Supporting Information

Direct Conversion of Carboxylic Acids to Alkyl Ketones

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Contents:

EXPERIMENTAL

General considerations: All reactions were carried out under an inert atmosphere of nitrogen or argon unless otherwise noted. THF was dried over activated alumina. $IrCl_3 \cdot xH_2O$ was purchased from commercial sources. $[Ni(dtbbpy)(H_2O)_4]Cl_2$ was synthesized according to the literature procedure.[1] LED irradiation was accomplished using the LED reactors described in our previous reports.[2] All other reagents were purchased commercially and used as received. Melting points (°C) are uncorrected. NMR spectra were recorded on a 500 MHz spectrometer. Data are presented as follows: chemical shift (ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, br = broad), coupling constant *J* (Hz) and integration. Analytical thin-layer chromatography (TLC) was performed on TLC silica gel plates (0.25 mm) precoated with a fluorescent indicator. TLC analysis was performed using hexanes/EtOAc as the eluant and visualized using permanganate stain, Seebach's stain, ninhydrin stain, and/or UV light. Standard flash chromatography procedures were followed using 100-200 mesh silica gel. HRMS data were obtained by either ESI or CI using a TOF mass spectrometer.

Synthesis of potassium secondary alkyltrifluoroborates:

Most of the potassium alkyltrifluoroborates were purchased commercially. In cases where the desired potassium alkyltrifluoroborate was not available, the corresponding boronic acid derivative was converted to the trifluoroborate by the following procedure.

General procedure for conversion of boronic acids to trifluoroborates. To a solution of the boronic acid derivative (2.0 mmol) in MeOH (0.1 M) at 0 ^{o}C, saturated aq KHF_2 (10.0 mmol, 5 equiv, 4.5 M) was added dropwise over 30 min. After completion of the reaction, as determined

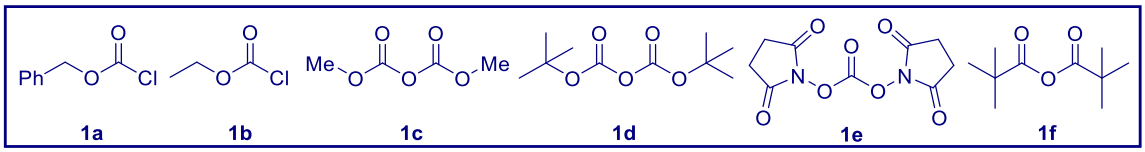
by ^{11}B NMR, the resulting suspension was concentrated under reduced pressure. H_2O was removed by lyophilization. The remaining solid was suspended in hot acetone (3 x 100 mL) and filtered. The filtrate was concentrated to a minimal volume (5 – 20 mL), and hexane or Et_2O (~200 mL) were added to yield a white precipitate. The precipitate was isolated by filtration, washing with hexanes (~30 mL) and CH_2Cl_2 (~30 mL), to afford the desired secondary alkyltrifluoroborate.

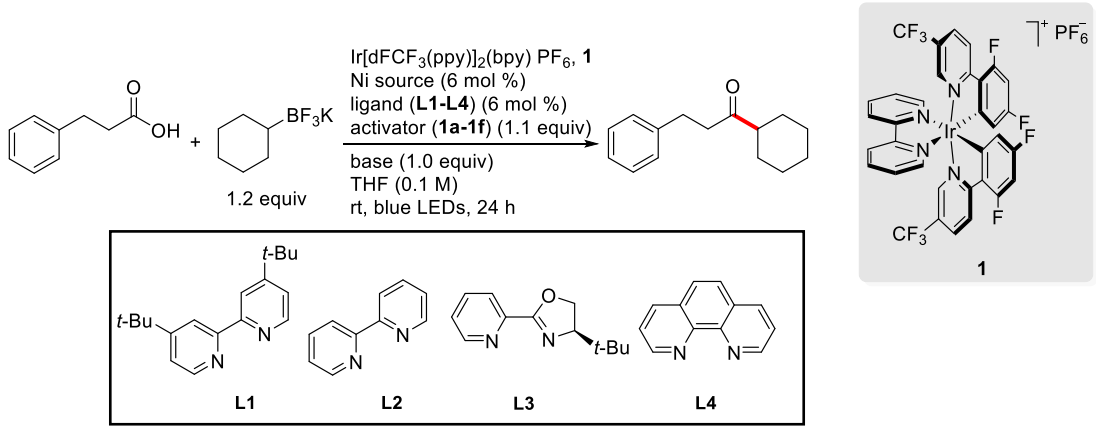
Synthesis of Ir[dF(CF$_3$)ppy]$_2$(bpy)PF$_6$ as the photocatalyst 1:

Photocatalyst **I** was synthesized according to the literature procedure.[3]

High-throughput experiments in design and optimization of the photoredox cross-coupling of hydrocinnamic acid with potassium cyclohexyltrifluoroborate. High Throughput Experimentation (HTE) was performed at the Penn/Merck Center for High Throughput Experimentation at the University of Pennsylvania. The screens were performed on a 10 μmol scale. To reaction vials equipped with a Teflon-coated magnetic stir bar in a glovebox was added a solution of Ni source and ligand [1:1] dissolved in THF. The solvent was removed in vacuo under an inert atmosphere. Then solutions of desired additives, potassium cyclohexyltrifluoroborate, hydrocinnamic acid, and photocatalyst **1** in a desired solvent, were added to each vial. The vials were sealed and stirred over blue LED lights. After 24 h the reactions were opened to air, 1 μmol of 4,4'-di-*tert*-butylbiphenyl (500 μL of a 0.002 μM solution in MeCN) was added to each vial as an internal standard, and the reaction mixtures were diluted with MeCN. The reaction mixtures were then analyzed by UPLC. The product-to-internal standard (P/IS) ratios from the UPLC are shown in Figures S1-S6.

To find the best conditions first, the photoredox cross-coupling of hydrocinnamic acid with potassium cyclohexyltrifluoroborate was investigated in the presence of a variety of activating reagents (**1a-1f**) for the in-situ activation of carboxylic acids, two different nickel sources, and four different ligands, either with or without base (Figure S1).





