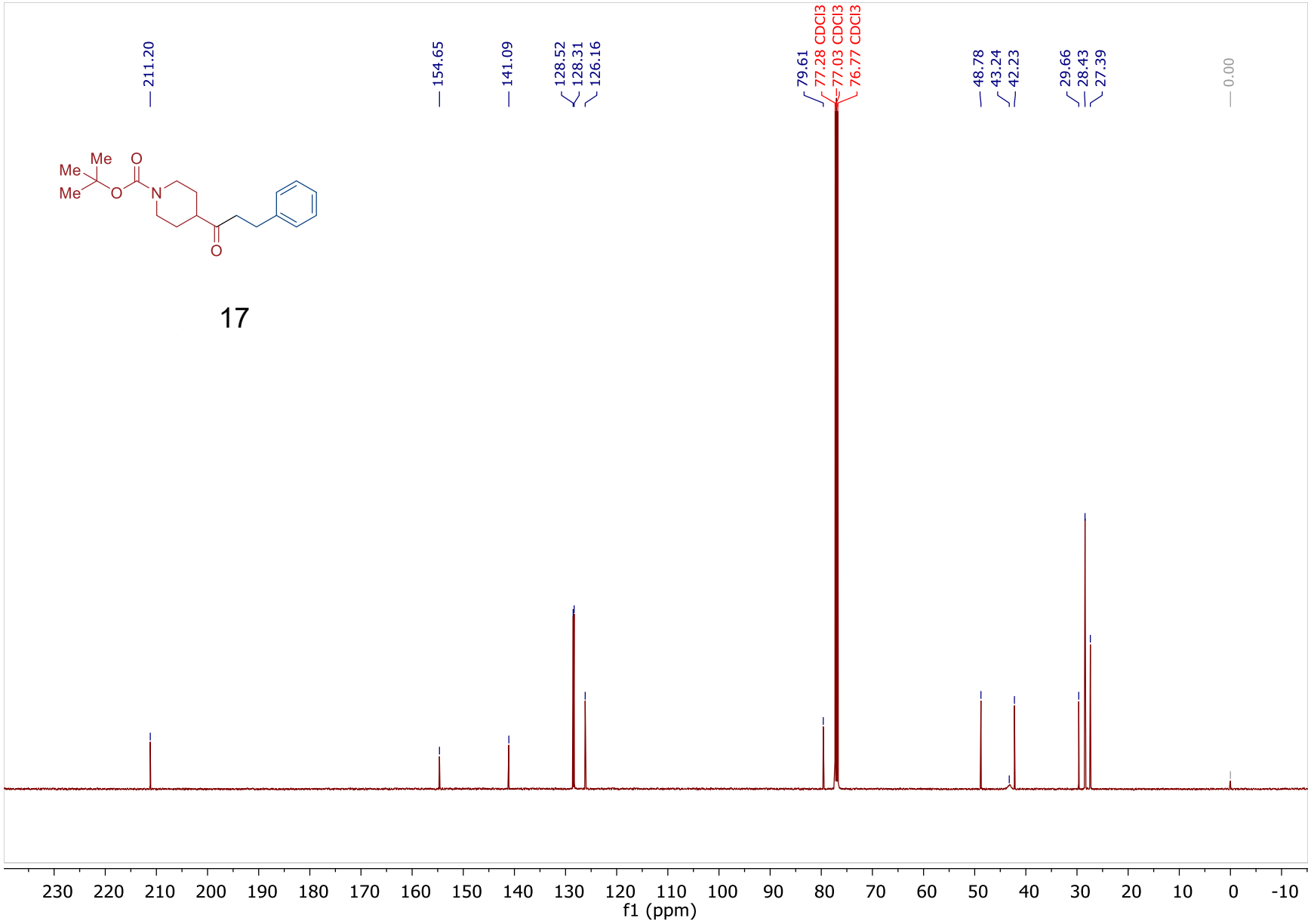

O
15
2.17
2.93
4.10
1.00
2.01
2.12
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.(
f1 (ppm)

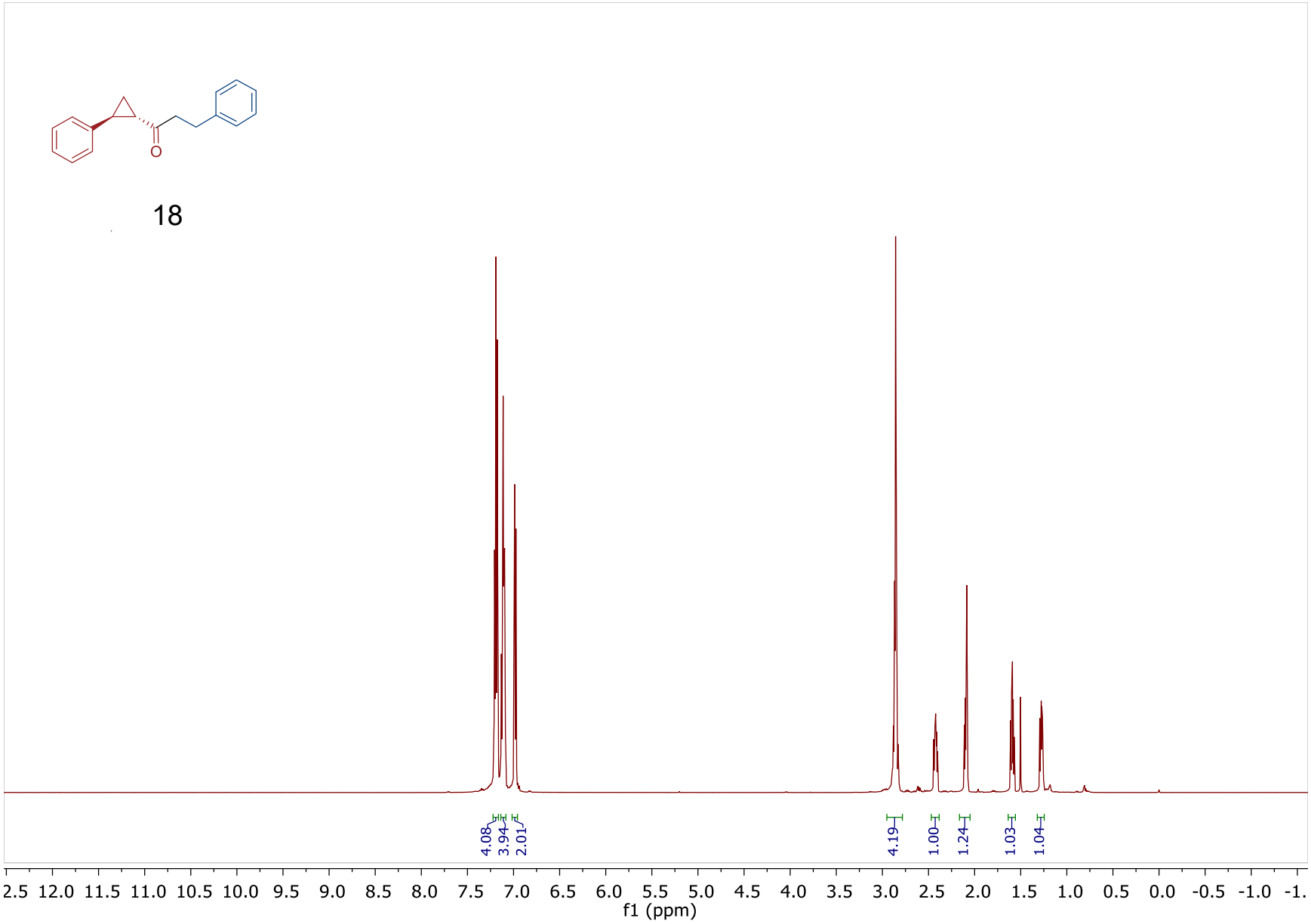

209.96
141.23
128.46
128.34
126.05
77.29 CDCl3
77.03 CDCl3
76.78 CDCl3
44.98
29.95
20.56
10.71
O
15
250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -3
f1 (ppm)

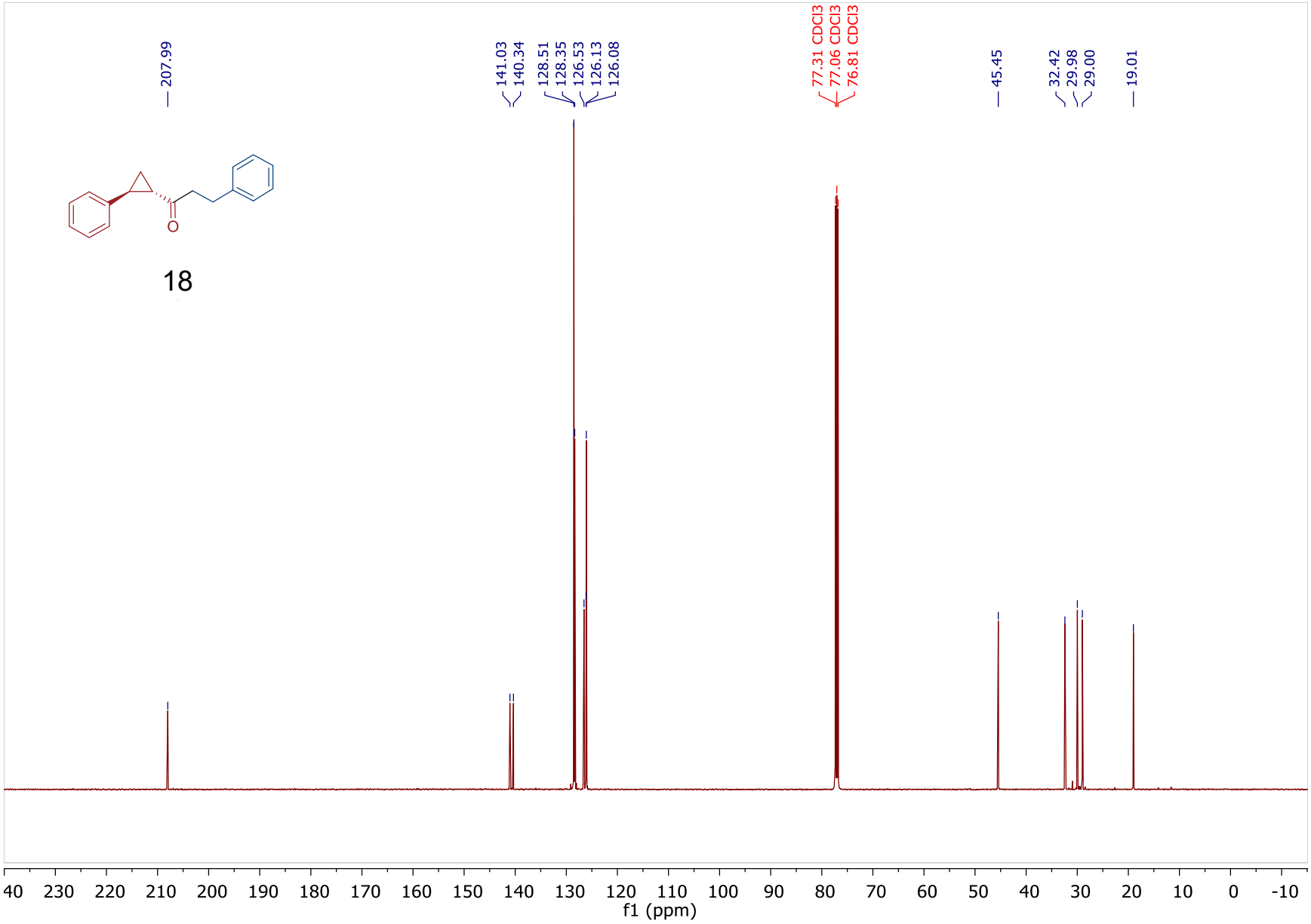

16
2.44
3.10
1.16
1.81
0.98
1.00
1.90
2.17
2.04
f1 (ppm)

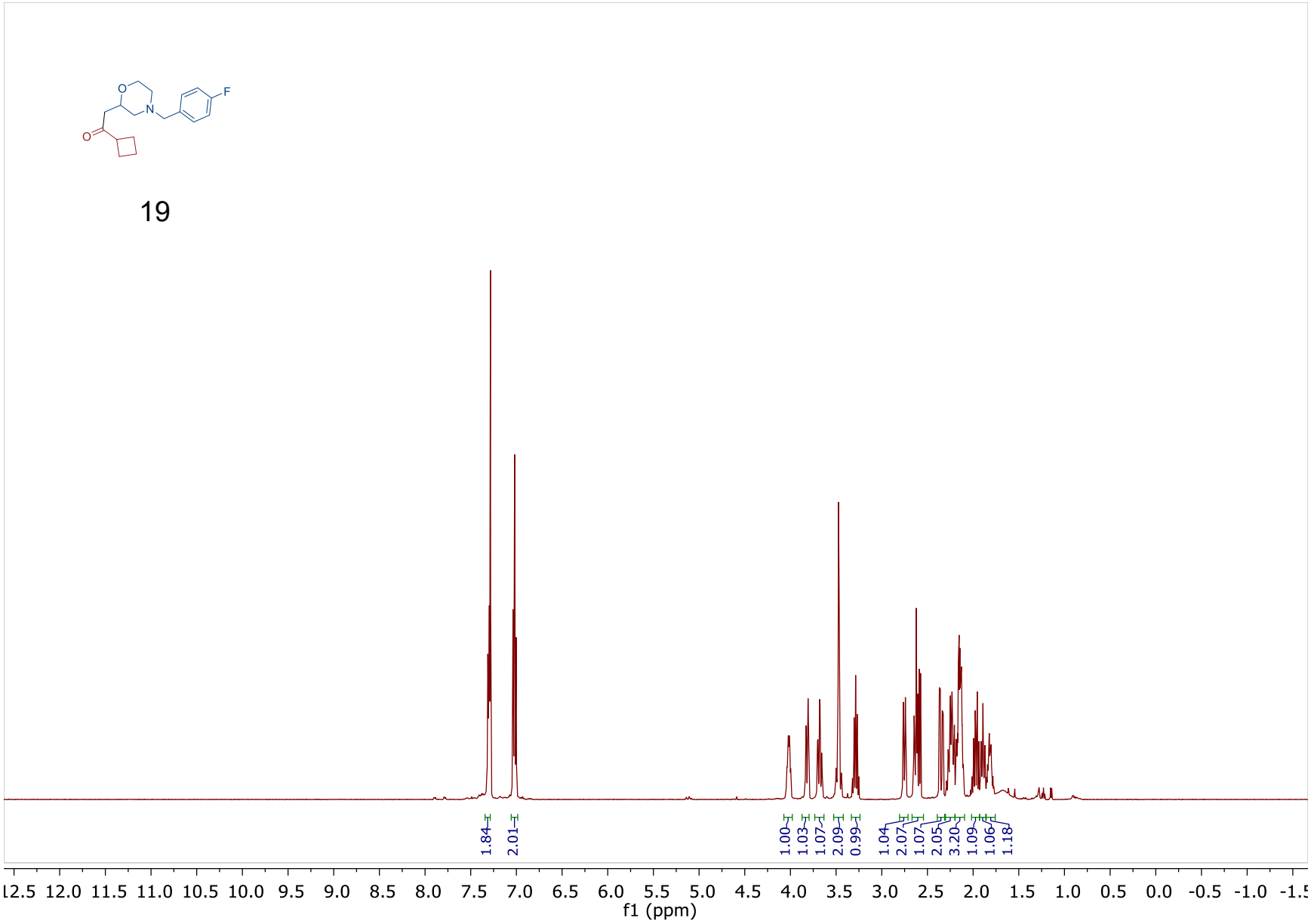

208.99
140.92
128.55
128.33
126.22
77.28 CDCl3
77.03 CDCl3
76.77 CDCl3
69.28
68.34
51.19
43.71
29.70
28.77
0.00
16
f1 (ppm)

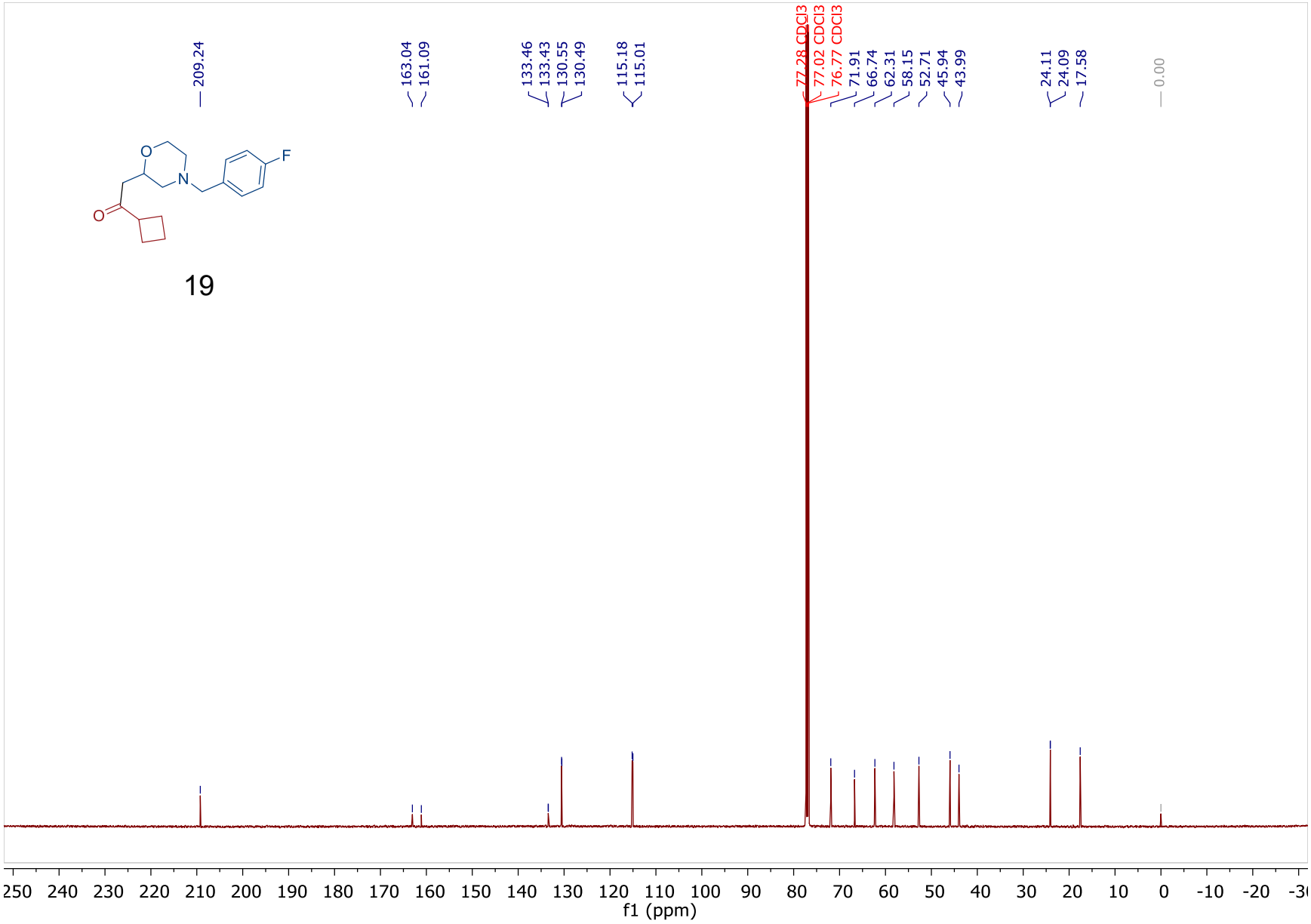

Me
Me
Me
O
O
N
O
17
2.41
2.94
1.94
2.00
3.93
1.02
1.91
1.87
9.31
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1
f1 (ppm)