First Screen Variables:

solvents	ligands	bases	activator	Ni source
THF	**L1**	no base	**1a**	$Ni(COD)_2$
	L2	lutidine	**1b**	$NiCl_2$•dme
	L3		**1c**	
	L4		**1d**	
			1e	
			1f	

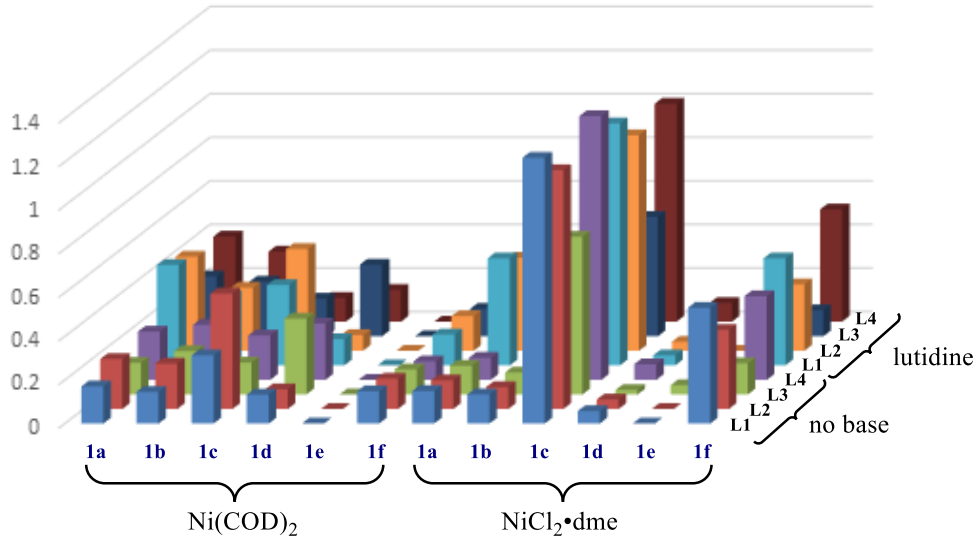
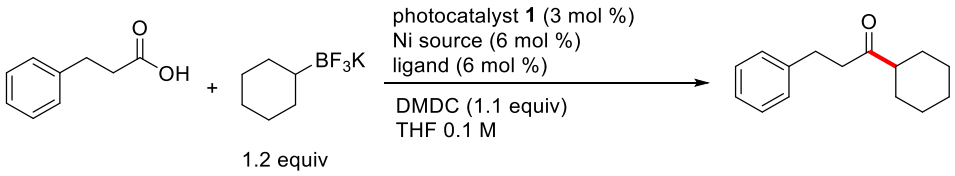


Figure S1. P/IS of the cross-coupling of hydrocinnamic acid with potassium cyclohexyltrifluoroborate using the first screen variables.

According to this screen, the best results were obtained for the reaction in the presence of DMDC, **1c**, as the activating reagent, $NiCl_2$·dme/**L1** or $NiCl_2$·dme/**L4** without any base.

Second Screens Variables:



solvents	ligands	bases	Ni source
THF	**L1**	no base	$NiCl_2$•dme
		pyridine	
	L4	Cs_2CO_3	
		$CsHCO_3$	
		K_2CO_3	
		$KHCO_3$	
		Na_2CO_3	
		$NaHCO_3$	
		Li_2CO_3	
		K_3PO_4	
		K_2HPO_4	
		NaH_2PO_4	

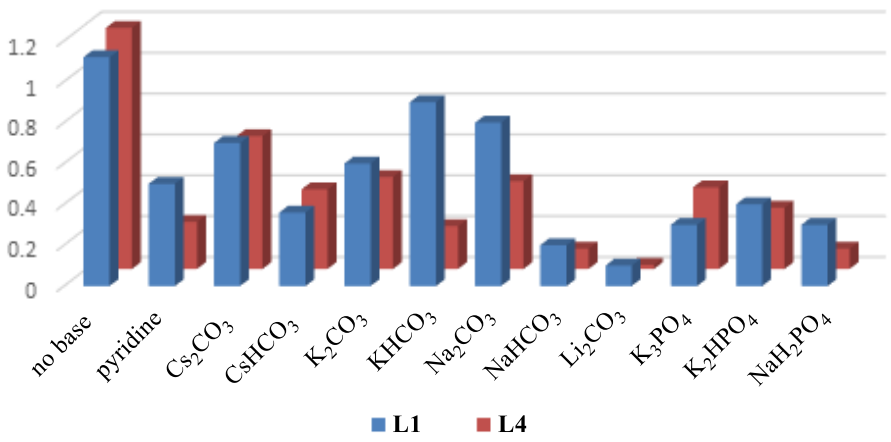
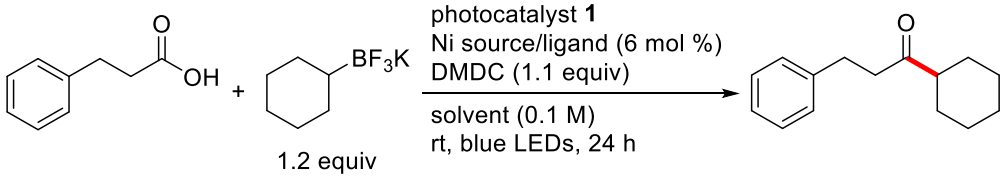


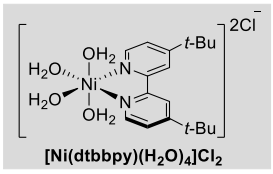
Figure S2. P/IS of the cross-coupling of hydrocinnamic acid with potassium cyclohexyltrifluoroborate using different bases.

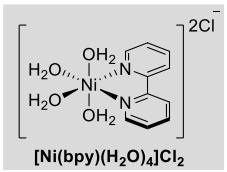
It was discovered that the identity of the base decreased the yield of the reaction.

Third Screen Variables:



Solvents	bases	Ni sources
THF	no base	$NiCl_2$•dme/**L1**
DME		$NiCl_2$•dme/**L4**
acetone		$NiBr_2$•dme/**L1**
CH_3CN		$NiBr_2$•dme/**L4**
DMF		$[Ni(dtbbpy)(H_2O)_4]Cl_2$
isopropyl acetate		$[Ni(bpy)(H_2O)_4]Cl_2$
2-propanol		
dioxane		





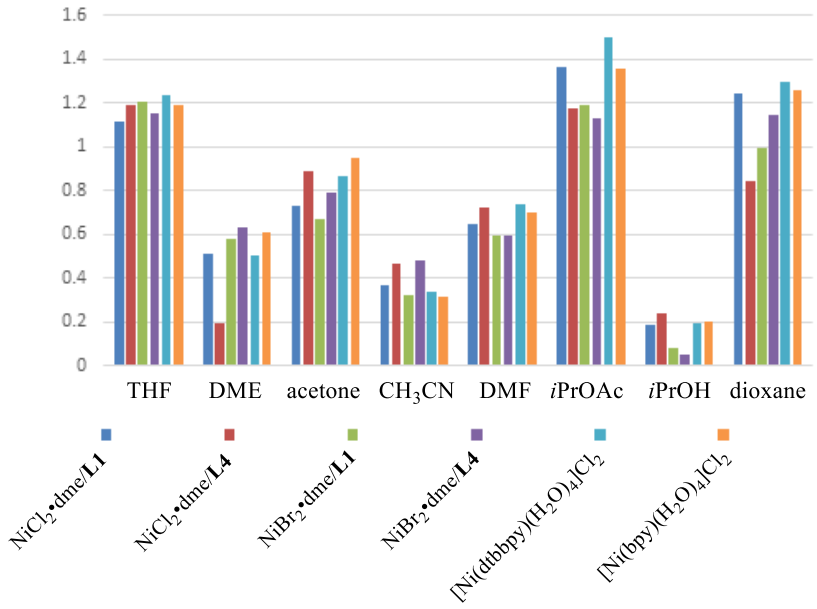


Figure S3. P/IS of the cross-coupling of hydrocinnamic acid with potassium cyclohexyltrifluoroborate using the third screen variables.

According to the third screen, the highest product to internal standard ratio was obtained for the reaction in the presence of $[Ni(dtbbpy)(H_2O)_4]Cl_2$ in isopropyl acetate as the solvent. To improve the yield of the reaction, further screens using a variety of co-solvents were carried out (Figure S4).

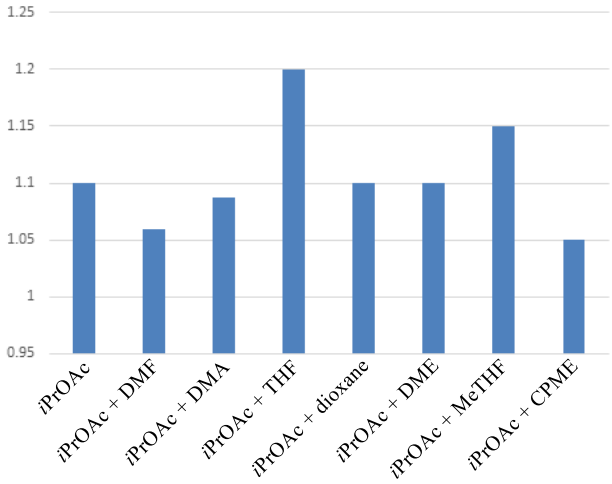


Figure S4. P/IS of the cross-coupling of hydrocinnamic acid with potassium cyclohexyltrifluoroborate in *i*-PrOAc using different co-solvents with a 4:1 ratio.

Consequently, the highest P/IS was obtained for the reaction in *i*-PrOAc/THF mixture. Further investigations to obtain the optimal ratio of the two solvents showed that the ratio of 2:1 (*i*-PrOAc/THF) generated the desired cross coupled product with the highest yield.

Our investigations disclosed that the presence of a base decreases the yield of the reaction (Figure S1 and S2). Therefore, we decided to explore the impact of some weak proton donors on this cross-coupling protocol (Figure S5).

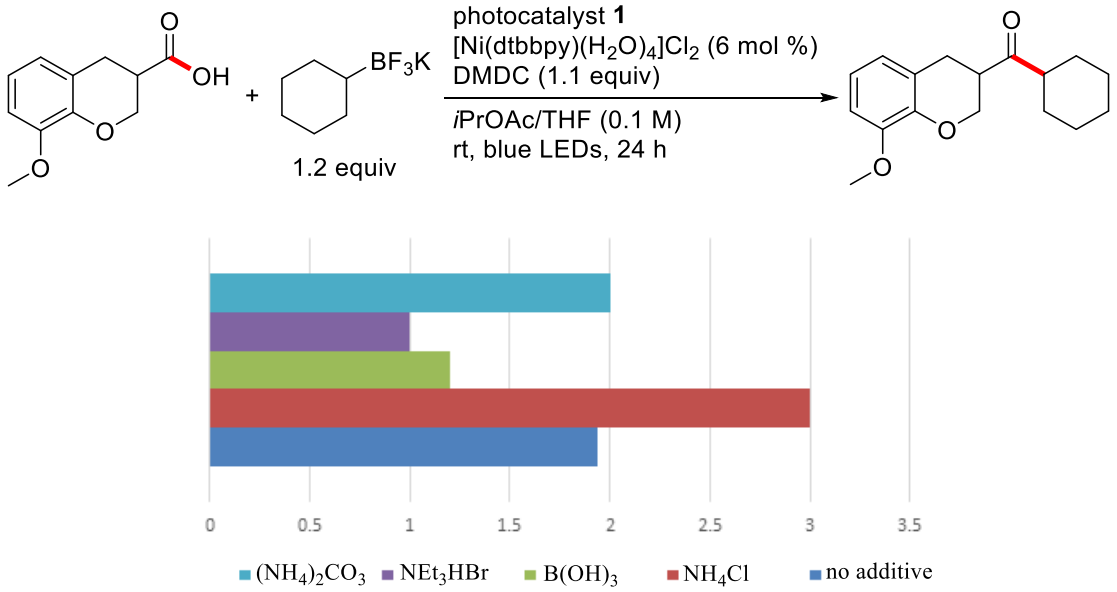


Figure S5. P/IS of the cross-coupling reaction in the presence of some weak proton donors.

It was found that NH_4Cl increased the yield of the reaction. Furthermore, to find the optimal amount of DMDC the cross-coupling reaction of hydrocinnamic acid with potassium cyclohexyltrifluoroborate was carried out using different amounts of the activator (Figure S6). The highest P/IS was obtained using 3.0 equiv of **1c**.

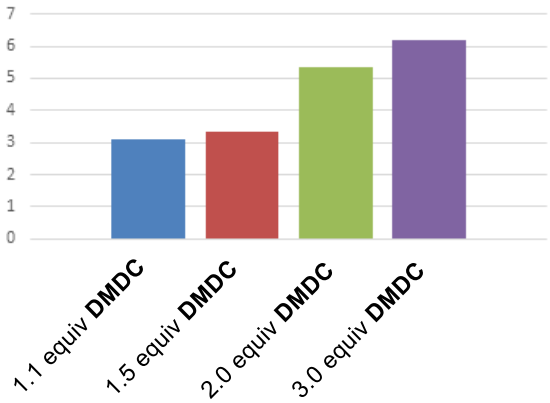
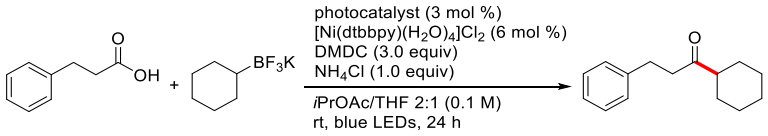


Figure S6. P/IS of the cross-coupling reaction using different amounts of the activator 1c.

Control experiments for the cross-coupling of carboxylic acids with potassium alkyltrifluoroborates:

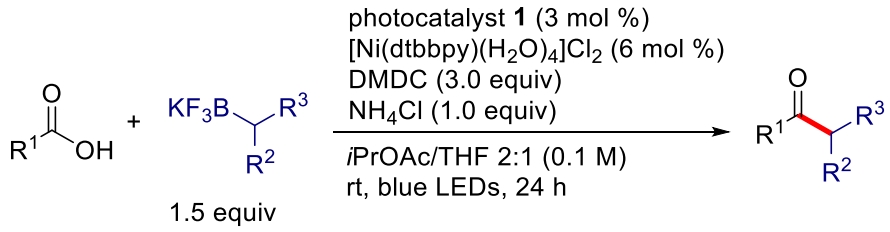
Table S1. Control Experiments.



entry	Conditions[a]	% Yield[c]
1	no Ir photocatalyst	<10
2	no $[Ni(dtbbpy)(H_2O)_4]Cl_2$	0
3	$[Ru(bpy)_3](PF_6)_3$	60
4	4CzIPN[b]	39
5	1.1 equiv of R-BF_3K	70
6	1.3 equiv of R-BF_3K	76
7	1.5 equiv of R-BF_3K	82

[a] Reactions were carried out at 0.1 M. [b] 2,4,5,6-Tetrakis(carbazol-9-yl)-1,3-dicyanobenzene. [c] Isolated yields.