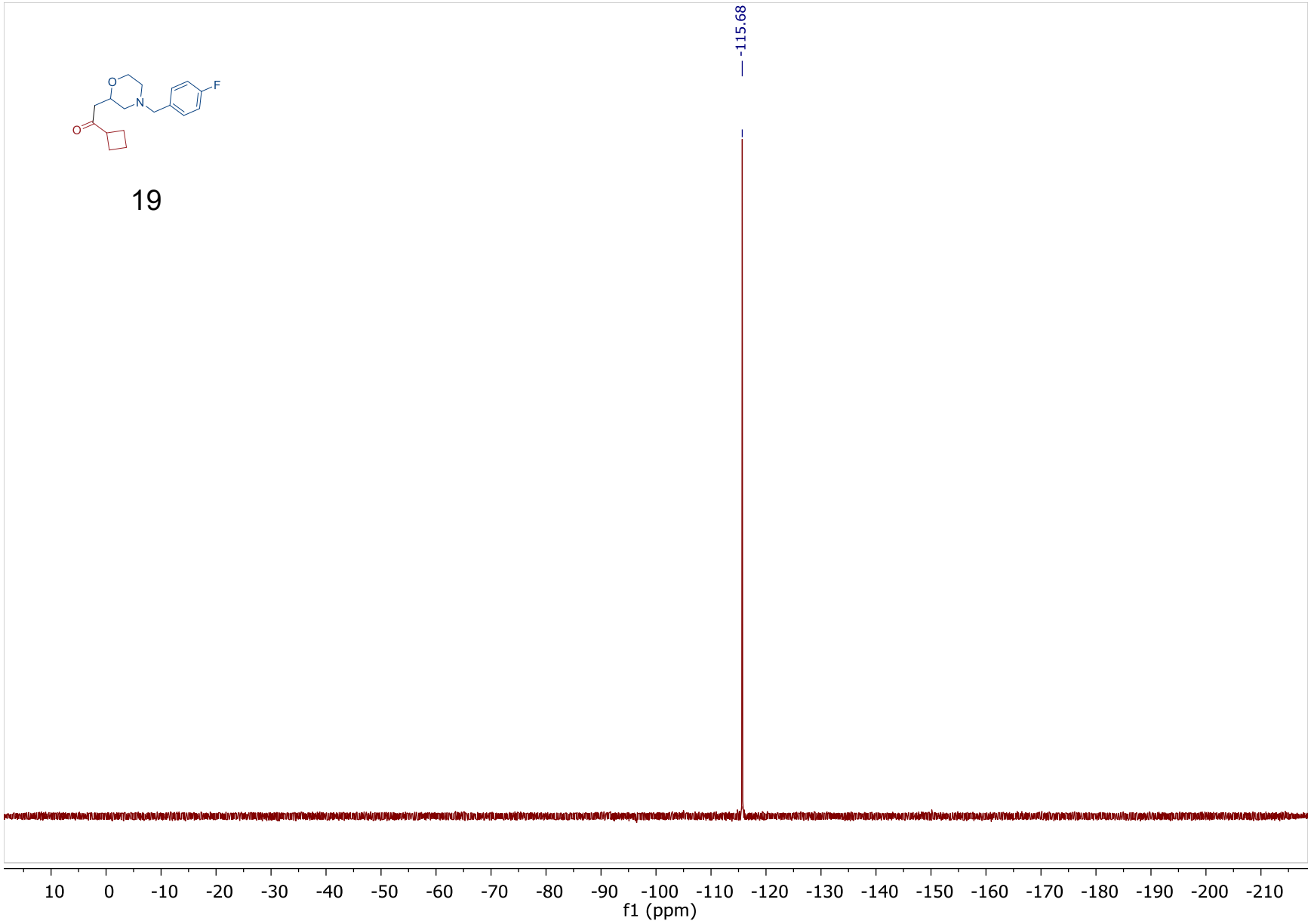

211.20
154.65
141.09
128.52
128.31
126.16
79.61
77.28 CDCl3
77.03 CDCl3
76.77 CDCl3
48.78
43.24
42.23
29.66
28.43
27.39
0.00
Me
Me
Me
O
O
N
O
17
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

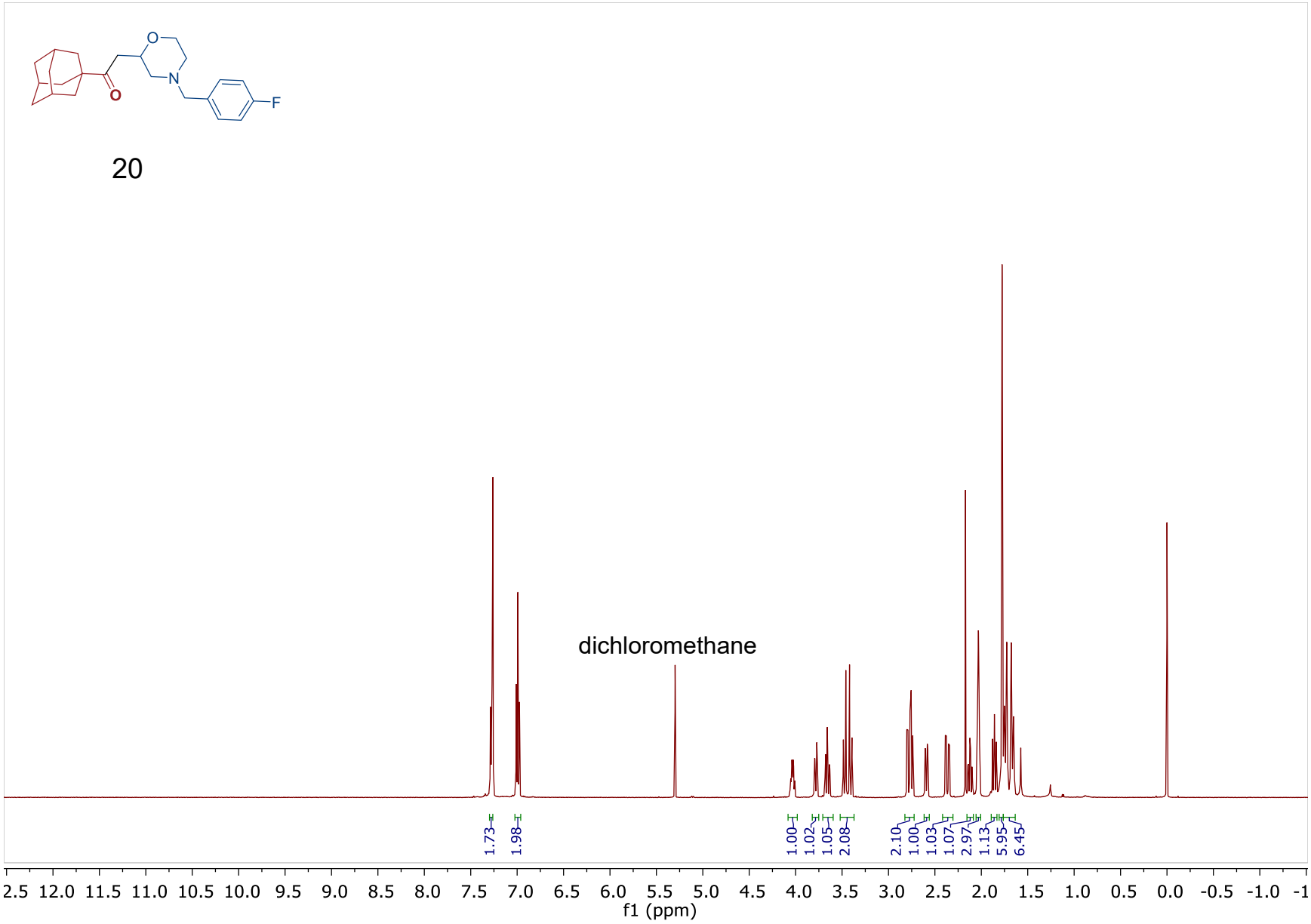

18
4.08
3.94
2.01
4.19
1.00
1.24
1.03
1.04
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.
f1 (ppm)

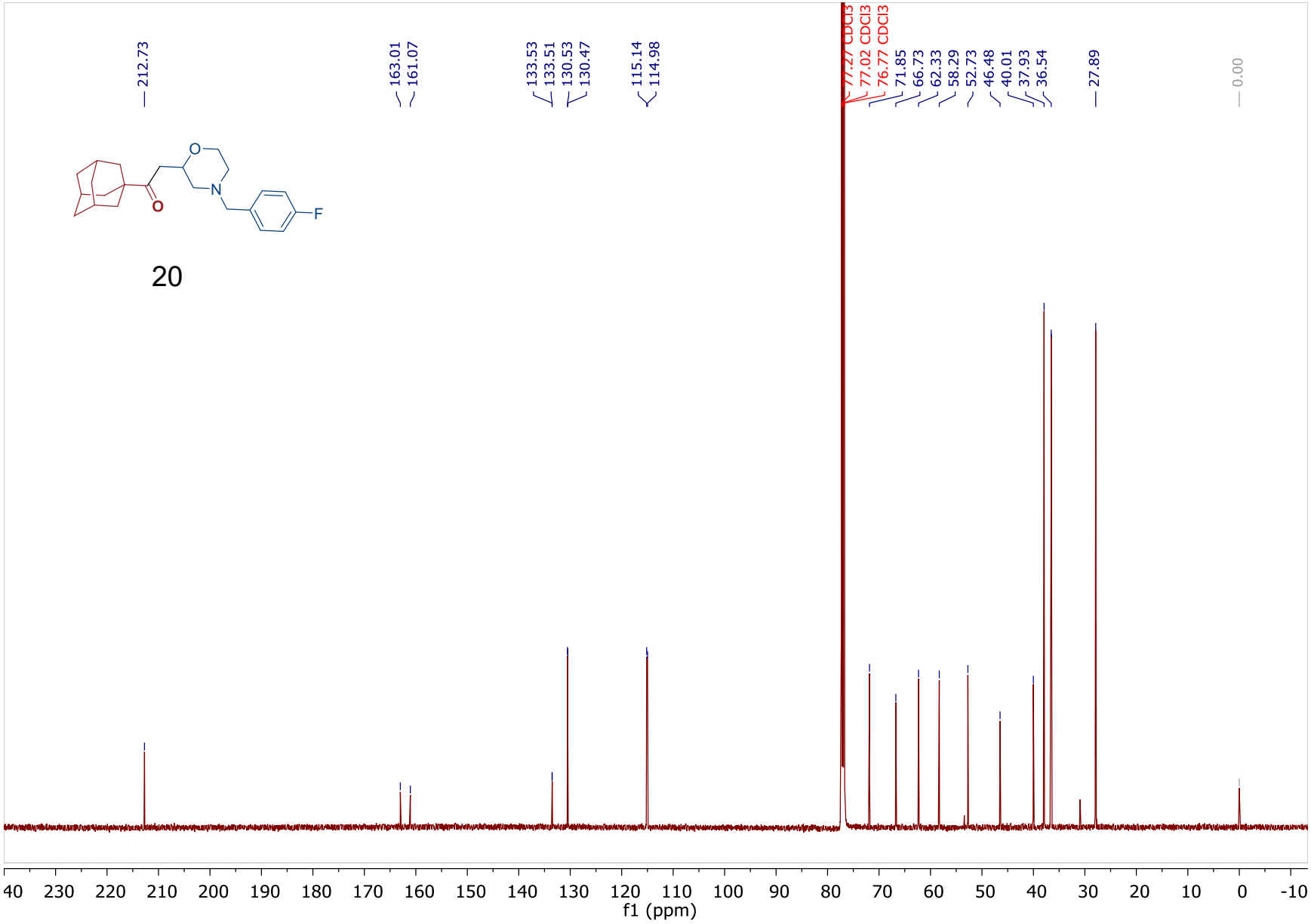

207.99
141.03
140.34
128.51
128.35
126.53
126.13
126.08
77.31 CDCl3
77.06 CDCl3
76.81 CDCl3
45.45
32.42
29.98
29.00
19.01
O
18
40 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

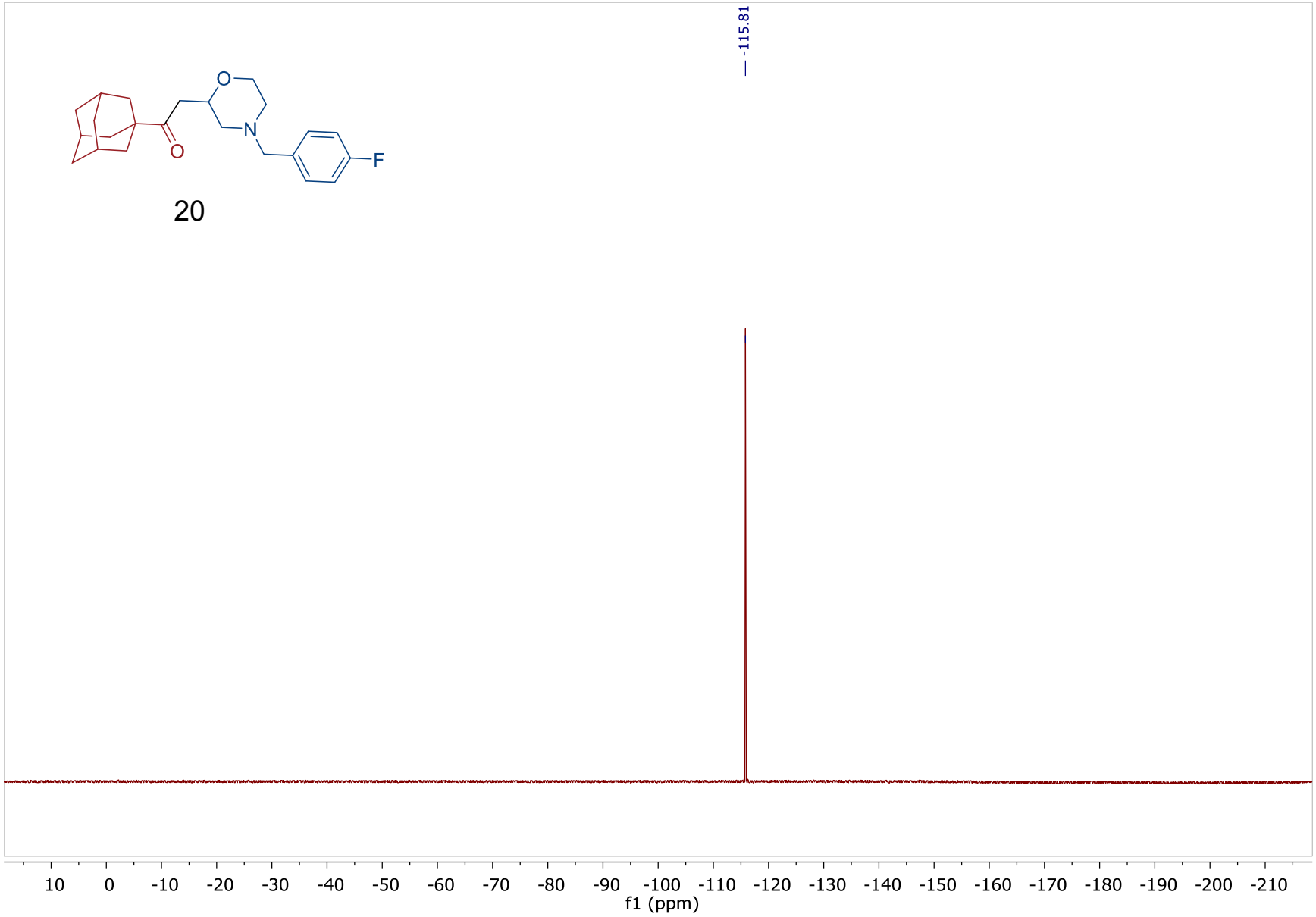

O
N
F
O
19
1.84
2.01
1.00
1.03
1.07
2.09
0.99
1.04
2.07
1.07
2.05
3.20
1.09
1.06
1.18
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)

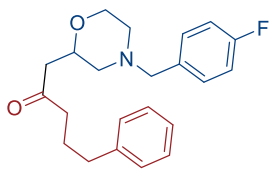

209.24
163.04
161.09
133.46
133.43
130.55
130.49
115.18
115.01
77.28 CDCl3
77.02 CDCl3
76.77 CDCl3
71.91
66.74
62.31
58.15
52.71
45.94
43.99
24.11
24.09
17.58
0.00
O
N
F
O
19
f1 (ppm)


-115.68
O
N
F
O
19
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)


O
N
O
F
20
dichloromethane
1.73
1.98
1.00
1.02
1.05
2.08
2.10
1.00
1.03
1.07
2.97
1.13
5.95
6.45
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1
f1 (ppm)

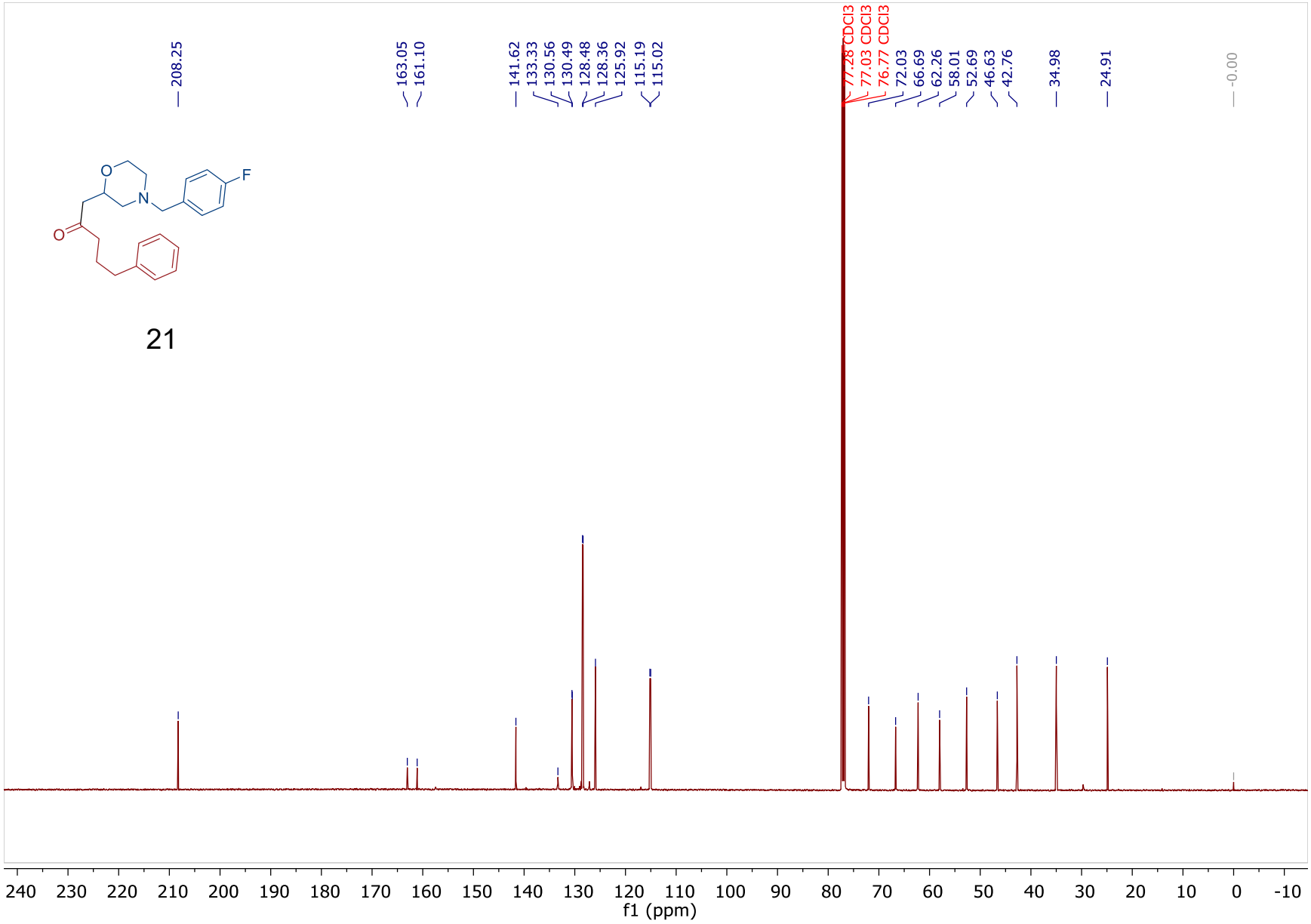

212.73
163.01
161.07
133.53
133.51
130.53
130.47
115.14
114.98
77.27 CDCl3
77.02 CDCl3
76.77 CDCl3
71.85
66.73
62.33
58.29
52.73
46.48
40.01
37.93
36.54
27.89
0.00
O
O
N
F
20
40 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