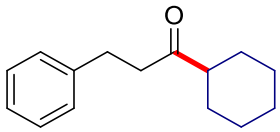
General procedure for the photoredox cross-coupling reaction of carboxylic acids with potassium alkyltrifluoroborates:



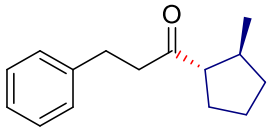
To a two dram (8 mL) borosilicate glass vial equipped with a Teflon-coated magnetic stir bar was added $[Ni(dtbbpy)(H_2O)_4]Cl_2$ (14.1 mg, 0.3 mmol), the corresponding carboxylic acid (0.5 mmol), $Ir[dFCF_3ppy]_2(bpy)PF_6$ **1** (15.1 mg, 0.015 mmol), potassium alkyltrifluoroborate (0.75 mmol, 1.5 equiv), and NH_4Cl (26.8 mg, 0.5 mmol). The vial was sealed and subsequently purged and evacuated with Ar four times. A mixture of degassed *i*-PrOAc and anhydrous THF (2/1, 5 mL) followed by DMDC (161 µL, 1.5 mmol) were then added by syringe under Ar. The resulting reaction mixture was stirred for 24 h in the presence of blue LEDs while a fan was blown across the reaction setup to maintain an ambient temperature of 24 ^{o}C. After completion, the crude reaction mixture was filtered through a plug of Celite and rinsed with EtOAc (20 mL). The resulting solution was concentrated, and the residue was purified by column chromatography on silica gel, with EtOAc/hexanes mixtures as the eluent, to obtain products in pure form.

Gram scale reaction: To a ~125 mL long, thin-walled vacuum flask equipped with a Teflon-coated magnetic stir bar was added $[Ni(dtbbpy)(H_2O)_4]Cl_2$ (98.7 mg, 0.21 mmol), potassium cyclohexyltrifluoroborate (1.99 g, 10.5 mmol, 1.5 equiv), $Ir[dFCF_3ppy]_2(bpy)PF_6$ **1** (105.9 mg, 0.105 mmol), hydrocinnamic acid (1.05 g, 7.0 mmol), and NH_4Cl (374.43 mg, 7.0 mmol). The vial was sealed and subsequently purged and evacuated with Ar four times. A mixture of degassed *i*-PrOAc and anhydrous THF (2:1, 70 mL) followed by DMDC (2.25 mL, 21.0 mmol) were added

by syringe under Ar. The resulting mixture was stirred vigorously for 48 h in the presence of blue LEDs while a fan was blown across the reaction setup to maintain an ambient temperature of 24 ^{o}C. After completion, the crude reaction mixture was filtered through a plug of Celite and rinsed with EtOAc (50 mL). The resulting solution was concentrated and the residue was purified by column chromatography on silica gel, with EtOAc/hexanes mixtures as the eluent, to obtain the product in pure form.

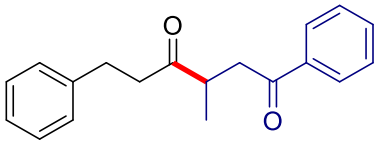


1-Cyclohexyl-3-phenylpropan-1-one (2a).[4] The title compound was obtained as a liquid in 82% yield (0.5 mmol scale, 88.6 mg) and in 70% yield on gram scale (4.5 mmol scale, 700.9 mg). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.26-7.23 (m, 2H), 7.16-7.14 (m, 3H), 2.86 (t, J = 7.5 Hz, 2H), 2.74 (t, J = 7.5 Hz, 2H), 2.31-2.25 (m, 1H), 1.79-1.73 (m, 4H), 1.64-1.62 (m, 1H), 1.33-1.22 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 213.2, 141.6, 128.6, 128.5, 126.2, 51.2, 42.4, 30.0, 28.6, 26.1, 25.9

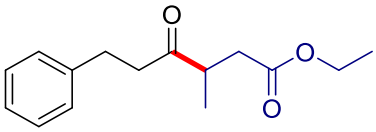


1-(2-Methylcyclopentyl)-3-phenylpropan-1-one (2b).[6] The title compound was obtained as a liquid in 69% yield (0.5 mmol scale, 74.6 mg). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.28-7.25 (m, 2H), 7.18-7.15 (m, 3H), 2.89 (t, J = 7.5 Hz, 2H), 2.82-2.72 (m, 2H), 2.40-2.36 (m, 1H), 2.16-2.07 (m, 1H), 1.86-1.81 (m, 2H), 1.72-1.61 (m, 3H), 1.22-1.15 (m, 1H), 1.00 (d, J = 6.5 Hz, 3H); ^{13}C NMR

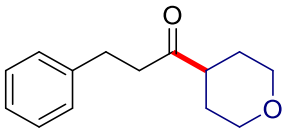
(125.8 MHz, $CDCl_3$): δ 212.6, 141.6, 128.6, 128.5, 126.2, 59.9, 44.1, 37.9, 35.1, 30.0, 29.9, 24.9, 20.2.



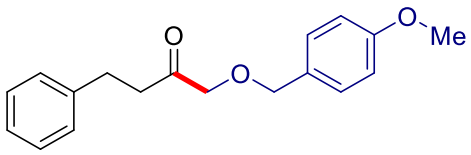
3-Methyl-1,6-diphenylhexane-1,4-dione (2c).[6] The title compound was obtained as a liquid in 63% yield (0.5 mmol scale, 88.3 mg). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.93-7.91 (m, 2H), 7.54.7.51 (m, 1H), 7.43-7.40 (m 2H), 7.27-7.24 (m, 2H), 7.22-7.15 (m, 3H), 3.54-3.48 (m, 1H), 3.22-3.15 (m, 1H), 2.98-2.88 (m, 5H), 1.11 (dd, J = 7.0, 0.5 Hz, 3H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 212.7, 198.7, 141.6, 136.8, 133.4, 128.8, 128.7, 128.6, 128.2, 126.2, 43.3, 42.1, 41.4, 29.9, 16.9.



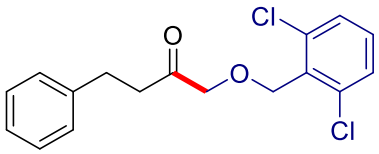
Ethyl 3-Methyl-4-oxo-6-phenylhexanoate (2d).[7] The title compound was obtained as a liquid in 60% yield (0.5 mmol scale, 74.5 mg). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.28-7.25 (m, 2H), 7.19-7.16 (m, 3H), 4.09 (q, *J* = 7.0 Hz, 2H), 3.01-2.95 (m, 1H), 2.93-2.87 (m, 2H), 2.87-2.81 (m, 2H), 2.79-2.74 (m, 1H), 2.31-2.26 (m, 1H), 1.23 (t, *J* = 7.0 Hz, 3H), 1.08 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 212.0, 172.5, 141.4, 128.7, 128.5, 126.2, 60.7, 43.0, 42.3, 37.2, 29.8, 16.7, 14.4.



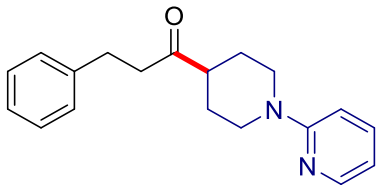
3-Phenyl-1-(tetrahydro-2H-pyran-4-yl)propan-1-one (2e).[6] The title compound was obtained as a liquid in 75% yield (0.5 mmol scale, 81.8 mg). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.26-7.23 (m, 2H), 7.17-7.14 (m, 3H), 3.95-3.92 (m, 2H), 3.35 (dt, *J* = 11.5, 3.0 Hz, 2H), 2.87 (t, *J* = 7.5 Hz, 2H), 2.74 (t, *J* = 7.5 Hz, 2H), 2.49-2.44 (m, 1H), 1.69-1.58 (m, 4H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 211.1, 141.3, 128.7, 128.5, 126.3, 67.4, 47.8, 42.1, 29.8, 28.2.



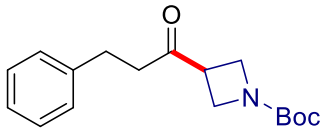
1-((4-Methoxybenzyl)oxy)-4-phenylbutan-2-one (2f). The title compound was obtained as a liquid in 90% yield (0.5 mmol scale, 127.9 mg). ^{1}H NMR (500 MHz, CD_3CN): δ 7.25-7.21 (m, 4H), 7.17-7.13 (m, 3H), 6.85-6.84 (m, 2H), 4.44 (s, 2H), 3.95 (s, 2H), 3.75 (s, 3H), 2.87 (t, *J* = 7.5 Hz, 2H), 2.73 (t, *J* = 7.5 Hz, 2H); ^{13}C NMR (125.8 MHz, CD_3CN): δ 208.2, 159.6, 141.0, 129.7, 129.3, 128.6, 128.5, 126.3, 114.0, 74.6, 73.1, 55.3, 40.6, 29.4; FT-IR (neat): 1719, 1611, 1512, 1246, 1030, 699 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{18}H_{21}O_3$ $[M+H]^+$ 285.1491, found 285.1514.



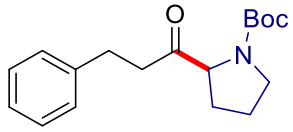
1-(2,6-Dichlorobenzyloxy)-4-phenylbutan-2-one (2g).[8] The title compound was obtained as a white solid in 86% yield (0.5 mmol scale, 138.9 mg). mp 62-64 °C; ^{1}H NMR (500 MHz, $CDCl_3$): 7.30-7.22 (m, 4H), 7.18-7.14 (m, 4H), 4.82 (s, 2H), 4.06 (s, 2H), 2.90-2.87 (m, 2H), 2.82-2.78 (m, 2H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 208.1, 141.0, 137.1, 132.7, 130.4, 128.6, 128.5, 126.2, 75.7, 67.7, 40.6, 29.3.



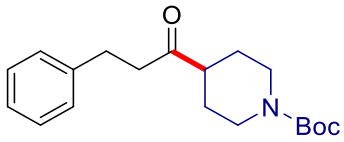
3-Phenyl-1-(1-(pyridin-2-yl)piperidin-4-yl)propan-1-one (2h). The title compound was obtained as a liquid in 80% yield (0.5 mmol scale, 117.7 mg). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.17-8.16 (m, 1H), 7.45-7.41 (m, 1H), 7.29-7.25 (m, 2H), 7.20-7.17 (m, 3H), 6.63 (d, *J* = 8.5 Hz, 1H), 6.59-6.56 (m, 1H), 4.28-4.25 (m, 2H), 2.92-2.83 (m, 4H), 2.79 (t, *J* = 7.5 Hz, 2H), 2.53-2.47 (m, 1H), 1.88-1.85 (m, 2H), 1.67-1.59 (m, 2H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 211.5, 159.4, 148.1, 141.3, 137.6, 128.6, 128.5, 126.3, 113.1, 107.4, 49.2, 45.2, 42.3, 29.8, 27.2; FT-IR (neat): 1734, 1705, 1593, 1480, 1436, 1311, 1240, 976, 731 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{19}H_{23}N_2O$ $[M+H]^+$ 295.1810, found 295.1812.



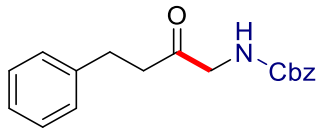
***tert*-Butyl 3-(3-Phenylpropanoyl)azetidine-1-carboxylate (2i).**[6] The title compound was obtained as a liquid in 65% yield (0.5 mmol scale, 94.0 mg). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.26-7.23 (m, 2H), 7.18-7.13 (m, 3H), 3.96-3.93 (m, 4H), 3.36-3.30 (m, 1H), 2.89 (t, *J* = 7.0 Hz, 2H), 2.71 (t, *J* = 7.0 Hz, 2H), 1.39 (s, 9H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 207.3, 156.4, 140.8, 128.8, 128.5, 126.5, 80.0, 50.6, 42.5, 38.9, 29.7, 28.5.



***tert*-Butyl 2-(3-Phenylpropanoyl)pyrrolidine-1-carboxylate (2j).** The title compound was obtained as a liquid in 80% yield (0.5 mmol scale, 121.3 mg). ^{1}H NMR (500 MHz, CD_3CN): δ 7.32-7.29 (m, 2H), 7.26-7.25 (m, 2H), 7.22-7.19 (m, 1H), 4.32 (dd, J = 8.5, 5.0 Hz, 1H), 3.51-3.40 (m, 2H), 2.95-2.79 (m, 4H), 2.18-2.11 (m, 1H), 1.86-1.75 (m, 3H), 1.45 (s, 9H); ^{13}C NMR (125.8 MHz, CD_3CN): δ 210.5, 156.1, 143.7, 130.2, 127.7, 81.2, 67.2, 48.7, 41.9, 31.1, 30.8, 29.7, 25.5; FT-IR (neat): 1726, 1690, 1390, 1365, 1160, 749, 699 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{18}H_{25}NO_3Na$ $[M+Na]^+$ 326.1732, found 326.1711.

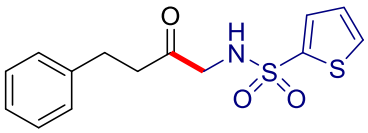


***tert*-Butyl 4-(3-Phenylpropanoyl)piperidine-1-carboxylate (2k).** The title compound was obtained as a liquid in 89% yield (0.5 mmol scale, 141.2 mg). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.24-7.21 (m, 2H), 7.16-7.12 (m, 3H), 4.04-4.01 (m, 2H), 2.87 (t, J = 7.5 Hz, 2H), 2.76-2.71 (m, 4H), 2.42-2.36 (m, 1H), 1.73-1.70 (m, 2H), 1.51-1.40 (m, 11H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 210, 154.8, 141.3, 128.6, 128.4, 126.3, 79.6, 48.9, 43.4, 42.2, 29.9, 28.6, 27.6; FT-IR (neat): 1687, 1448, 1419, 1365, 1234, 1163, 698 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{19}H_{27}NO_3Na$ $[M+Na]^+$ 340.1889, found 340.1880.