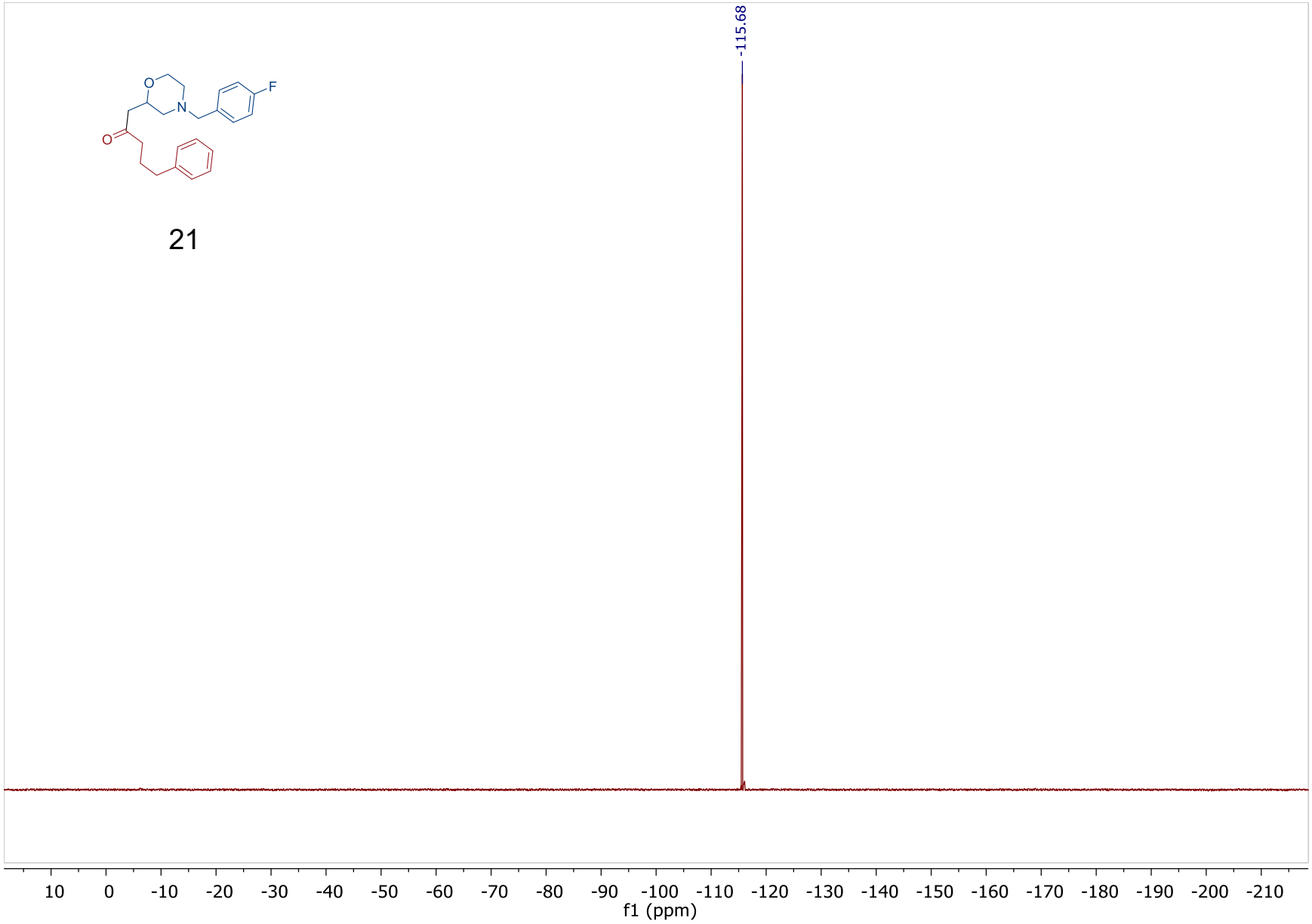

-115.81
O
N
O
F
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

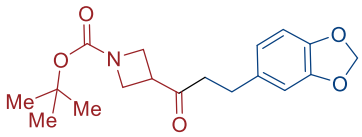

O
N
F
O

21

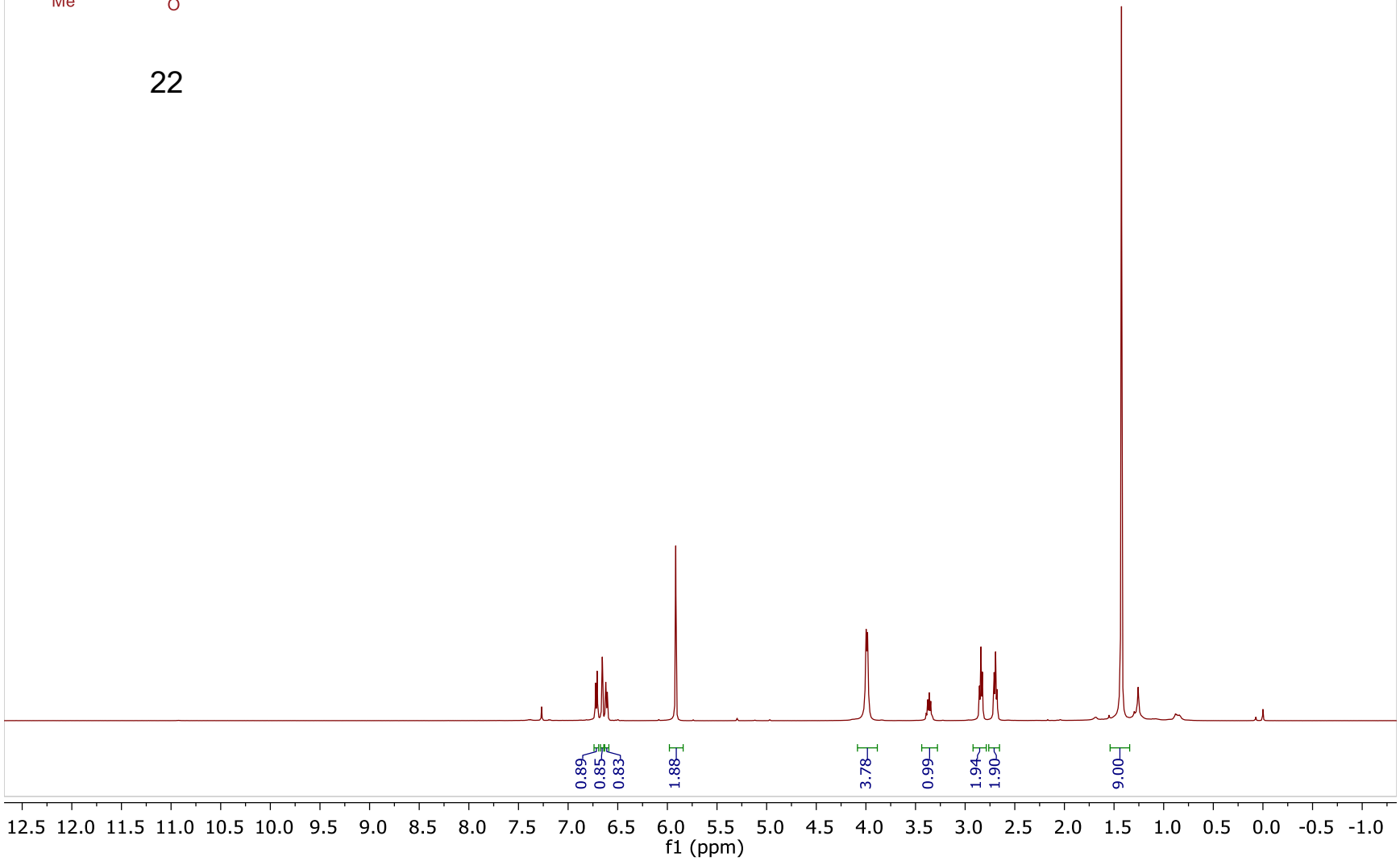
4.26
2.98
2.03
1.00
1.01
1.06
1.95
1.11
3.98
2.02
1.05
1.05
2.95
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

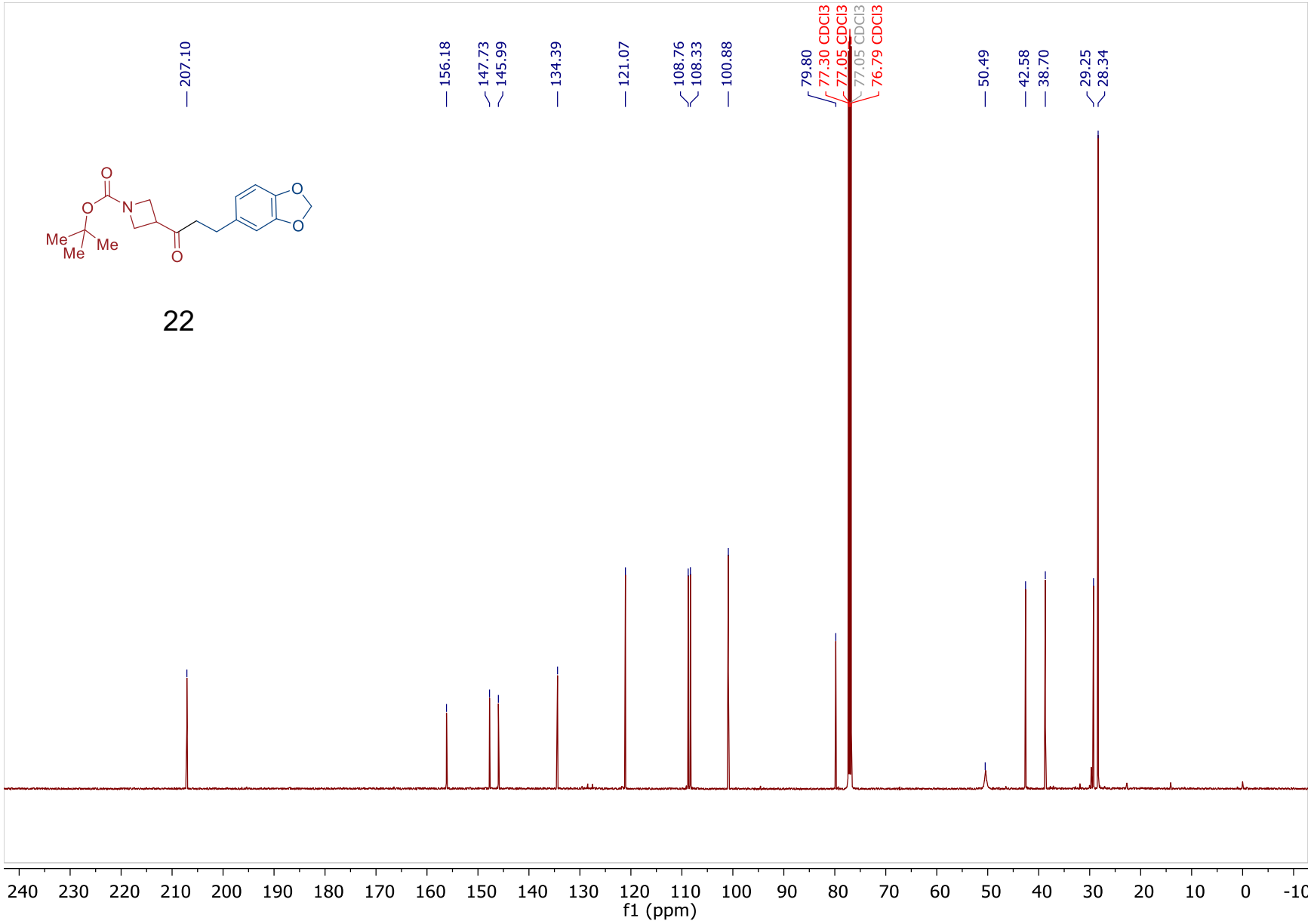

208.25
163.05
161.10
141.62
133.33
130.56
130.49
128.48
128.36
125.92
115.19
115.02
77.28 CDCl3
77.03 CDCl3
76.77 CDCl3
72.03
66.69
62.26
58.01
52.69
46.63
42.76
34.98
24.91
-0.00
O
N
F
O
21
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

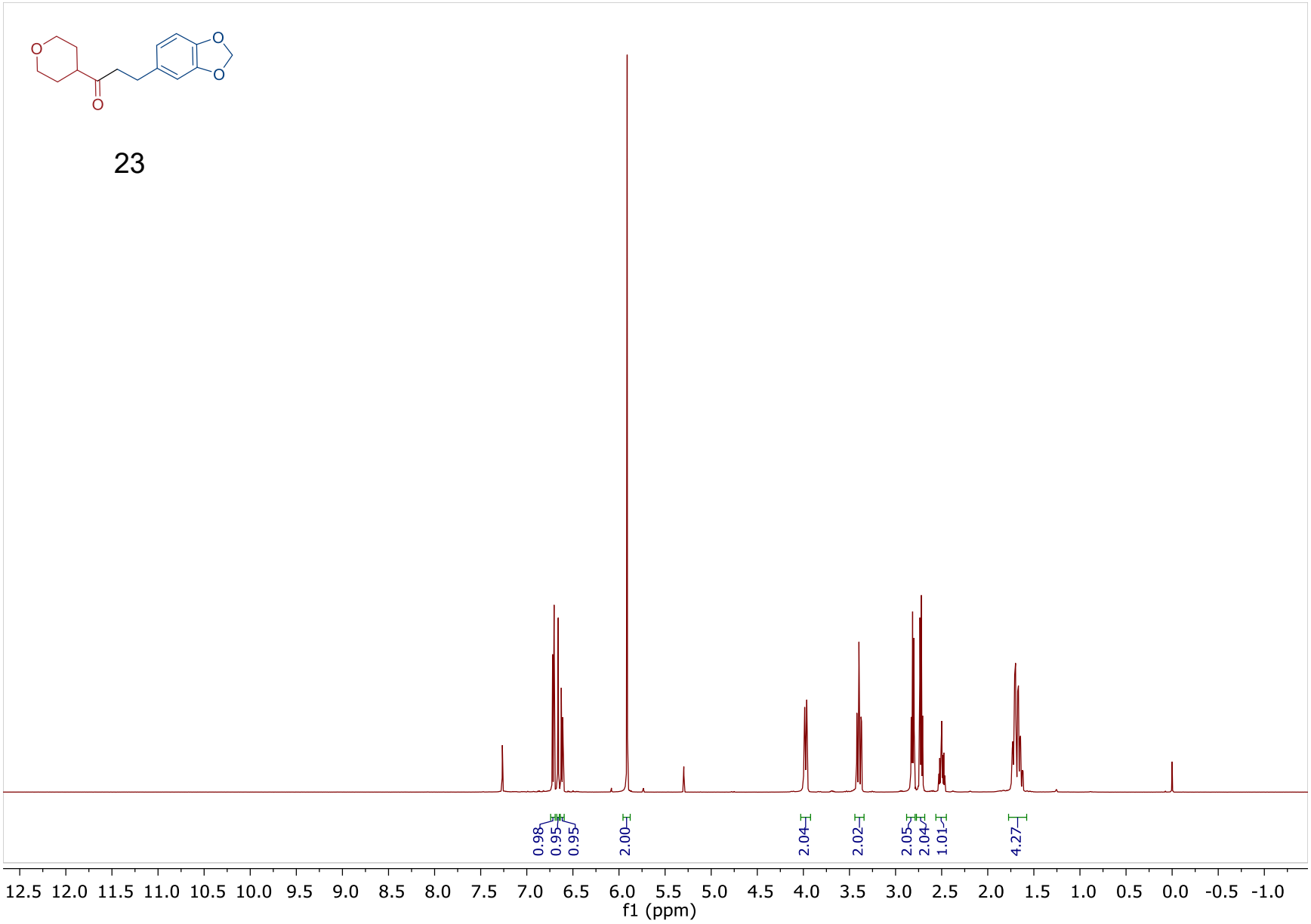

-115.68
O
N
F
O
21
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

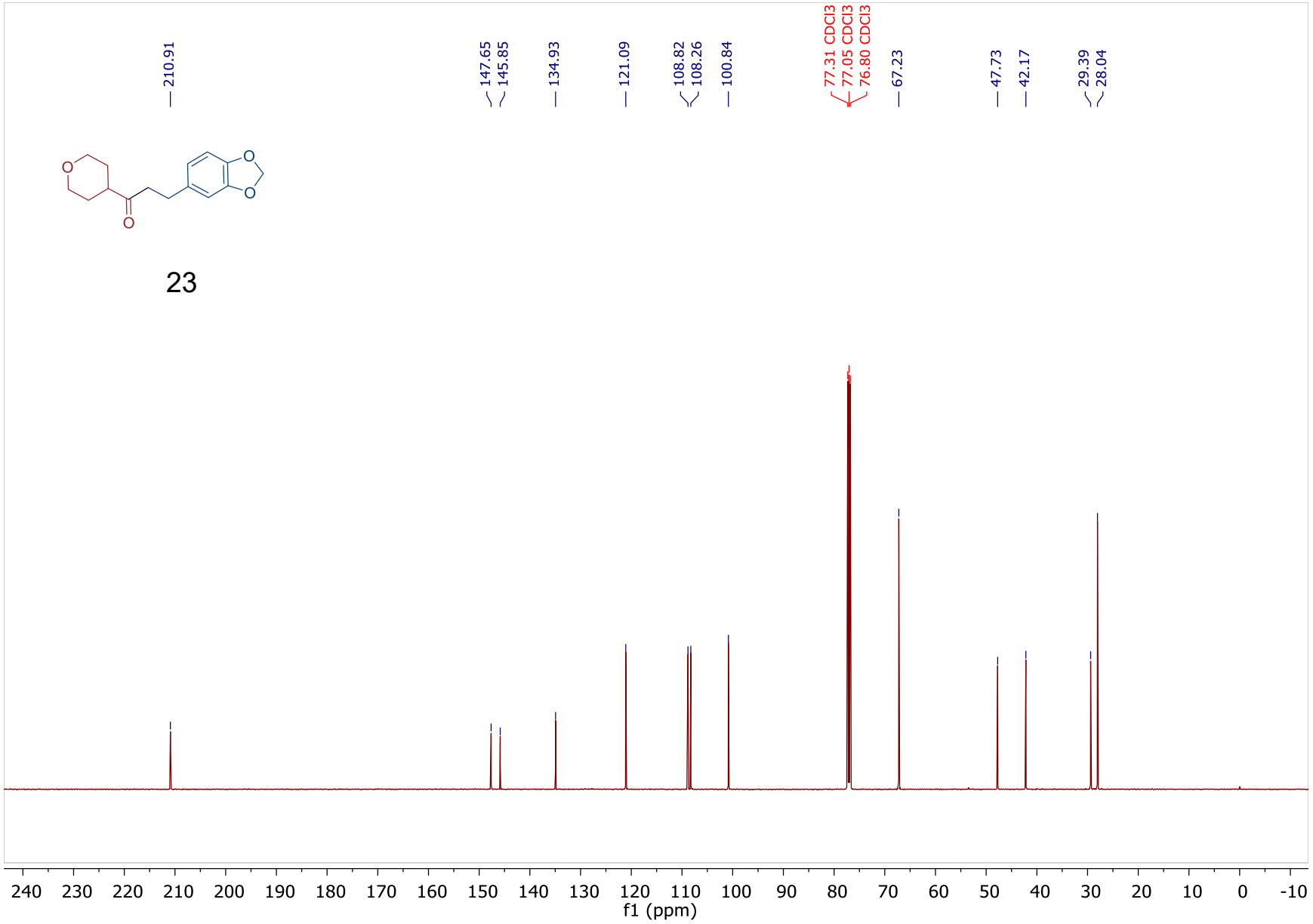

O
O
N
Me
Me
Me
O
O
O

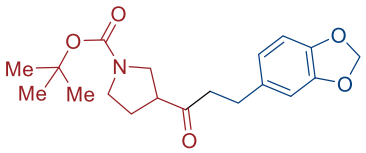
22


0.89
0.85
0.83
1.88
3.78
0.99
1.94
1.90
9.00
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


207.10
156.18
147.73
145.99
134.39
121.07
108.76
108.33
100.88
79.80
77.30 CDCl3
77.05 CDCl3
77.05 CDCl3
76.79 CDCl3
50.49
42.58
38.70
29.25
28.34
O
O
N
Me
Me
Me
O
O
O
O
22
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

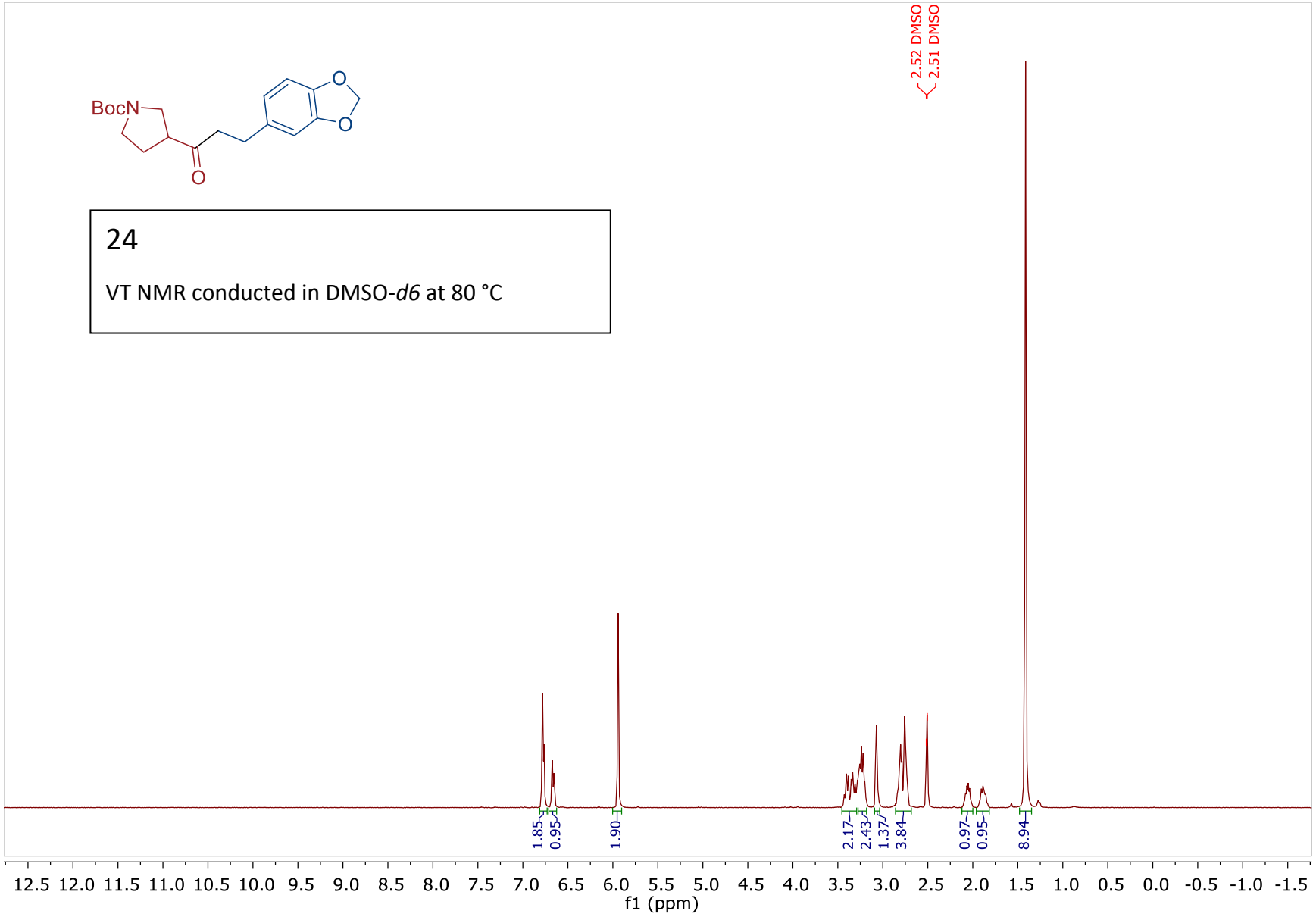

23
0.98
0.95
0.95
2.00
2.04
2.02
2.05
2.04
1.01
4.27
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

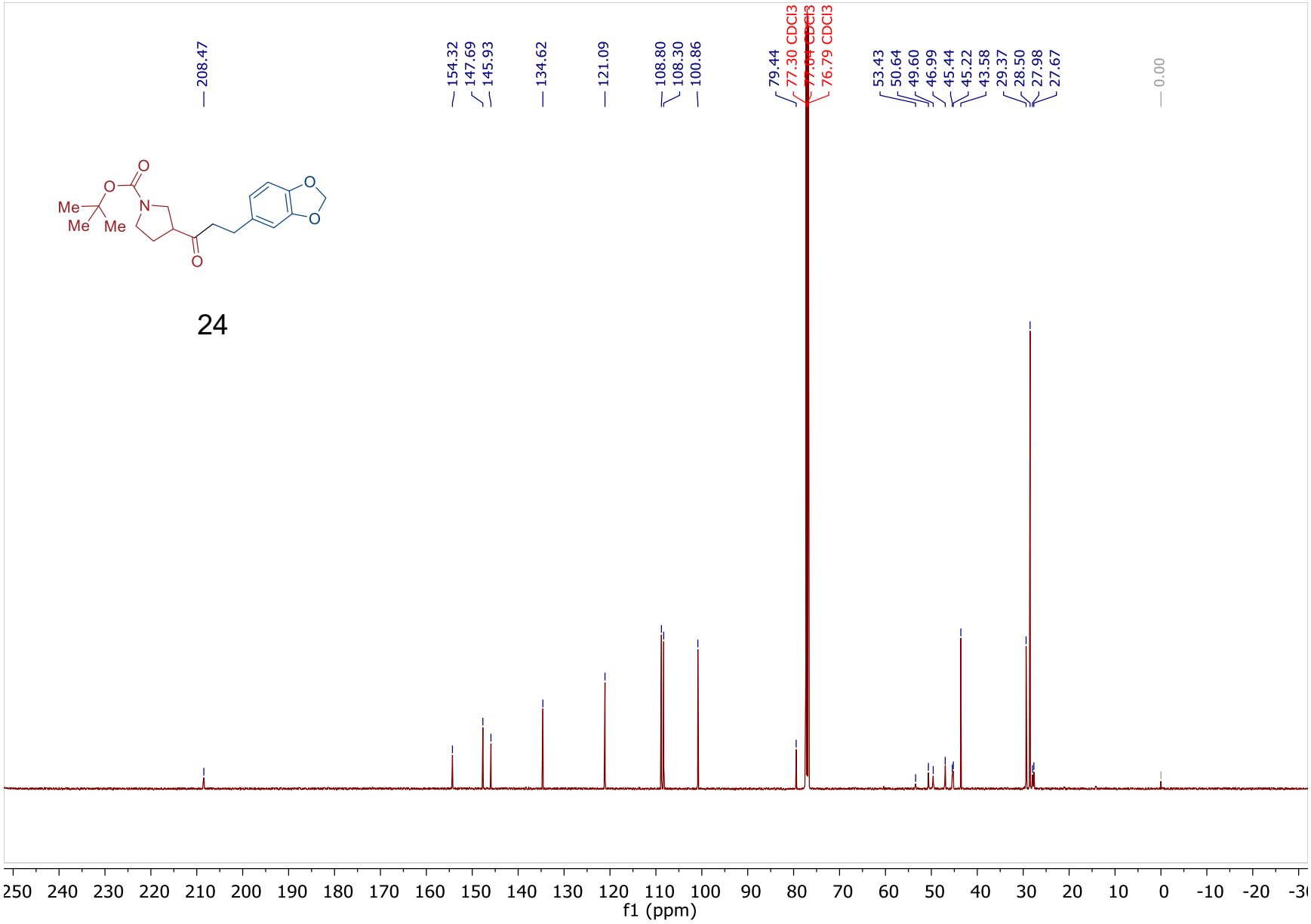

210.91
147.65
145.85
134.93
121.09
108.82
108.26
100.84
77.31 CDCl3
77.05 CDCl3
76.80 CDCl3
67.23
47.73
42.17
29.39
28.04
O
O
O
O
23
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

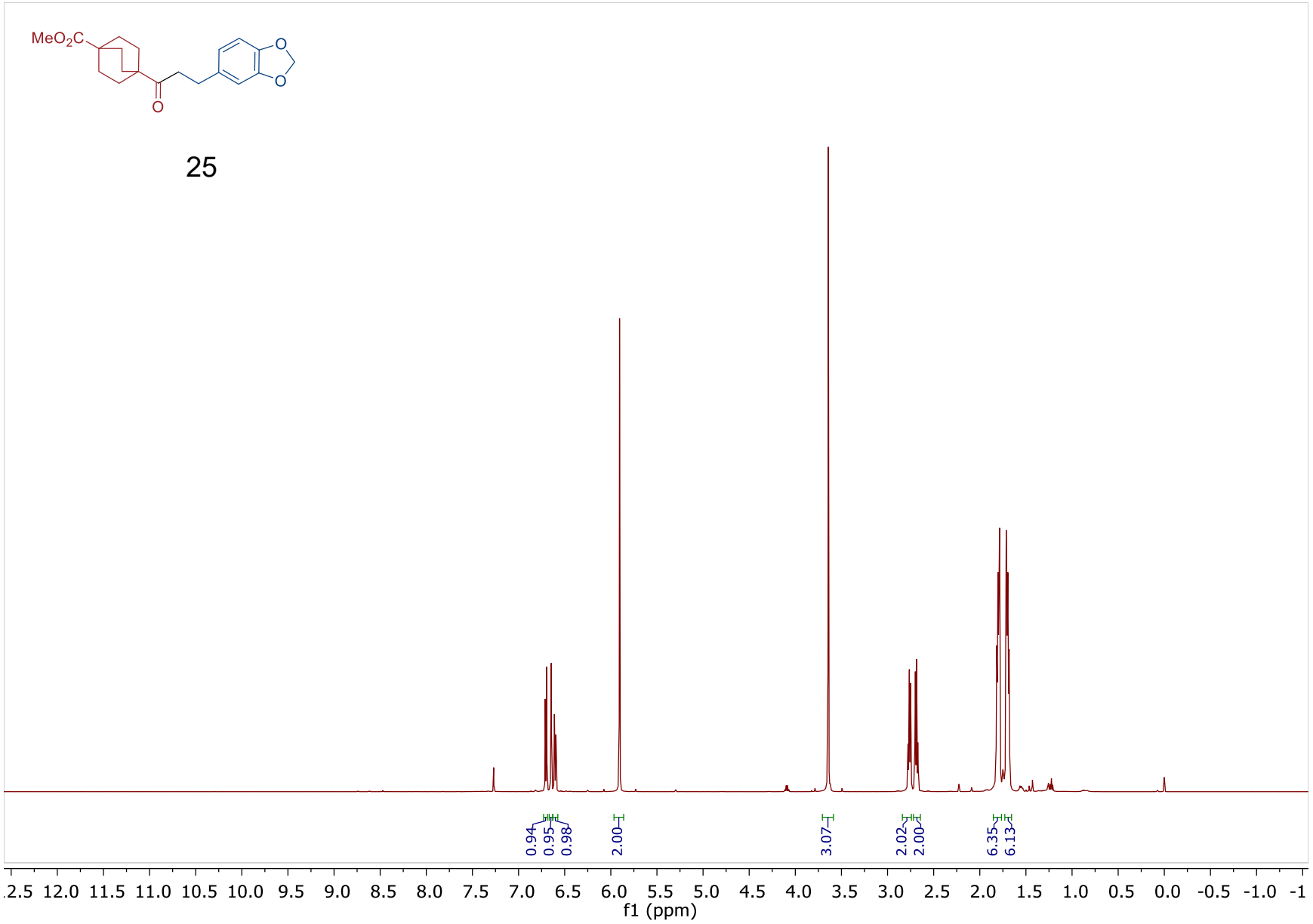

O
O
Me
Me
Me
N
O
O
O

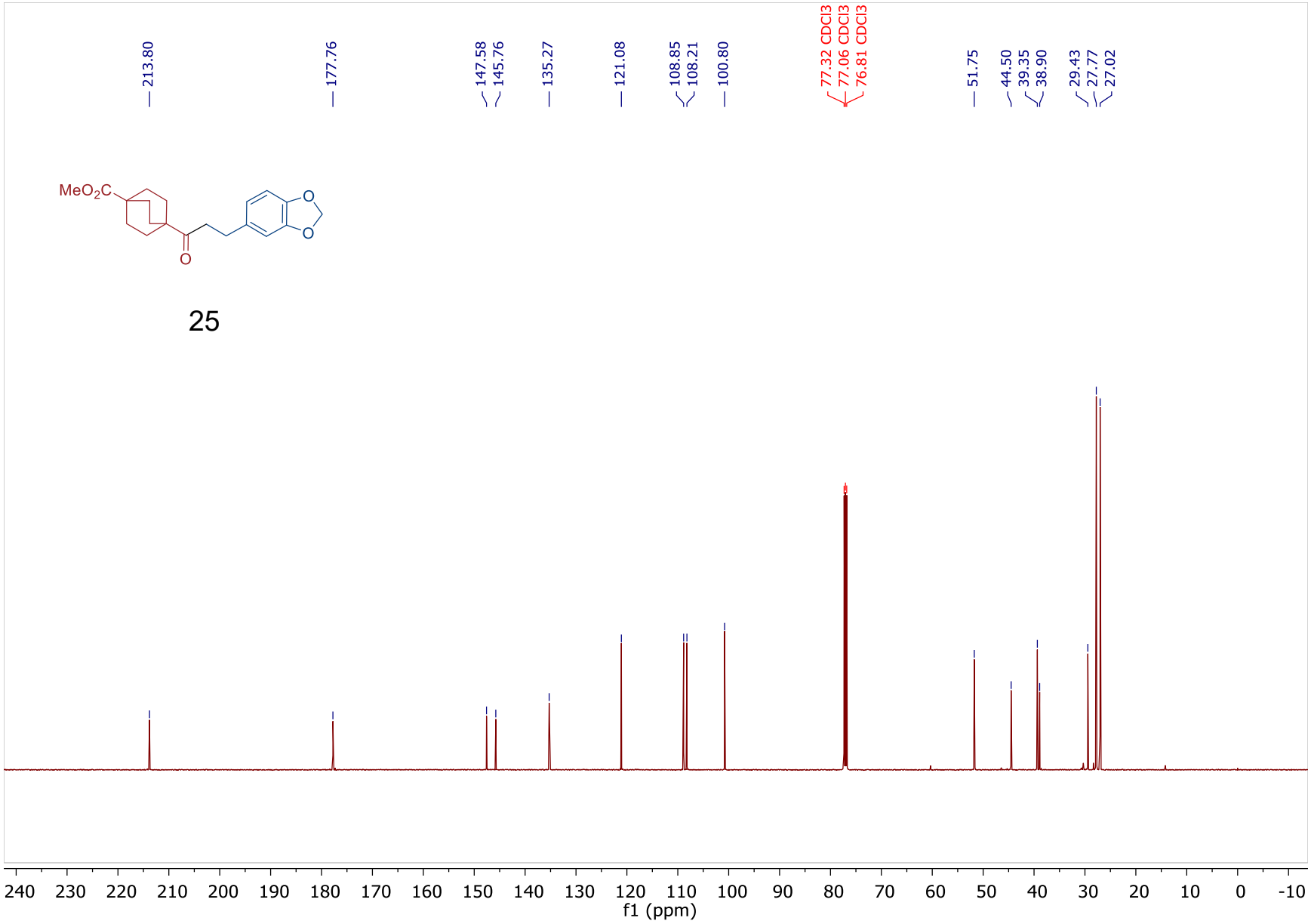
24

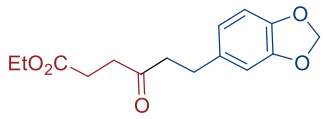
1.00
0.97
0.97
1.91
4.11
1.00
4.01
2.13
9.00
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


BocN
O
O
O
24
VT NMR conducted in DMSO-d6 at 80 °C
2.52 DMSO
2.51 DMSO
1.85
0.95
1.90
2.17
2.43
1.37
3.84
0.97
0.95
8.94
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)


208.47
154.32
147.69
145.93
134.62
121.09
108.80
108.30
100.86
79.44
77.30 CDCl3
77.04 CDCl3
76.79 CDCl3
53.43
50.64
49.60
46.99
45.44
45.22
43.58
29.37
28.50
27.98
27.67
0.00
Me
Me
Me
O
O
N
O
O
O
24
250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

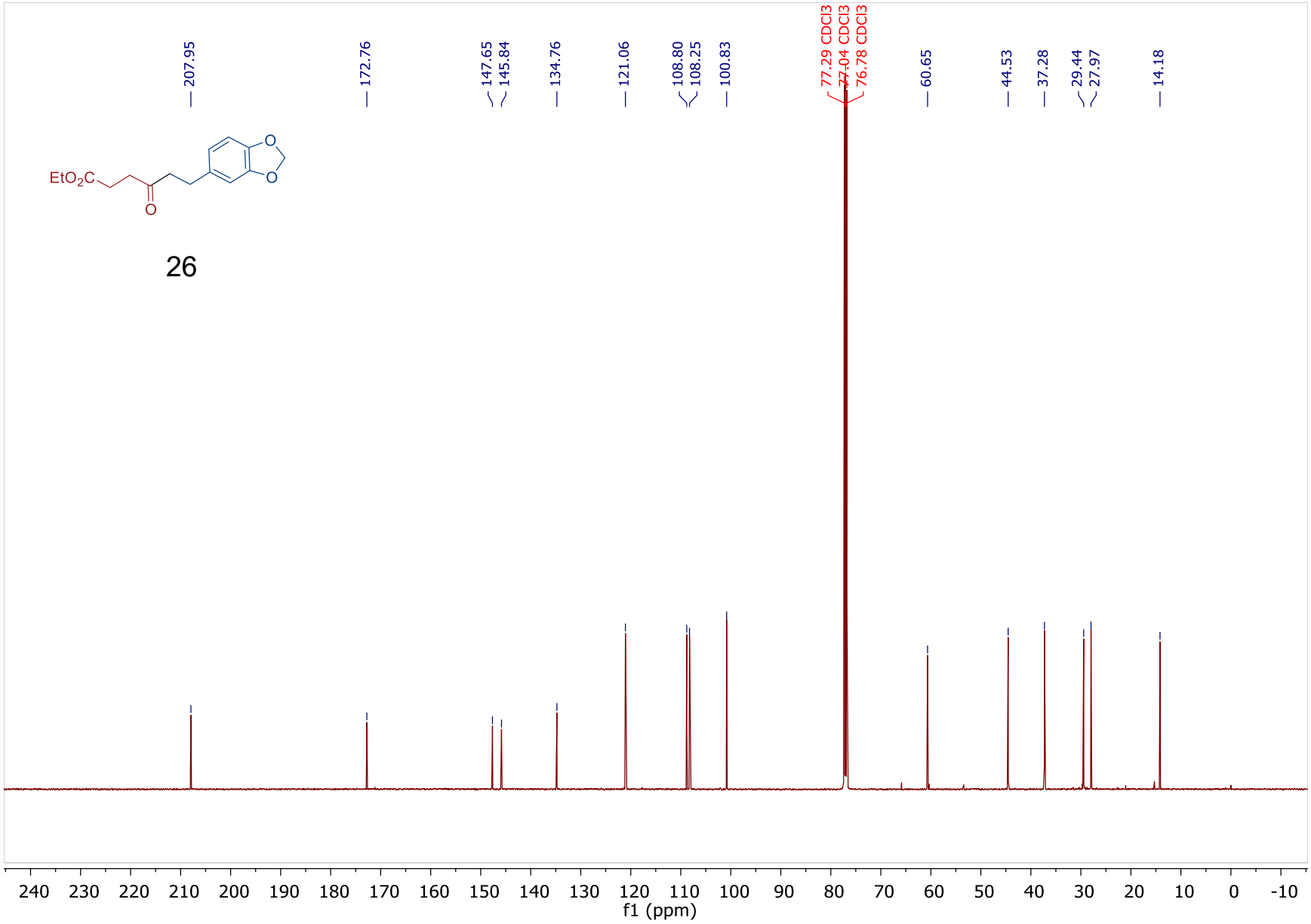

MeO2C
O
O
O
25
0.94
0.95
0.98
2.00
3.07
2.02
2.00
6.35
6.13
f1 (ppm)