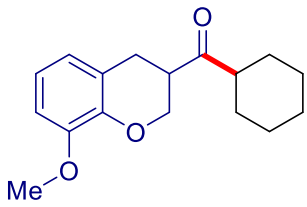


Benzyl (2-Oxo-4-phenylbutyl)carbamate (2l). The title compound was obtained as a liquid in 83% yield (0.5 mmol scale, 123.4 mg). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36-7.27 (m, 7H), 7.22-7.16 (m, 3H), 5.53 (s, 1H), 5.13 (s, 2H), 4.00 (d, J = 5.0 Hz, 2H), 2.94 (t, J = 7.5 Hz, 2H), 2.73 (t,

J = 7.5 Hz, 2H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 204.6, 156.3, 140.6, 136.7, 128.8, 128.7, 128.4, 128.3, 128.2, 126.5, 67.2, 51.0, 41.6, 29.8; FT-IR (neat): 1728, 1713, 1697, 1538, 1267, 1160, 1041, 701 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{18}H_{20}NO_3$ $[M+H]^+$ 298.1443, found 298.1432.

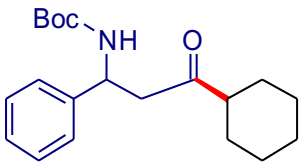


***N*-(2-Oxo-4-phenylbutyl)thiophene-2-sulfonamide (2m).** The title compound was obtained as a liquid in 77% yield (0.5 mmol scale, 119.1 mg). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.57-7.56 (m, 2H), 7.27-7.24 (m, 2H), 7.20-7.18 (m, 1H), 7.11-7.09 (m, 2H), 7.07-7.05 (m, 1H), 5.44 (s, 1H), 3.86 (d, J = 4.5 Hz, 2H), 2.87 (t, J = 7.5 Hz, 2H), 2.71 (t, J = 7.5 Hz, 2H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 202.9, 140.2, 140.1, 132.7, 132.5, 128.9, 128.4, 127.8, 126.7, 52.1, 41.8, 29.7; FT-IR (neat): 1719, 1155, 1149, 1031, 738, 588 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{14}H_{16}NO_3S_2$ $[M+H]^+$ 310.0572, found 310.0569.

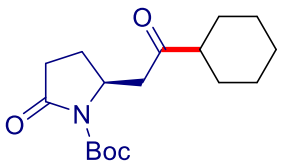


Cyclohexyl(8-methoxychroman-3-yl)methanone (3a). The title compound was obtained as a white solid in 70% yield (0.5 mmol scale, 96.0 mg). mp 39-40 °C; ^{1}H NMR (500 MHz, $CDCl_3$): 6.81-6.78 (m, 1H), 6.72-6.66 (m, 2H), 4.48-4.45 (m, 1H), 3.98 (m, 1H), 3.84 (s, 3H), 3.25-3.19 (m, 1H), 2.99 (dd, J = 16.0, 11.0 Hz, 1H), 2.84-2.79 (m, 1H), 2.58-2.52 (m, 1H), 1.87-1.77 (m, 4H), 1.68-1.66 (m, 1H), 1.41-1.18 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 213.3, 148.5, 143.6, 121.9, 121.7, 120.4, 109.5, 67.4, 56.0, 50.3, 43.3, 28.6, 28.4, 28.2, 26.0, 25.8, 25.7; FT-IR (neat):

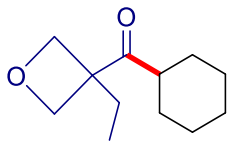
1700, 1485, 1449, 1210, 1084, 726 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{17}H_{23}O_3$ $[M+H]^+$ 275.1647, found 275.1656.



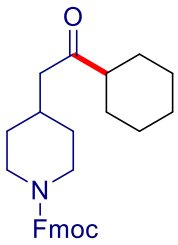
***tert*-Butyl (3-Cyclohexyl-3-oxo-1-phenylpropyl)carbamate (3b).** The tile compound was obtained as a liquid in 85% yield (0.5 mmol scale, 140.8 mg). ^{1}H NMR (500 MHz, $CDCl_3$): 7.30-7.25 (m, 4H), 7.22-7.19 (m, 1H), 5.46 (s, 1H), 5.05 (dd, *J* = 14.0, 6.0 Hz, 1H), 3.03 (dd, *J* = 16.5, 6.0 Hz, 1H), 2.88 (dd, *J* = 16.5, 6.0 Hz, 1H), 2.22-2.19 (m, 1H), 1.72-1.60 (m, 5H), 1.40 (s, 9H), 1.25-1.22 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 211.9, 155.4, 142.4, 128.7, 127.4, 126.5, 79.7, 51.8, 51.5, 46.5, 28.6, 28.3, 26.1, 25.9, 25.8; FT-IR (neat): 3352, 1696, 1687, 1531, 1171, 698 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{20}H_{29}NO_3Na$ $[M+Na]^+$ 354.2045, found 354.2054.



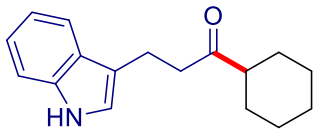
5-(2-Cyclohexyl-2-oxoethyl)pyrrolidin-2-one (3c). The title compound was obtained as a liquid in 81% yield (0.5 mmol scale, 125.3 mg). ^{1}H NMR (500 MHz, $CDCl_3$): 4.45-4.41 (m, 1H), 2.94 (dd, *J* = 17.0, 2.5 Hz, 1H), 2.59 (dd, *J* = 17.0, 10.0 Hz, 1H), 2.50-2.43 (m, 1H), 2.36-2.30 (m, 1H), 2.26-2.21 (m, 1H), 2.18-2.10 (m, 1H), 1.75-1.68 (m, 4H), 1.63-1.57 (m, 2H), 1.42 (s, 9H), 1.29-1.09 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 211.1, 173.8, 150.0, 83.0, 54.2, 51.2, 44.1, 31.1, 28.5, 28.4, 28.2, 25.9, 25.7, 25.6, 23.6; FT-IR (neat): 1784, 1749, 1704, 1368, 1307, 1148, 730 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{17}H_{27}NO_4Na$ $[M+Na]^+$ 332.1838, found 332.1840.



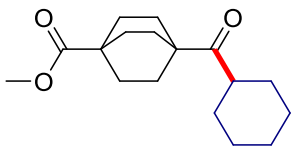
Cyclohexyl(3-ethyloxetan-3-yl)methanone (3d). The title compound was obtained as a liquid in 60% yield (0.5 mmol scale, 58.8 mg). ^{1}H NMR (500 MHz, $CDCl_3$): 4.83 (d, J = 6.0 Hz, 2H), 4.38 (d, J = 6.0 Hz, 2H), 2.54-2.48 (m, 1H), 2.02 (q, J = 7.5 Hz, 2H), 1.77-1.74 (m, 2H), 1.68-1.66 (m, 3H), 1.44-1.37 (m, 2H), 1.25-1.19 (m, 3H), 0.80 (t, J = 7.5 Hz, 3H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 213.6, 77.3, 55.6, 46.6, 29.5, 28.5, 25.9, 25.8, 8.9; FT-IR (neat): 2929, 2856, 1699, 1449, 981, cm^{-1}; HRMS (CI+) m/z calcd. for $C_{12}H_{21}O_2$ $[M+H]^+$ 197.1542, found 197.1547.