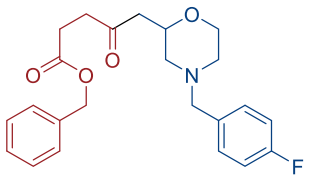

213.80
177.76
147.58
145.76
135.27
121.08
108.85
108.21
100.80
77.32 CDCl3
77.06 CDCl3
76.81 CDCl3
51.75
44.50
39.35
38.90
29.43
27.77
27.02
MeO2C
O
O
O
25
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


EtO2C
O
O
O

26

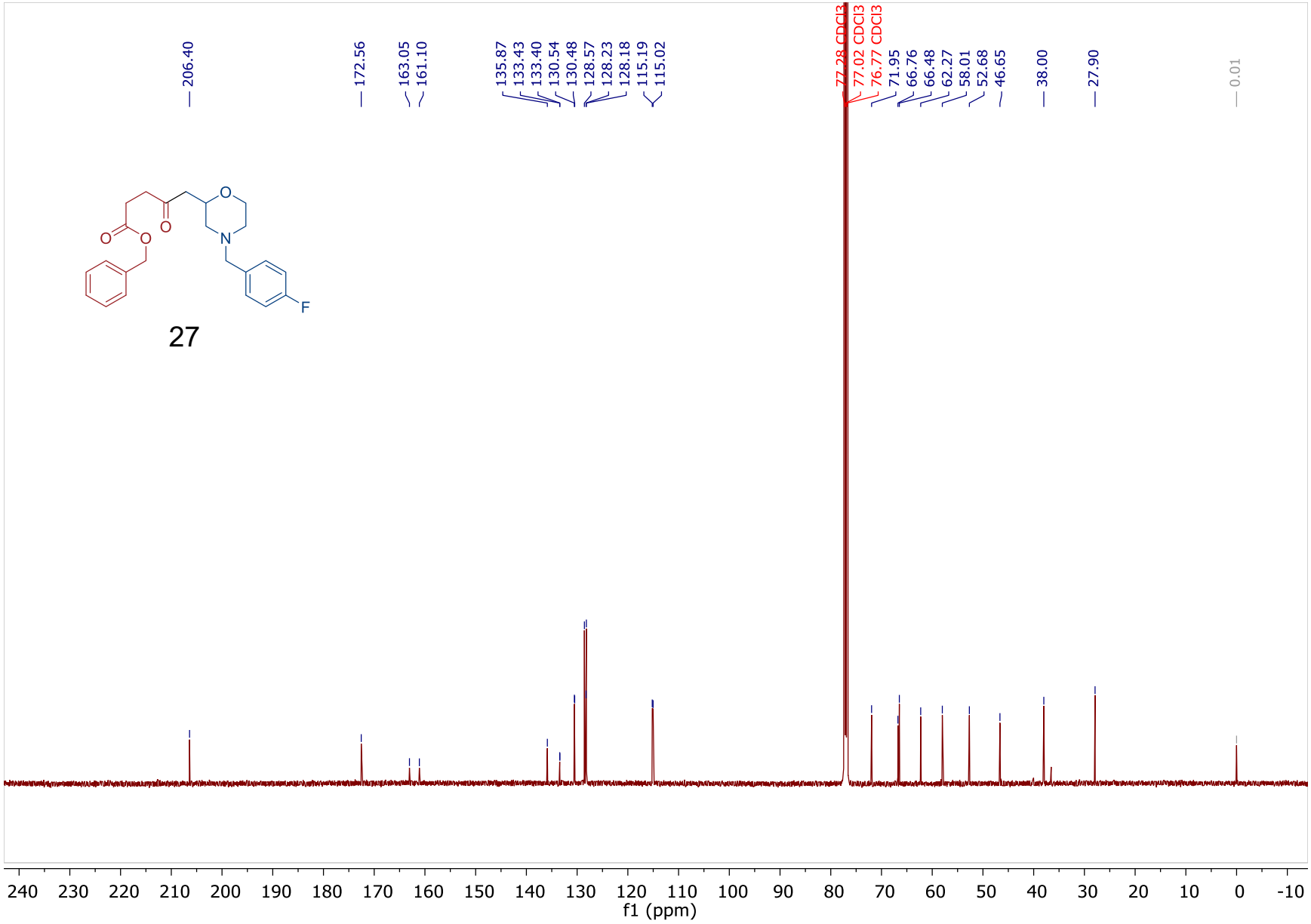
0.97
0.93
0.96
2.00
2.11
2.08
1.97
2.00
1.98
3.49
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.
f1 (ppm)

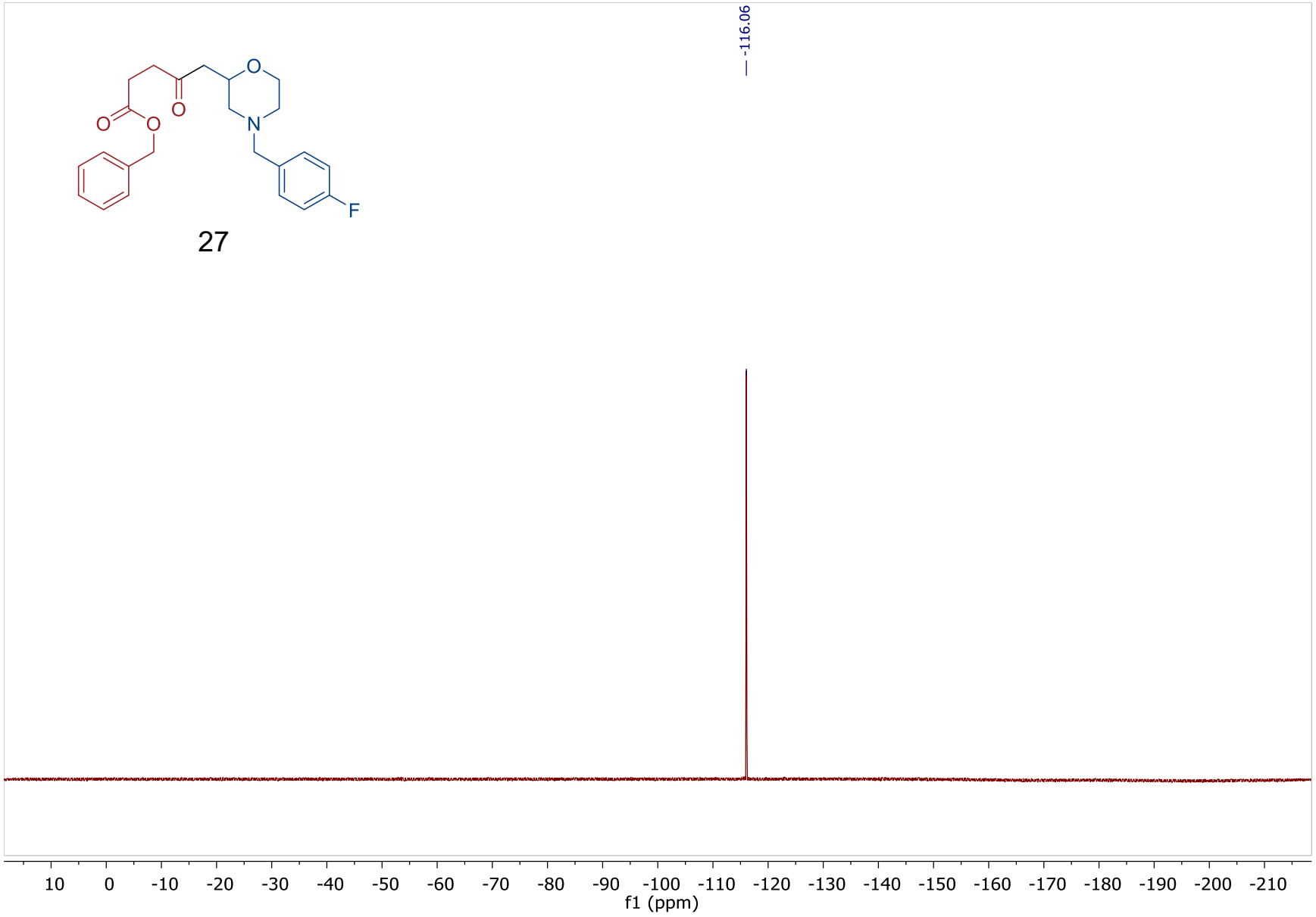

207.95
172.76
147.65
145.84
134.76
121.06
108.80
108.25
100.83
77.29 CDCl3
77.04 CDCl3
76.78 CDCl3
60.65
44.53
37.28
29.44
27.97
14.18
EtO2C
O
O
O
26
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

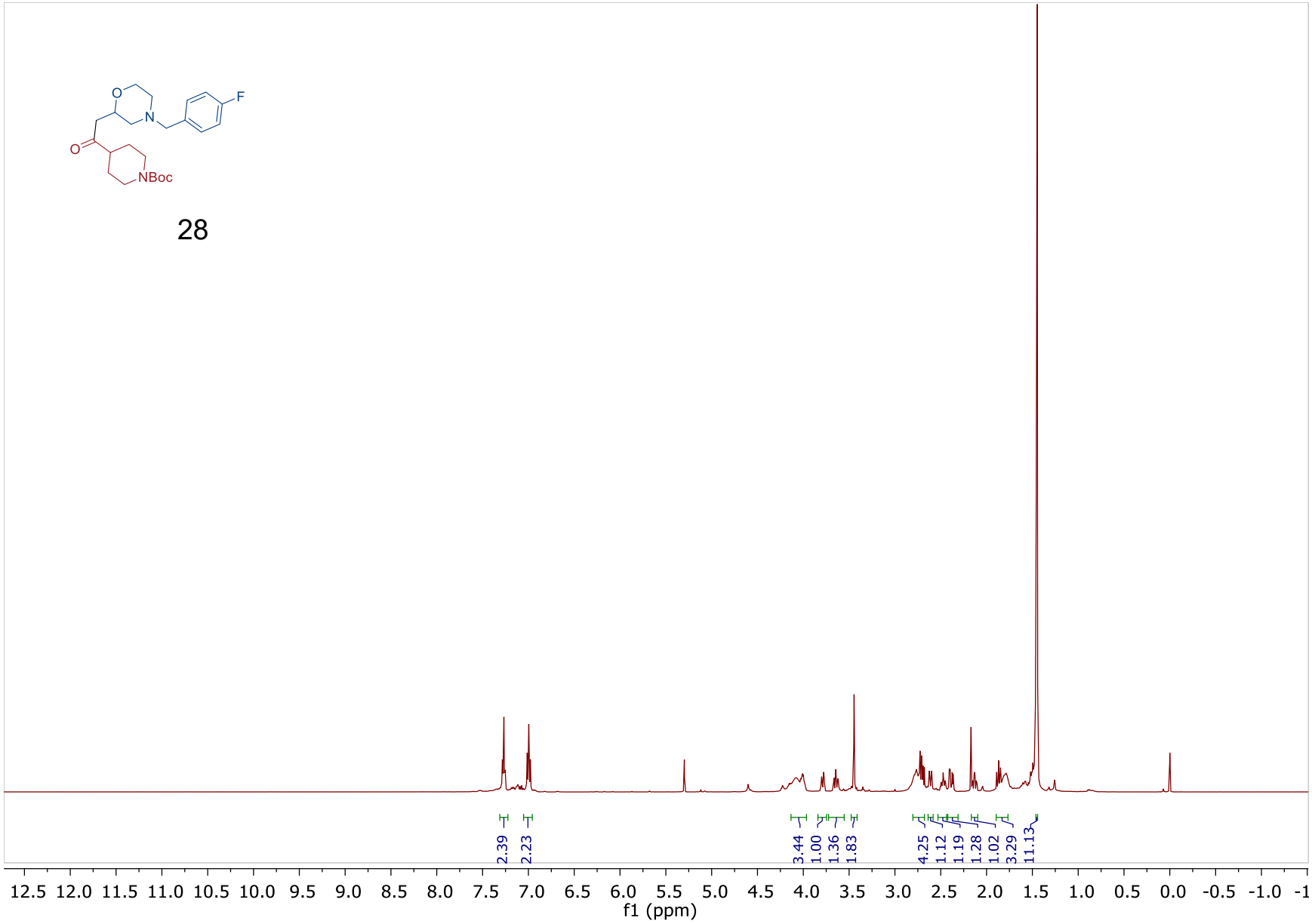

O
O
O
O
N
F

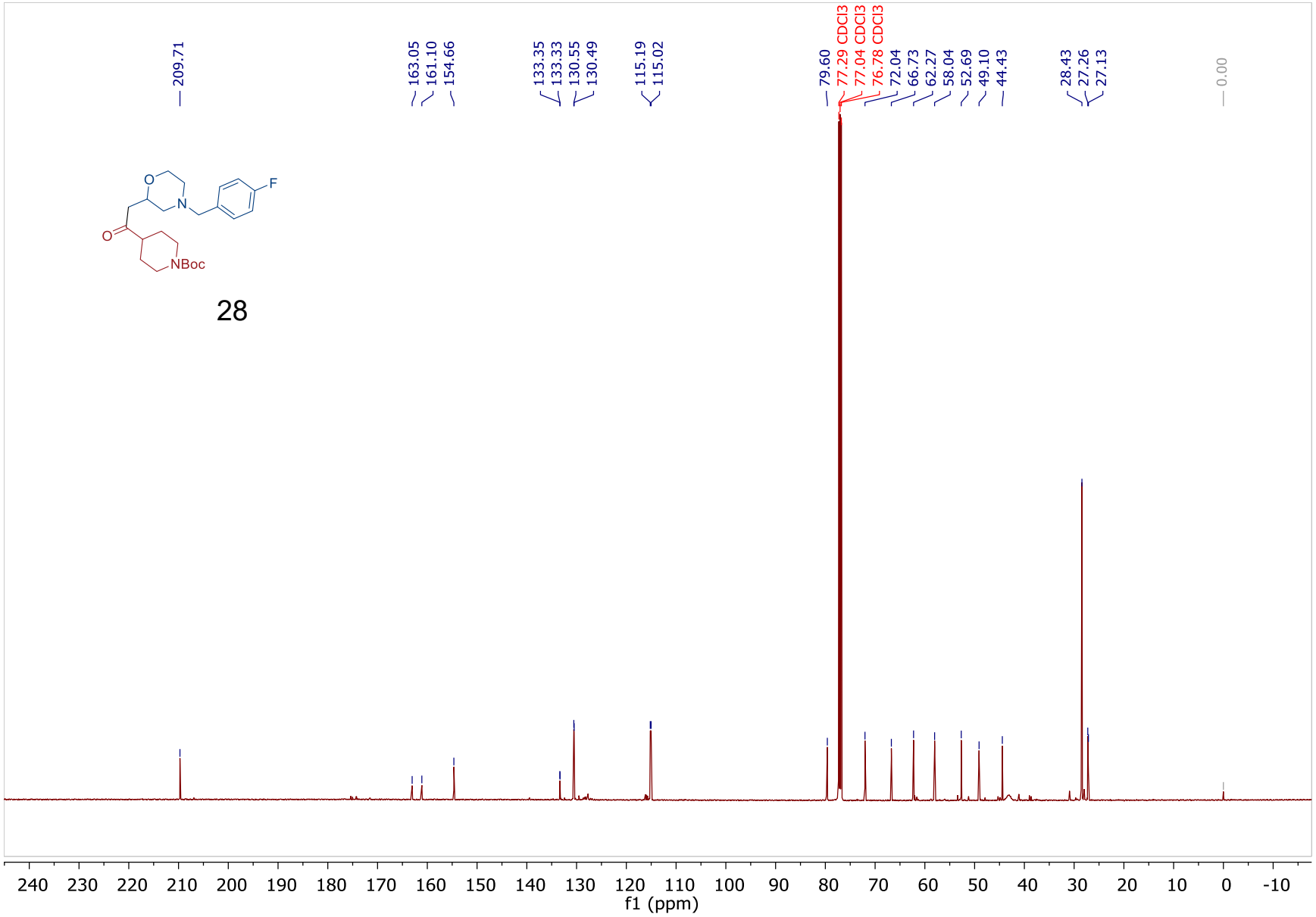
27

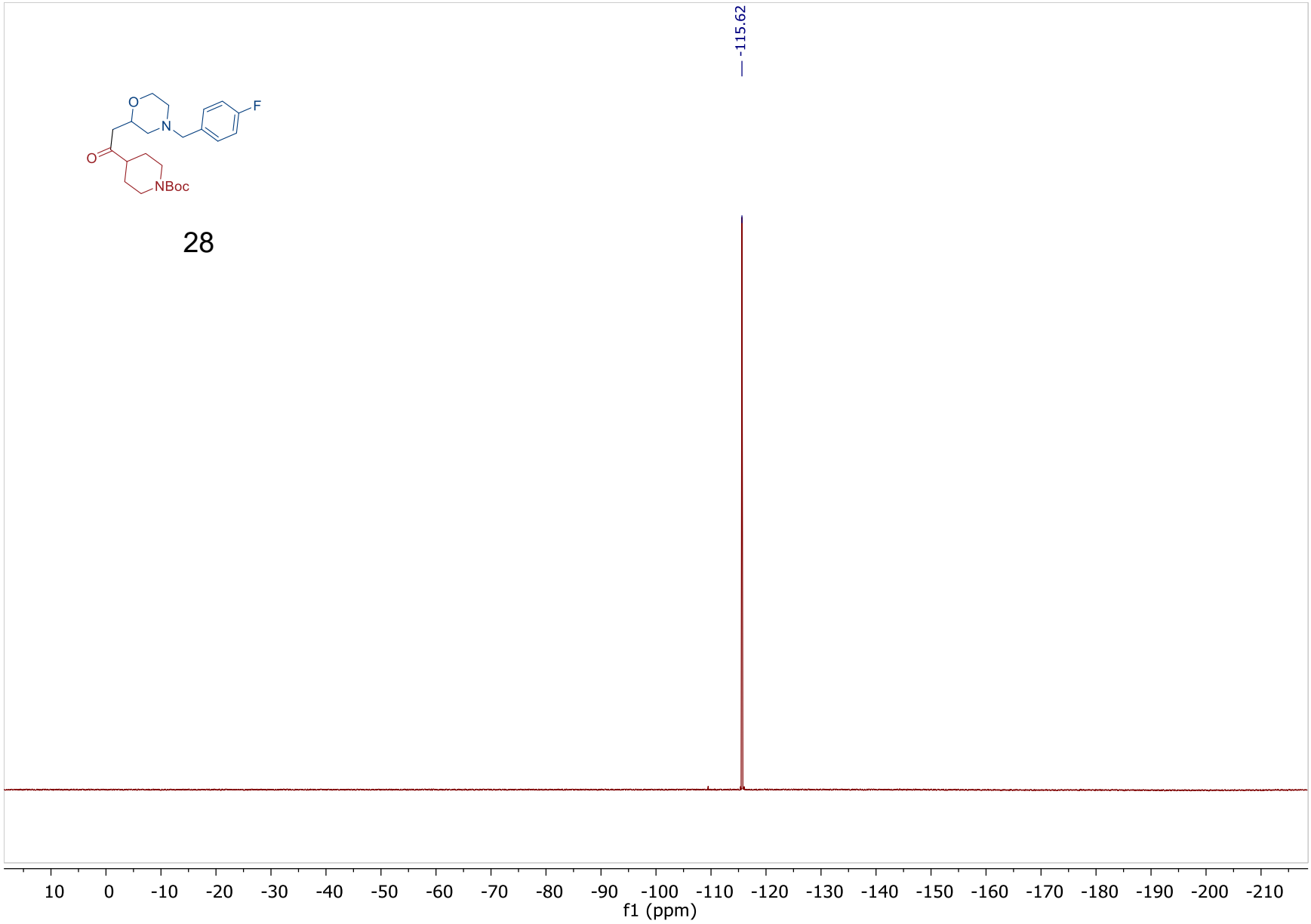
water
4.72
1.80
2.02
2.00
1.00
1.04
1.05
2.07
2.01
1.07
2.21
1.75
0.95
1.04
1.03
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

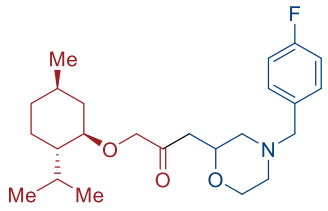

206.40
172.56
163.05
161.10
135.87
133.43
133.40
130.54
130.48
128.57
128.23
128.18
115.19
115.02
77.28 CDCl3
77.02 CDCl3
76.77 CDCl3
71.95
66.76
66.48
62.27
58.01
52.68
46.65
38.00
27.90
0.01
O
O
O
O
N
F
27
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-116.06
O
O
O
O
N
F
27
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)


O
N
F
O
NBoc
28
2.39
2.23
3.44
1.00
1.36
1.83
4.25
1.12
1.19
1.28
1.02
3.29
11.13
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1
f1 (ppm)

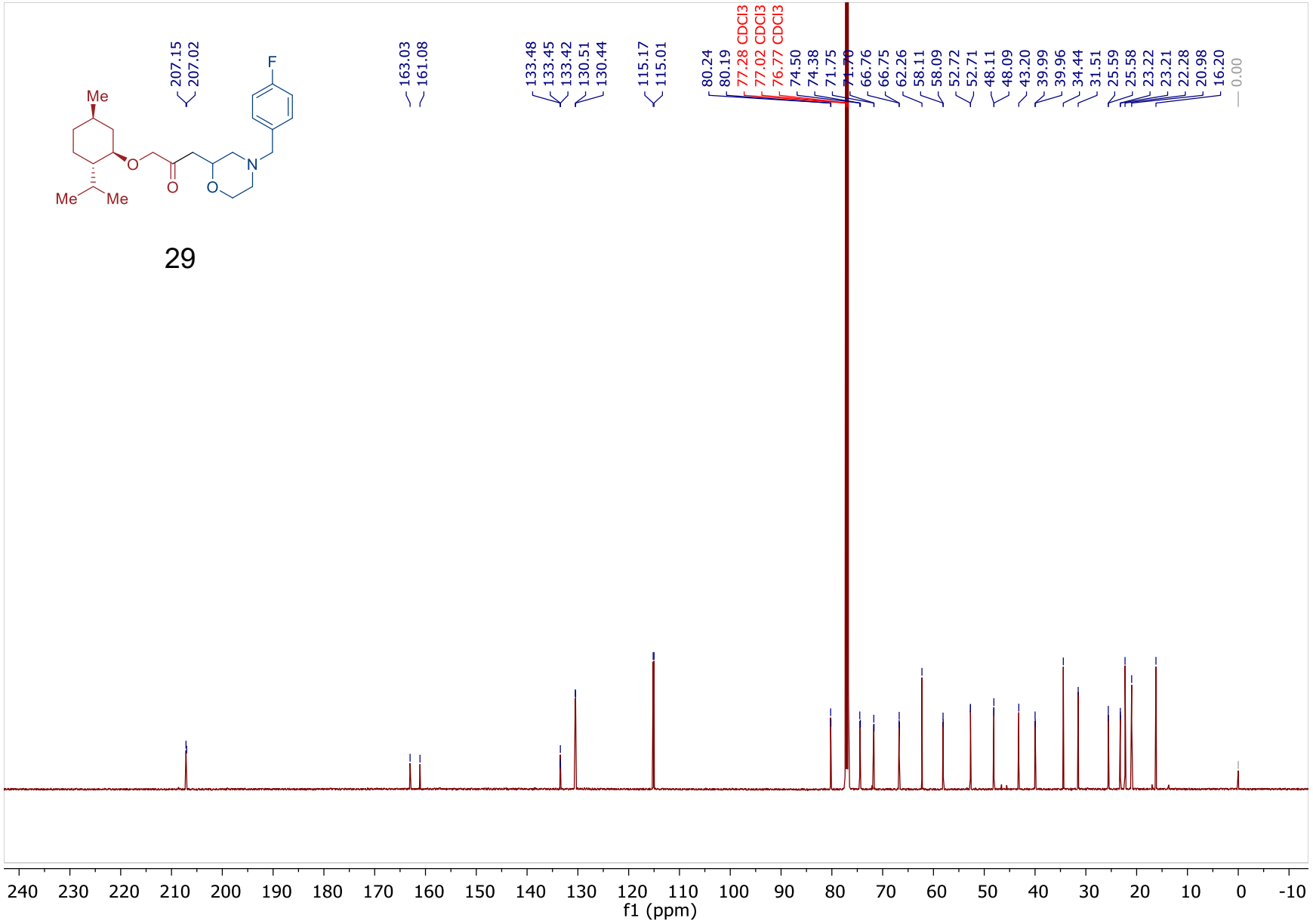

209.71
163.05
161.10
154.66
133.35
133.33
130.55
130.49
115.19
115.02
79.60
77.29 CDCl3
77.04 CDCl3
76.78 CDCl3
72.04
66.73
62.27
58.04
52.69
49.10
44.43
28.43
27.26
27.13
0.00
O
N
F
O
NBoc
28
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-115.62
O
N
F
O
NBoc
28
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)

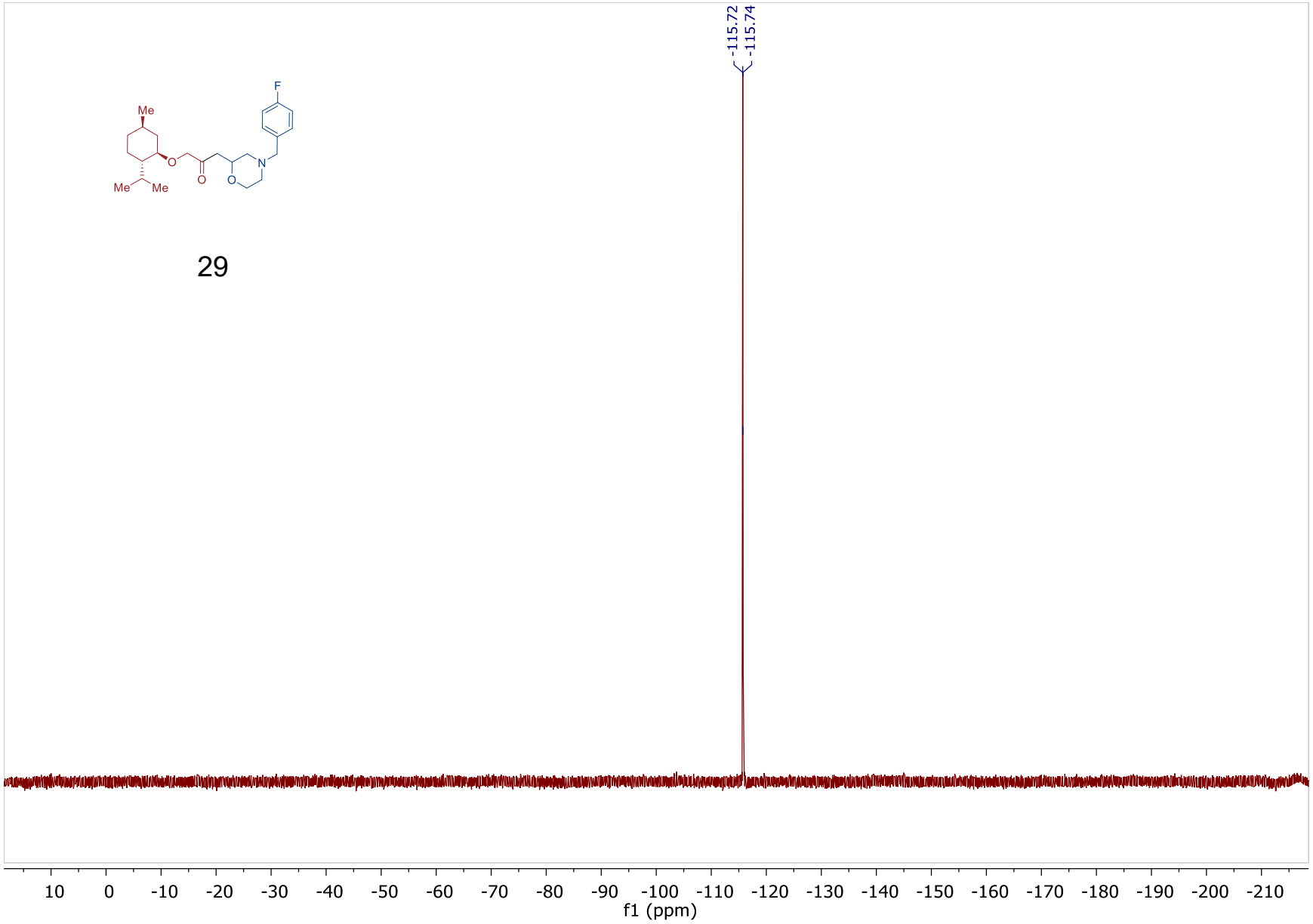

F
Me
O
N
O
O
Me
Me

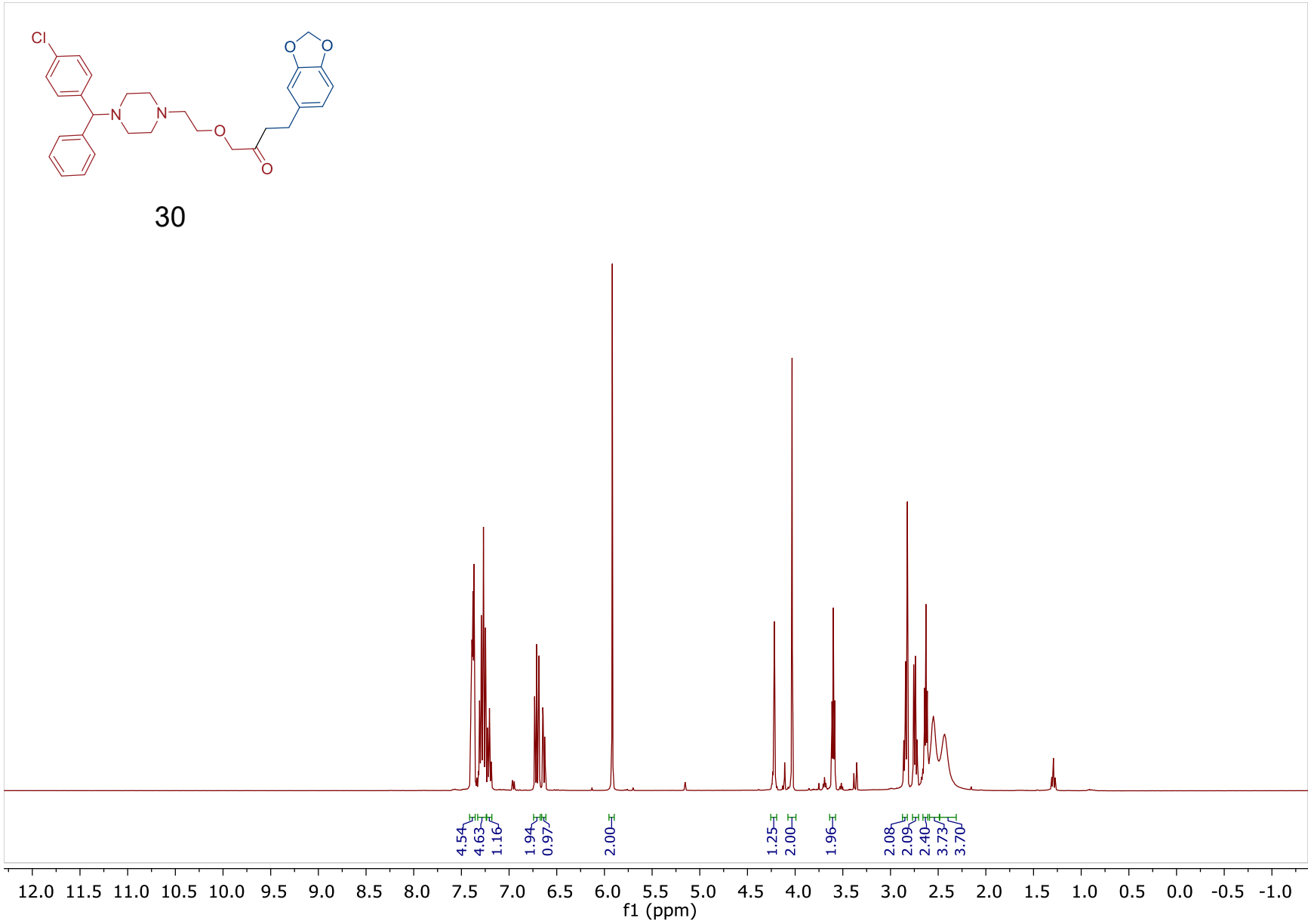
29

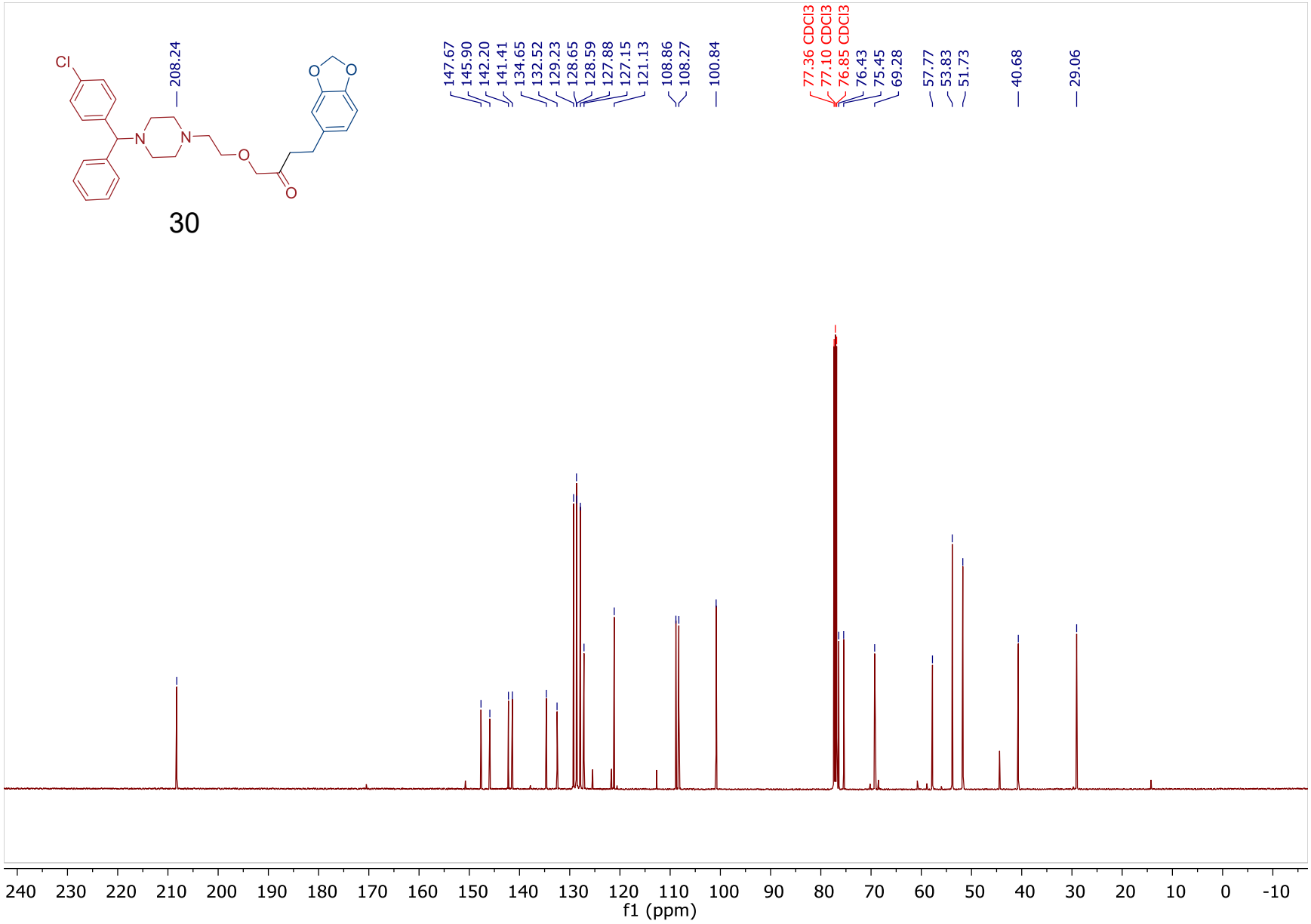
2.49
2.07
1.03
2.05
1.01
1.07
2.06
1.00
2.04
1.09
1.01
1.02
1.09
1.06
1.05
2.49
2.26
9.76
3.07
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

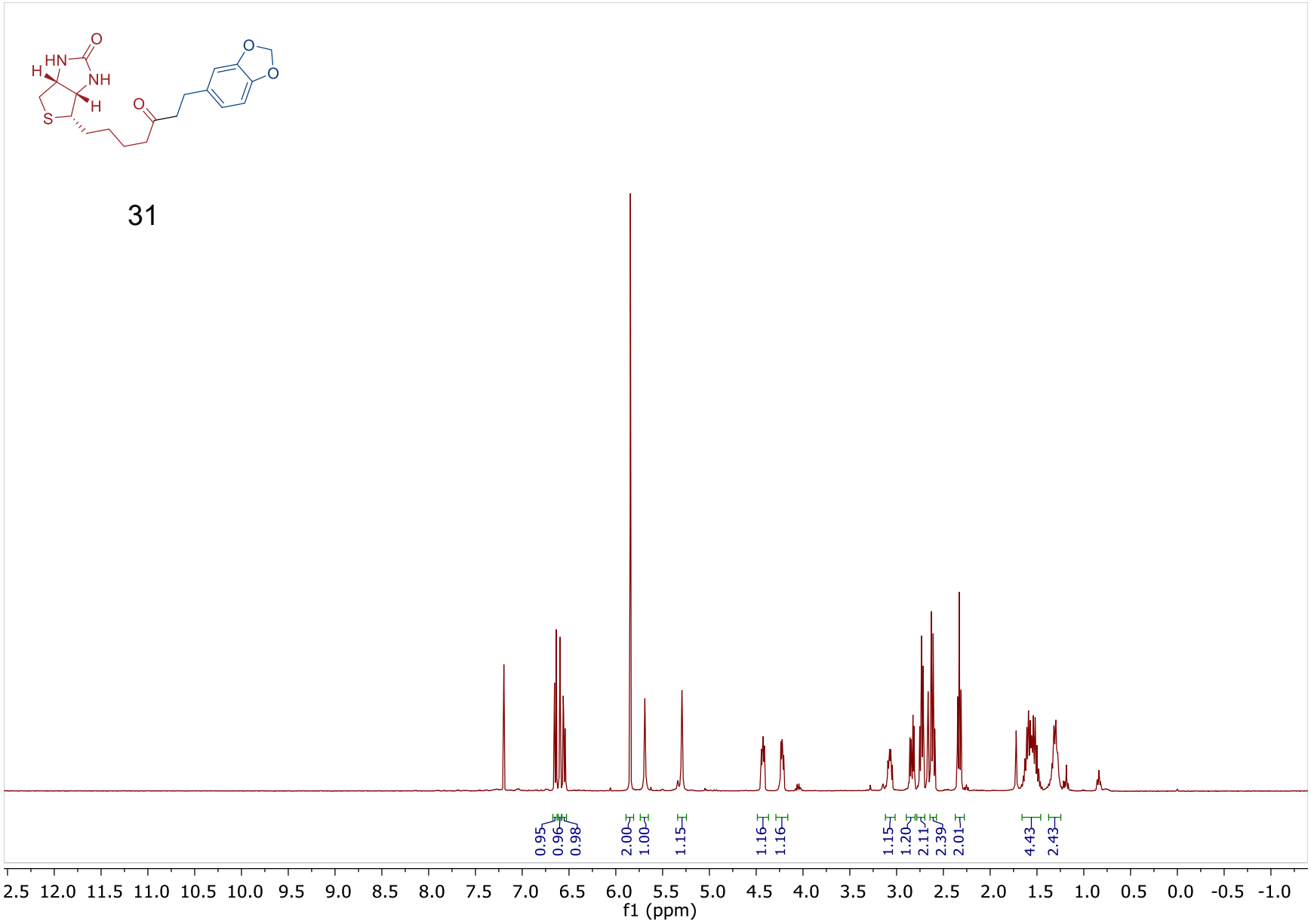

207.15
207.02
163.03
161.08
133.48
133.45
133.42
130.51
130.44
115.17
115.01
80.24
80.19
77.28 CDCl3
77.02 CDCl3
76.77 CDCl3
74.50
74.38
71.75
71.70
66.76
66.75
62.26
58.11
58.09
52.72
52.71
48.11
48.09
43.20
39.99
39.96
34.44
31.51
25.59
25.58
23.22
23.21
22.28
20.98
16.20
0.00
Me
Me
Me
O
O
O
N
F
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