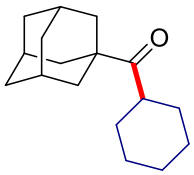
(9H-Fluoren-9-yl)methyl 4-(2-cyclohexyl-2-oxoethyl)piperidine-1-carboxylate (3e). The title compound was obtained as a yellow solid in 75% yield (0.5 mmol scale, 161.8 mg). mp 85-86 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$): 7.71 (d, J = 7.5 Hz, 2H), 7.54 (d, J = 7.5 Hz, 2H), 7.34 (t, J = 7.5 Hz, 2H), 7.26 (t, J = 7.5 Hz, 2H), 4.41 (d, J = 6.5 Hz, 2H), 4.19 (t, J = 6.5 Hz, 1H), 4.03-4.01 (m, 2H), 2.75 (t, J = 12.5 Hz, 2H), 2.29-2.22 (m, 3H), 2.00-1.96 (m, 1H), 1.79-1.74 (m, 4H), 1.65-1.58 (m, 3H), 1.35-1.17 (m, 5H), 1.05-0.99 (m, 2H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 212.4, 155.3, 144.4, 141.6, 127.7, 127.1, 125.1, 120.0, 67.2, 51.5, 47.8, 47.0, 44.3, 32.1, 31.7, 28.5, 26.0, 25.8; FT-IR (neat): 1692, 1441, 1277, 1236, 1089, 760 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{28}H_{34}NO_3$ $[M+H]^+$ 432.2539, found 432.2537.



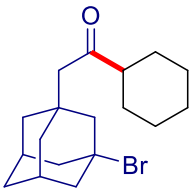
1-Cyclohexyl-3-(1H-indol-3-yl)propan-1-one (3f). The title compound was obtained as a pale yellow solid in 35% yield (0.5 mmol scale, 44.6 mg). mp 95-96 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$): 7.99 (s,1H), 7.59 (d, J = 8.0 Hz, 1H), 7.35 (d, J = 8.0, 0.5 Hz, 1H), 7.21-7.18 (m, 1H), 7.14-7.11 (m, 1H), 6.97 (d, J = 1.5 Hz, 1H), 3.04 (t, J = 7.5 Hz, 2H), 2.86-2.83 (m, 2H), 2.36-2.30 (m, 1H), 1.83-1.75 (m, 4H), 1.66-1.63 (m, 1H), 1.37-1.17 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 214.2, 136.6, 127.5, 122.2, 121.7, 119.5, 119.0, 115.8, 111.4, 51.2, 41.4, 28.7, 26.1, 25.9, 19.5; FT-IR (neat): 3405, 1694, 1338, 745 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{17}H_{22}NO$ $[M+H]^+$ 256.1701, found 256.1725.



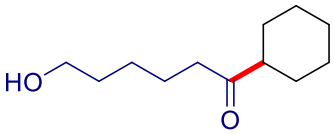
Methyl 4-(Cyclohexanecarbonyl)bicyclo[2.2.2]octane-1-carboxylate (3g). The title compound was obtained as a white solid in 78% yield (0.5 mmol scale, 108.5 mg). mp 104-105 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.61 (s, 3H), 2.76-2.72 (m, 1H), 1.79-1.73 (m, 6H), 1.72-1.69 (m, 8H), 1.63-1.62 (m, 1H), 1.55-1.52 (m, 2H), 1.31-1.18 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 217.9, 178.0, 51.9, 51.9, 45.4, 45.2, 39.1, 29.7, 27.9, 26.8, 25.9; FT-IR (neat): 1724, 1686, 1449, 1253, 1236, 1079, 1010, 854 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{17}H_{27}O_3$ $[M+H]^+$ 279.1960, found 279.1966.



(Adamantan-1-yl)(cyclohexyl)methanone (3h).[6] The title compound was obtained as a white solid in 70% yield (0.5 mmol scale, 86.2 mg). mp 80-81 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.85-2.80 (m, 1H), 2.02 (s, 3H), 1.79-1.65 (m, 15H), 1.56-1.53 (m, 2H), 1.35-1.21 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 218.5, 47.1, 44.2, 38.0, 36.9, 30.0, 28.2, 26.0.

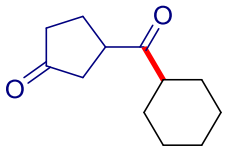


2-(3-Bromoadamantan-1-yl)-1-cyclohexylethan-1-one (3i). The title compound was obtained as a liquid in 88% yield (0.5 mmol scale, 149.3 mg). ^{1}H NMR (500 MHz, $CDCl_3$): 2.27-2.20 (m, 9H), 2.09 (m, 2H), 1.76-1.72 (m, 4H), 1.63-1.57 (m, 7H), 1.24-1.17 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 213.0, 65.7, 53.8, 52.7, 52.5, 48.6, 40.4, 38.1, 34.9, 32.6, 28.3, 26.0, 25.8; FT-IR (neat): 2925, 2852, 1704, 1448, 677 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{18}H_{28}OBr$ $[M+H]^+$ 339.1324, found 339.1334.

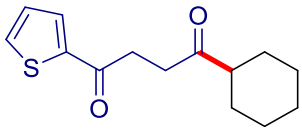


1-Cyclohexyl-6-hydroxyhexan-1-one (3j). The title compound was obtained as a liquid in 86% yield (0.5 mmol scale, 85.2 mg). ^{1}H NMR (500 MHz, $CDCl_3$): 3.54 (t, J = 6.5 Hz, 2H), 2.46 (s, 1H), 2.37 (t, J = 7.0 Hz, 2H), 2.28-2.23 (m, 1H), 1.75-1.68 (m, 4H), 1.60-1.58 (m, 1H), 1.53-1.47

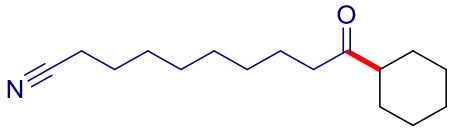
(m, 4H), 1.29-1.10 (m, 7H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 214.7, 62.5, 51.0, 40.6, 32.6, 28.6, 26.0, 25.8, 25.6, 23.5; FT-IR (neat): 3405, 2927, 2854, 1702, 1053, cm^{-1}; HRMS (ES+) m/z calcd. for $C_{12}H_{23}O_2$ $[M+H]^+$ 199.1698, found 199.1700.



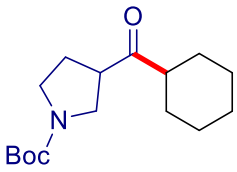
3-(Cyclohexanecarbonyl)cyclopentan-1-one (3k). The title compound was obtained as a liquid in 65% yield (0.5 mmol scale, 63.1 mg). ^{1}H NMR (500 MHz, $CDCl_3$): 3.39-3.32 (m, 1H), 2.48-2.39 (m, 2H), 2.34-2.12 (m, 4H), 1.97-1.91 (m, 1H), 1.83-1.75 (m, 4H), 1.66-1.64 (m, 1H), 1.41-1.15 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 217.0, 214.1, 50.2, 46.0, 40.8, 37.6, 28.8, 28.4, 26.5, 26.0, 25.9, 25.6; FT-IR (neat): 1741, 1700, 1449, 480 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{12}H_{19}O_2$ $[M+H]^+$ 195.1385, found 195.1358.