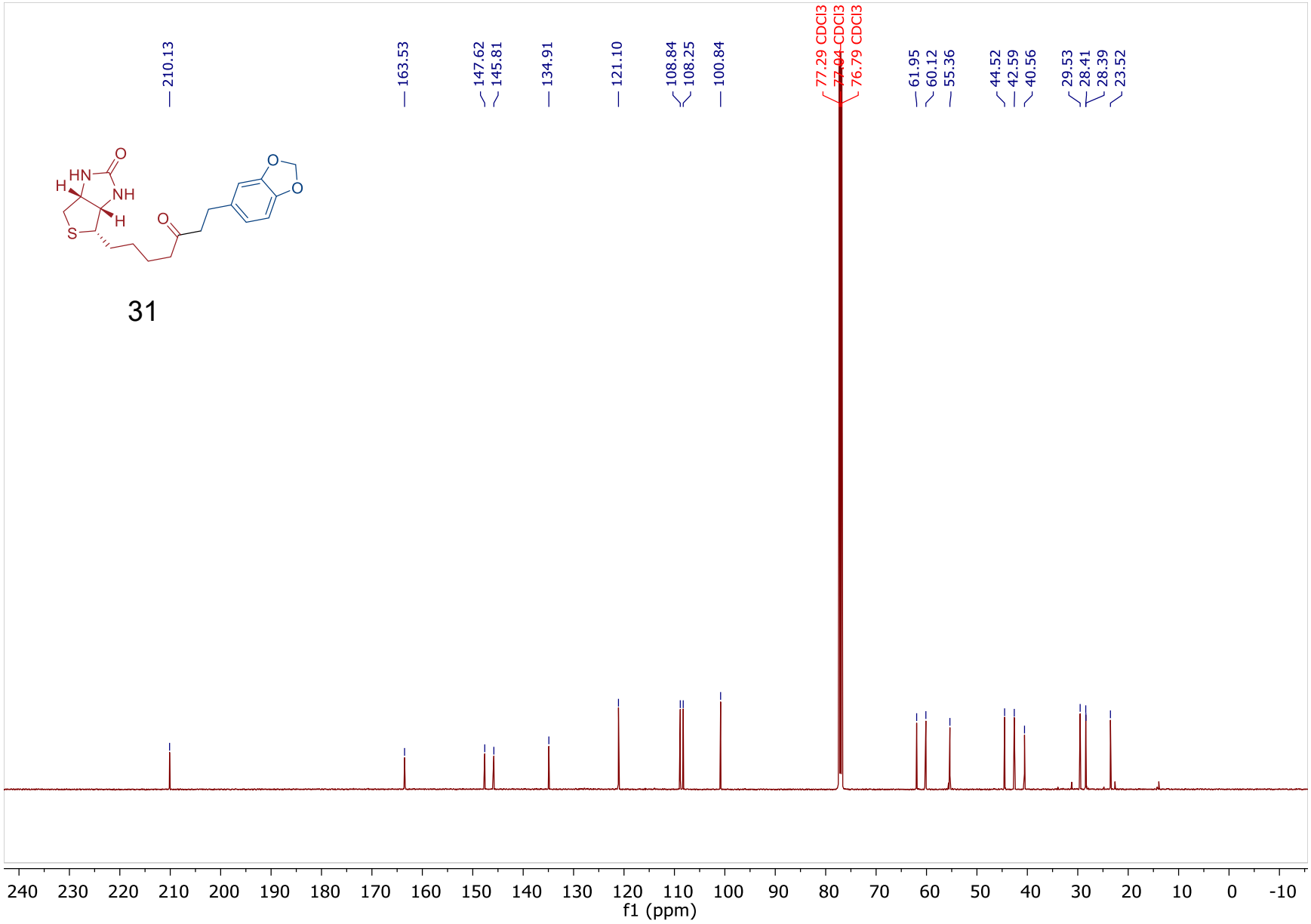
29

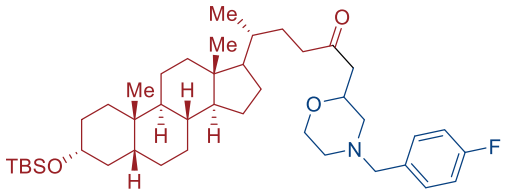

-115.72
-115.74
F
Me
O
O
N
O
Me
Me
29
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)


Cl
N
N
O
O
O
O
30
4.54
4.63
1.16
1.94
0.97
2.00
1.25
2.00
1.96
2.08
2.09
2.40
3.73
3.70
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


Cl
N
N
O
O
O
O
30
208.24
147.67
145.90
142.20
141.41
134.65
132.52
129.23
128.65
128.59
127.88
127.15
121.13
108.86
108.27
100.84
77.36 CDCl3
77.10 CDCl3
76.85 CDCl3
76.43
75.45
69.28
57.77
53.83
51.73
40.68
29.06
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

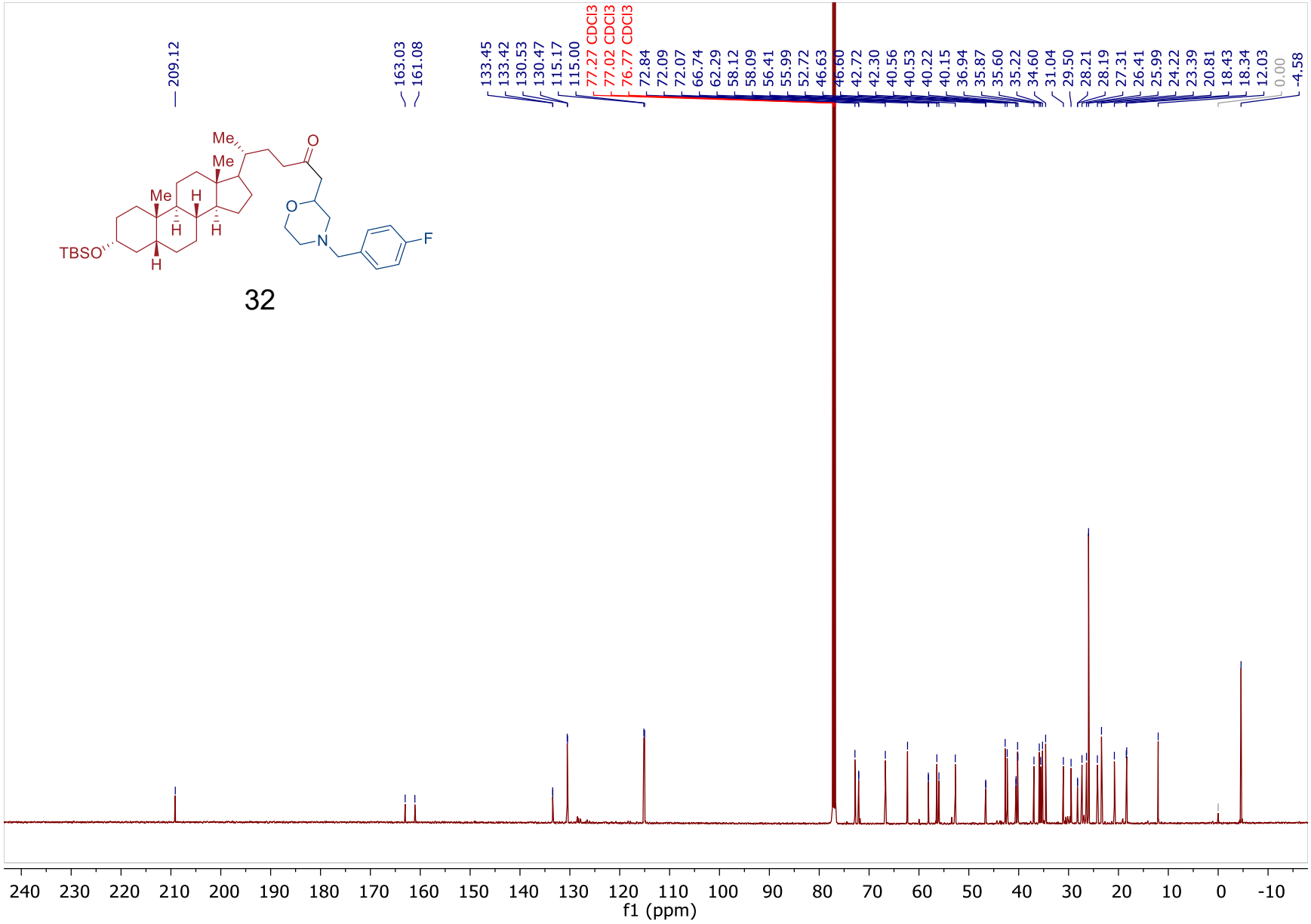

31
0.95
0.96
0.98
2.00
1.00
1.15
1.16
1.16
1.15
1.20
2.11
2.39
2.01
4.43
2.43
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

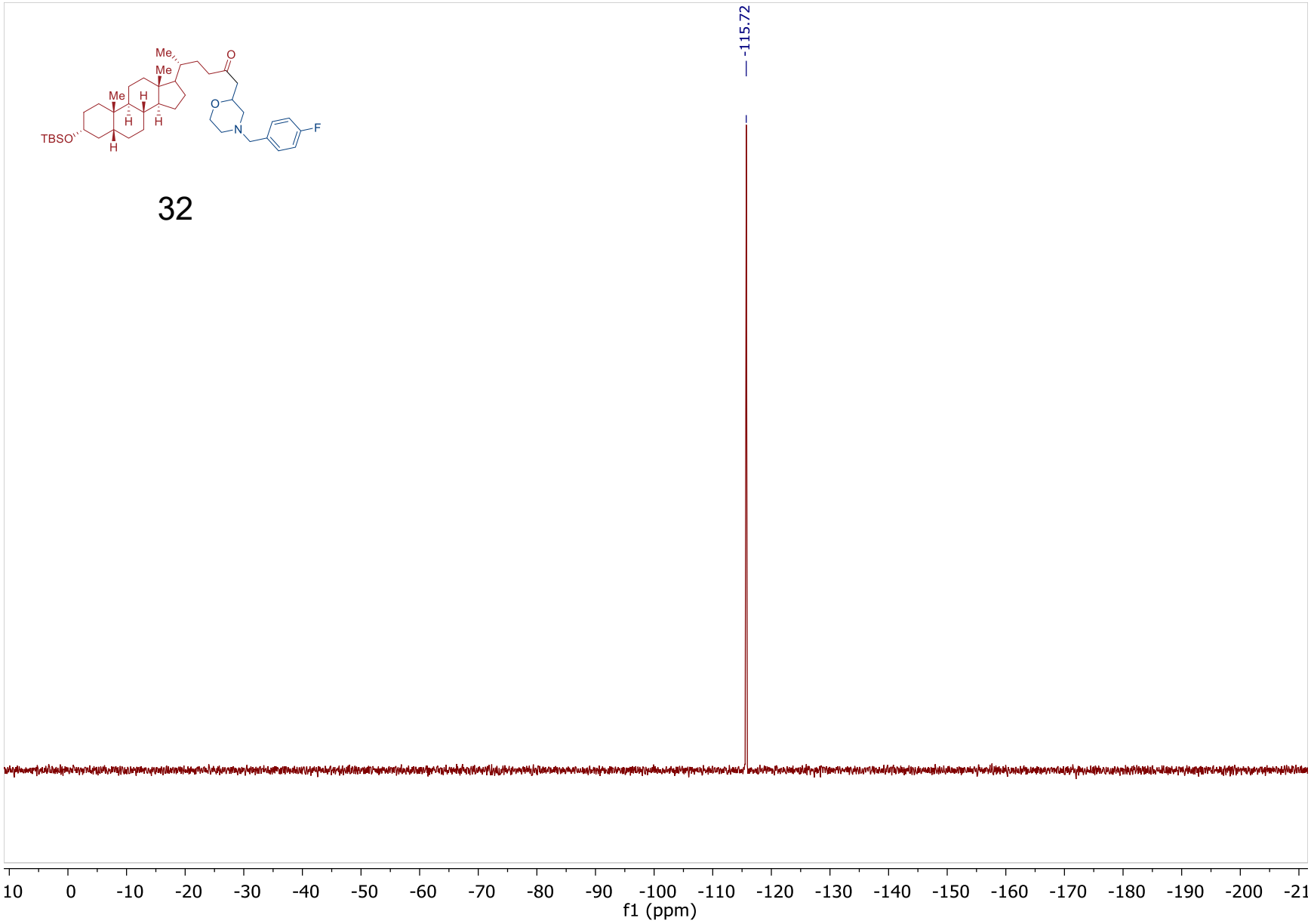

210.13
163.53
147.62
145.81
134.91
121.10
108.84
108.25
100.84
77.29 CDCl3
77.04 CDCl3
76.79 CDCl3
61.95
60.12
55.36
44.52
42.59
40.56
29.53
28.41
28.39
23.52
31
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

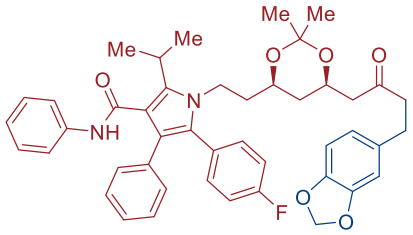

Me
Me
Me
H
H
H
H
TBSO
O
O
N
F

32

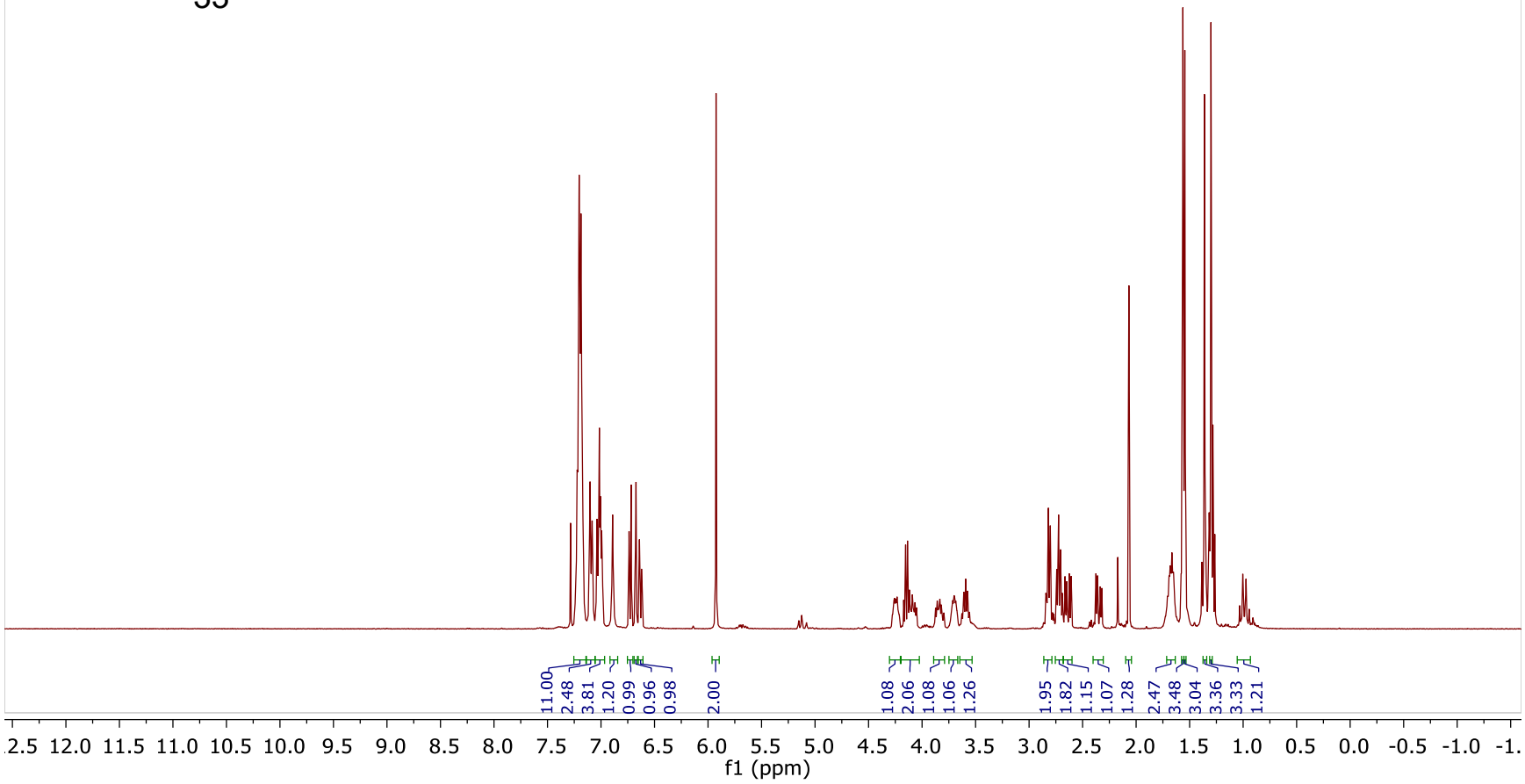
2.40
2.18
1.00
1.05
1.10
1.16
2.20
1.09
2.17
1.06
2.12
1.11
8.05
2.49
9.05
5.80
6.23
16.85
3.21
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.
f1 (ppm)

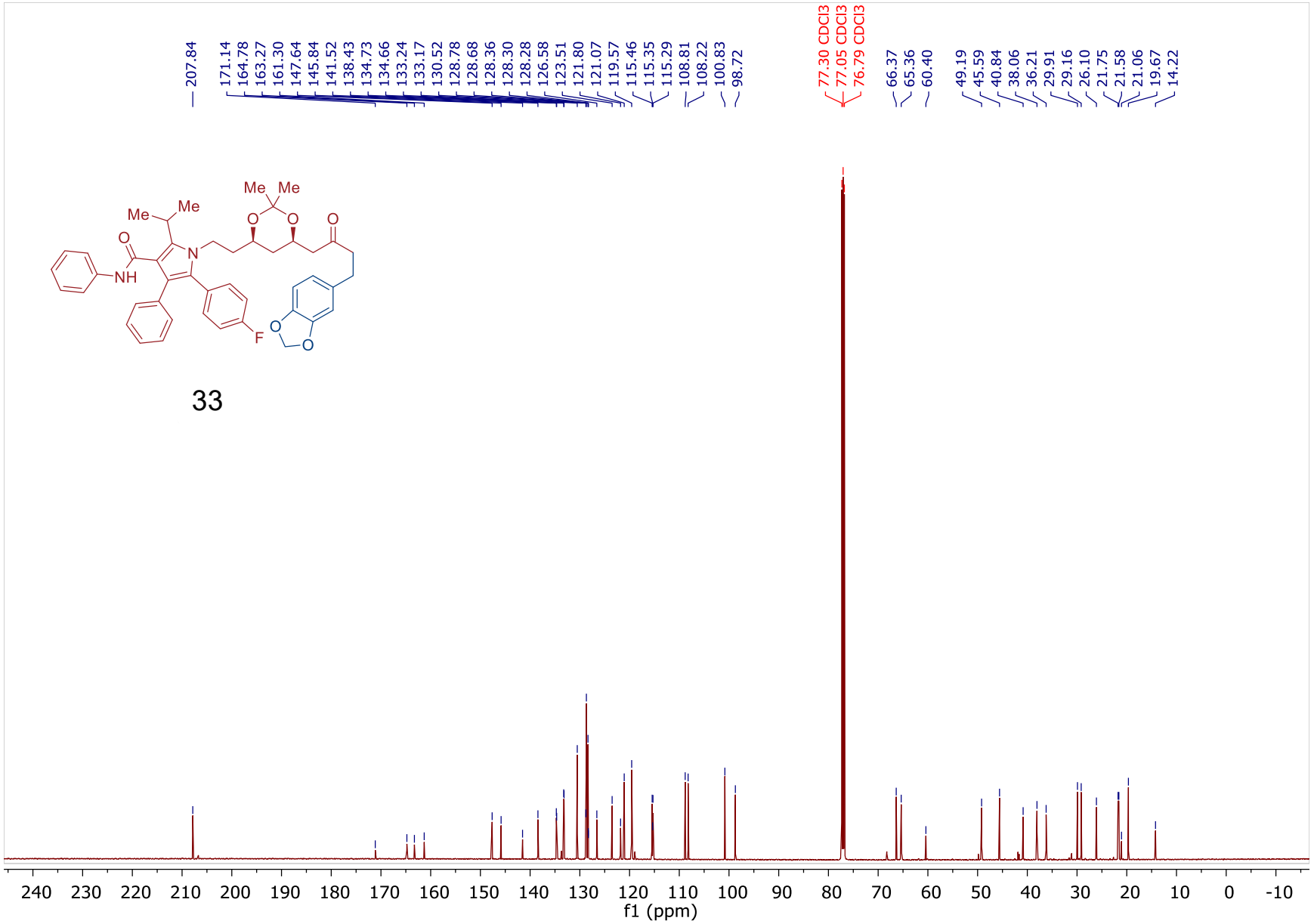

209.12
163.03
161.08
133.45
133.42
130.53
130.47
115.17
115.00
77.27 CDCl3
77.02 CDCl3
76.77 CDCl3
72.84
72.09
72.07
66.74
62.29
58.12
58.09
56.41
55.99
52.72
46.63
46.60
42.72
42.30
40.56
40.53
40.22
40.15
36.94
35.87
35.60
35.22
34.60
31.04
29.50
28.21
28.19
27.31
26.41
25.99
24.22
23.39
20.81
18.43
18.34
12.03
0.00
-4.58
Me
Me
Me
H
H
H
H
O
O
N
F
TBSO
32
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

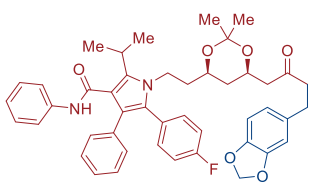

-115.72
Me
Me
Me
H
H
H
H
TBSO
O
O
N
F
32
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-21
f1 (ppm)


Me
Me
Me
Me
O
O
O
O
N
NH
F
O
O

33

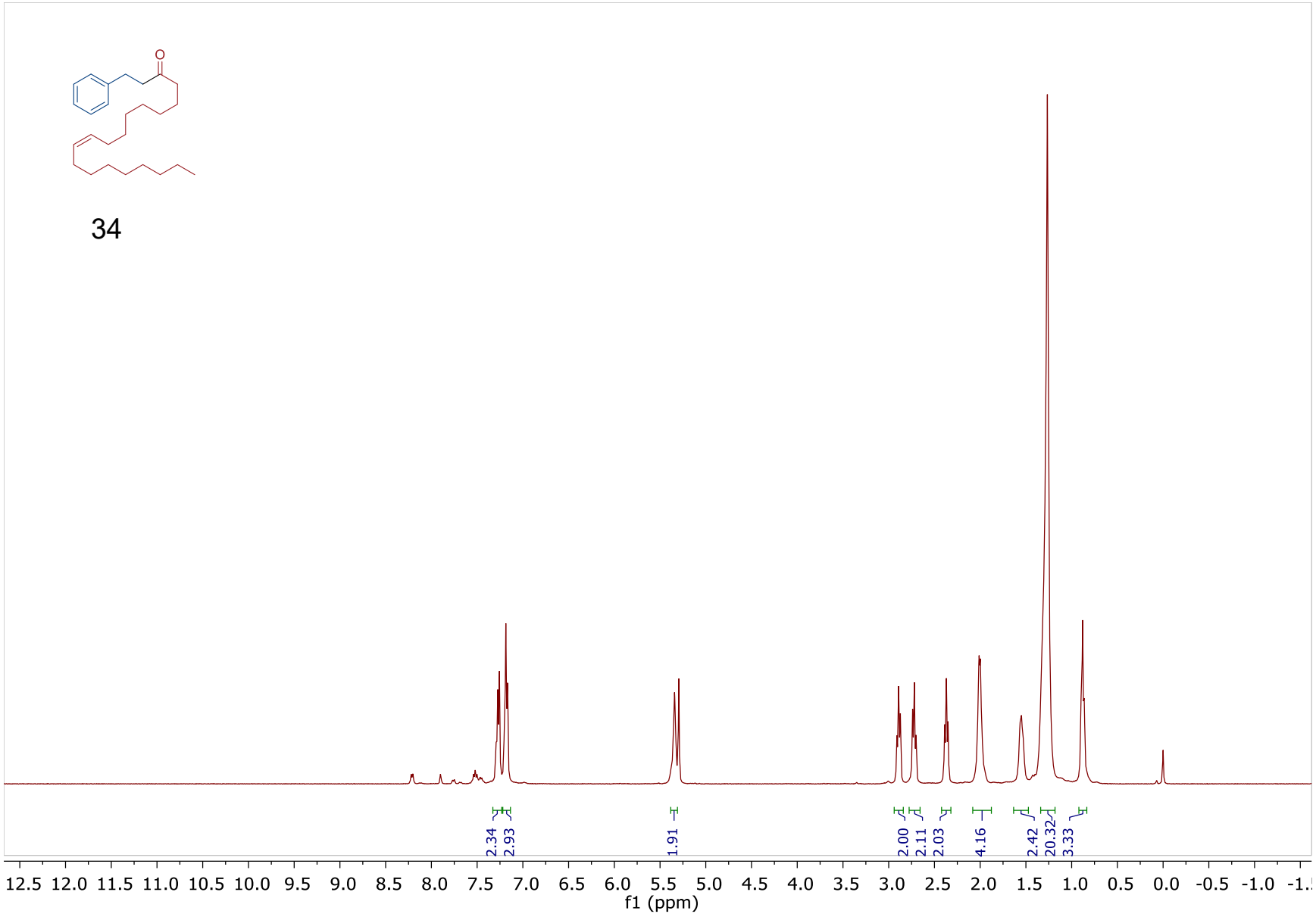

11.00
2.48
3.81
1.20
0.99
0.96
0.98
2.00
1.08
2.06
1.08
1.06
1.26
1.95
1.82
1.15
1.07
1.28
2.47
3.48
3.04
3.36
3.33
1.21
f1 (ppm)

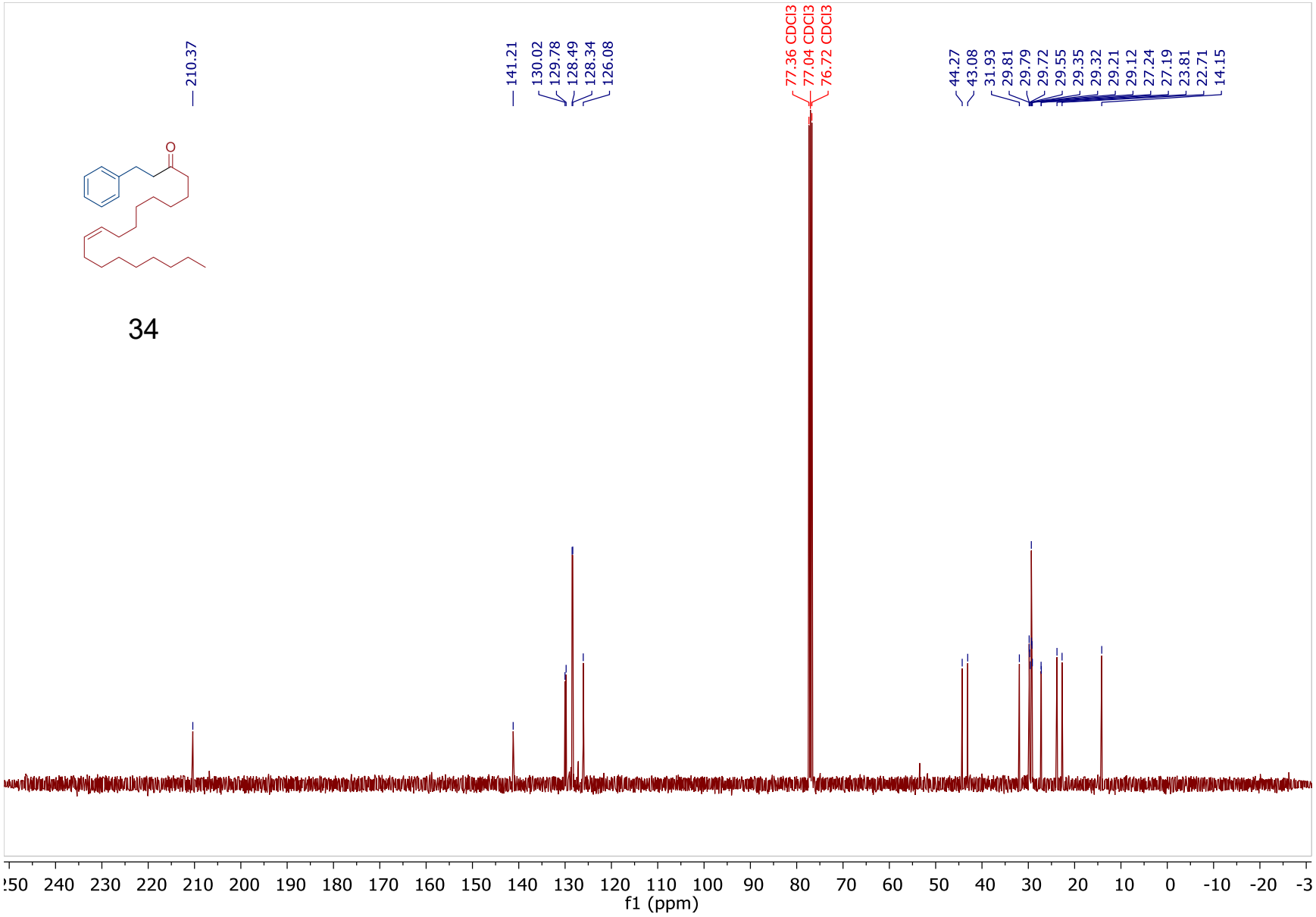

207.84
171.14
164.78
163.27
161.30
147.64
145.84
141.52
138.43
134.73
134.66
133.24
133.17
130.52
128.78
128.68
128.36
128.30
128.28
126.58
123.51
121.80
121.07
119.57
115.46
115.35
115.29
108.81
108.22
100.83
98.72
77.30 CDCl3
77.05 CDCl3
76.79 CDCl3
66.37
65.36
60.40
49.19
45.59
40.84
38.06
36.21
29.91
29.16
26.10
21.75
21.58
21.06
19.67
14.22
Me
Me
Me
Me
O
O
O
O
N
NH
F
O
O
33
240
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)

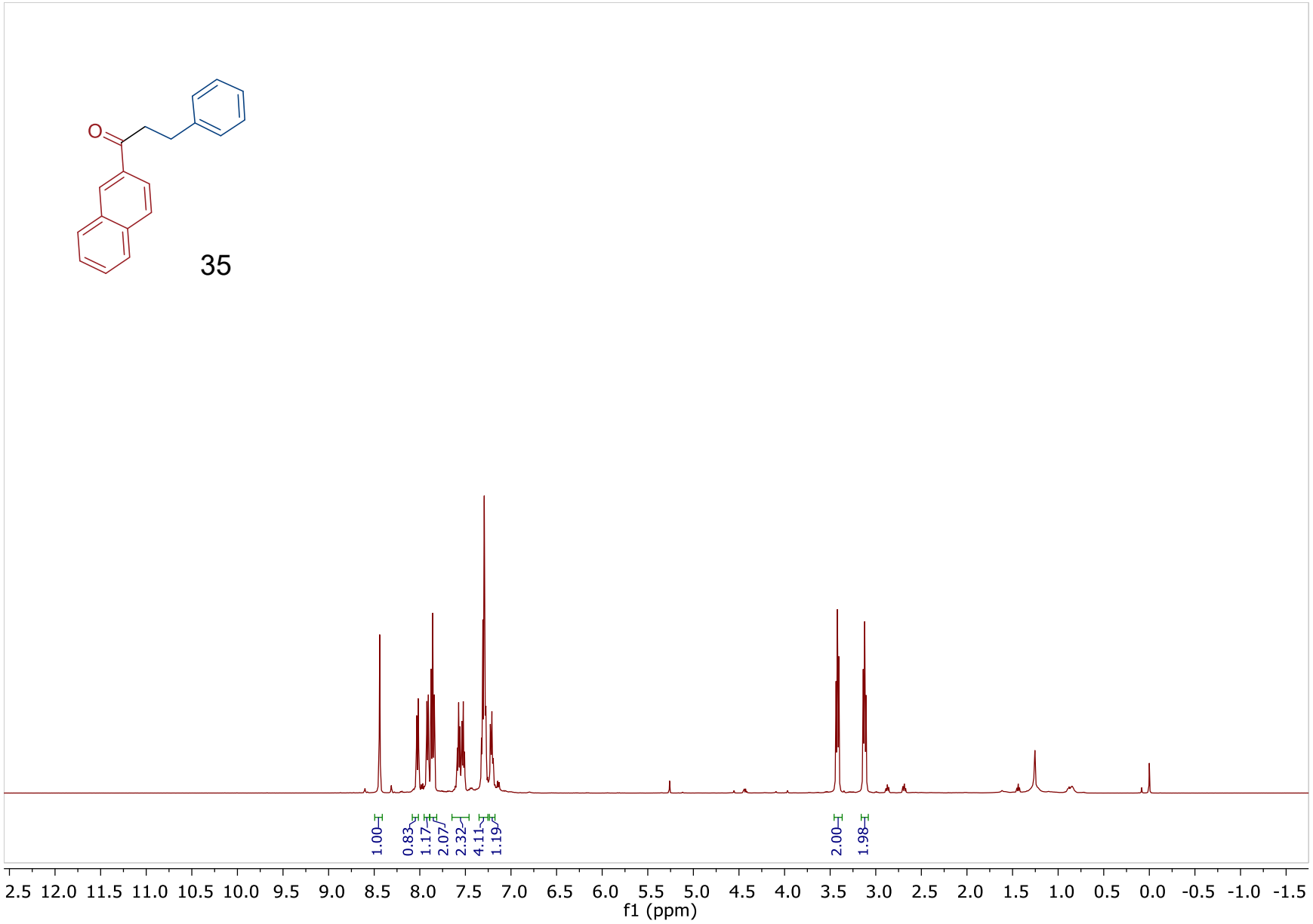

Me
Me
Me
Me
O
NH
N
O
O
O
F
O
O

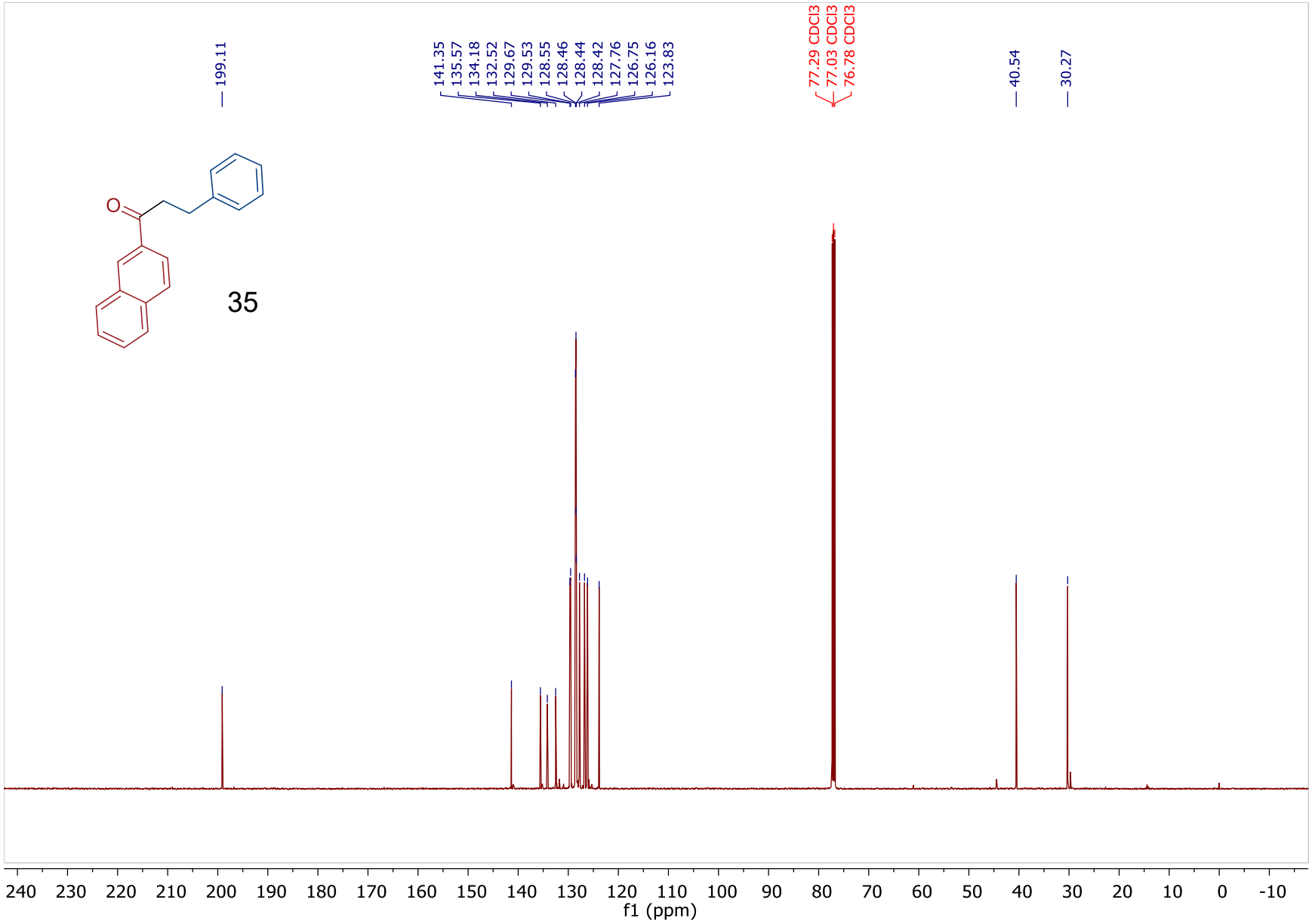
33

-113.71
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)


34
2.34
2.93
1.91
2.00
2.11
2.03
4.16
2.42
20.32
3.33
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


210.37
141.21
130.02
129.78
128.49
128.34
126.08
77.36 CDCl3
77.04 CDCl3
76.72 CDCl3
44.27
43.08
31.93
29.81
29.79
29.72
29.55
29.35
29.32
29.21
29.12
27.24
27.19
23.81
22.71
14.15
O
34
f1 (ppm)


O
35
1.00
0.83
1.17
2.07
2.32
4.11
1.19
2.00
1.98
2.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)


199.11
141.35
135.57
134.18
132.52
129.67
129.53
128.55
128.46
128.44
128.42
127.76
126.75
126.16
123.83
77.29 CDCl3
77.03 CDCl3
76.78 CDCl3
40.54
30.27
O
35
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)