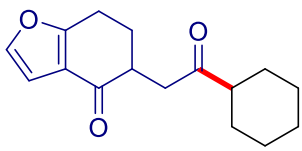
1-Cyclohexyl-4-(thiophen-2-yl)butane-1,4-dione (3l). The title compound was obtained as a yellow solid in 37% yield (0.5 mmol scale, 46.3 mg). mp: 47-49 ^{o}C; ^{1}H NMR (500 MHz, $CDCl_3$): 7.74 (d, J = 3.5 Hz, 1H), 7.59 (d, J = 5.0 Hz, 1H), 7.11-7.09 (m, 1H), 3.18 (t, J = 6.5 Hz, 2H), 2.86 (d, J = 6.5 Hz, 2H), 2.44-2.39 (m, 1H), 1.90-1.88 (m, 2H), 1.79-1.76 (m, 2H), 1.67-1.65 (m, 1H), 1.42-1.19 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 212.4, 191.9, 144.2, 133.6, 132.1, 128.3, 51.2, 34.5, 33.3, 28.9, 26.2, 26.0; FT-IR (neat): 1736, 1706, 1664, 1415, 1246, 849, 735 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{14}H_{19}O_2S$ $[M+H]^+$ 251.1106, found 251.1098.



10-Cyclohexyl-10-oxodecanenitrile (3m). The title compound was obtained as a liquid in 70% yield (0.5 mmol scale, 87.2 mg). ^{1}H NMR (500 MHz, $CDCl_3$): 2.36 (t, J = 7.0 Hz, 2H), 2.29-2.26 (m, 3H), 1.76-1.70 (m, 4H), 1.61-1.55 (m, 3H), 1.51-1.46 (m, 2H), 1.39-1.36 (m, 2H), 1.29-1.11 (m, 11H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 214.3, 119.9, 50.9, 40.6, 29.2, 28.7, 28.6, 26.0, 25.8, 25.5, 23.7, 17.2; FT-IR (neat): 2927, 2854, 2245, 1704, 1449, 1373 cm^{-1}; HRMS (CI+) m/z calcd. for $C_{16}H_{28}NO$ $[M+H]^+$ 250.2171, found 250.2175.

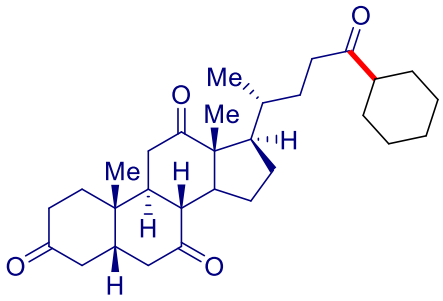


***tert*-Butyl 3-(Cyclohexanecarbonyl)pyrrolidine-1-carboxylate (3n).** The title compound was obtained as a liquid in 70% yield (0.5 mmol scale, 87.2 mg). ^{1}H NMR (500 MHz, $CDCl_3$): 3.48-3.32 (m, 3H), 3.27-3.17 (m, 2H), 2.41-2.35 (m, 1H), 1.96 (s, 2H), 1.76-1.70 (m, 4H), 1.62-1.60 (m, 1H), 1.38 (s, 9H), 1.32-1.14 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 212.5, 154.5, 79.5, 50.4, 48.0, 45.7, 28.7, 28.6, 26.0, 25.9, 25.8; FT-IR (neat): 1691, 1401, 1365, 1166, 1115, 731 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{16}H_{27}NO_3Na$ $[M+Na]^+$ 304.1889, found 304.1899.

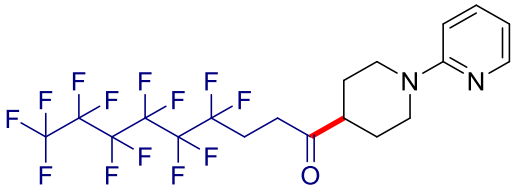


5-(2-Cyclohexyl-2-oxoethyl)-6,7-dihydrobenzofuran-4(5H)-one (3o). The title compound was obtained as a liquid in 68% yield (0.5 mmol scale, 88.5 mg). ^{1}H NMR (500 MHz, $CDCl_3$): 7.29

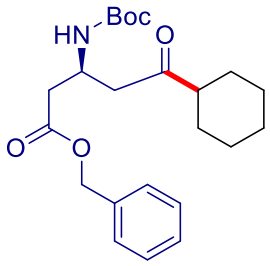
(d, J = 2.0 Hz, 1H), 6.62 (d, J = 2.0 Hz, 1H), 3.14 (dd, J = 17.5, 5.0 Hz, 1H), 3.04-2.85 (m, 3H), 2.46-2.38 (m, 2H), 2.22-2.17 (m, 1H), 1.95-1.86 (m, 3H), 1.80-1.76 (m, 2H), 1.67-1.64 (m, 1H), 1.43-1.18 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 212.5, 195.0, 166.6, 143.0, 120.8, 106.9, 51.3, 42.8, 40.4, 29.1, 28.8, 28.6, 26.1, 25.9, 25.8, 23.6; FT-IR (neat): 1736, 1704, 1673, 1443, 1118, 733 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{16}H_{21}O_3$ $[M+H]^+$ 261.1491, found 261.1491.



(5*S*,8*R*,9*S*,10*S*,13*R*,14*S*,17*R*)-17-((*R*)-5-Cyclohexyl-5-oxopentan-2-yl)-10,13-dimethyldodecahydro-3H-cyclopenta[a]phenanthrene-3,7,12(2H,4H)-trione (3p). The title compound was obtained as a liquid in 60% yield (0.5 mmol scale, 140.6 mg). ^{1}H NMR (500 MHz, $CDCl_3$): 2.91-2.79 (m, 3H), 2.50-2.43 (m, 1H), 2.40-2.17 (m, 8H), 2.12-2.07 (m, 2H), 2.03-1.91 (m, 4H), 1.85-1.73 (m, 6H), 1.67-1.55 (m, 2H), 1.37-1.17 (m, 12H), 1.04 (s, 3H), 0.80 (d, J = 6.0 Hz, 3H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 214.8, 212.3, 209.2, 208.9, 57.2, 52.0, 51.2, 49.3, 47.1, 45.9, 45.8, 45.2, 43.0, 38.9, 37.6, 36.7, 36.3, 35.6, 35.5, 29.3, 28.9, 28.8, 27.8; FT-IR (neat): 1704, 1448, 1385, 1123, 620 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{30}H_{45}O_4$ $[M+H]^+$ 469.3318, found 469.3316.

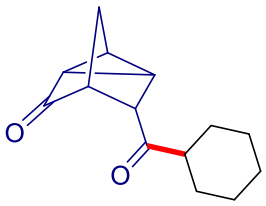


4,4,5,5,6,6,7,7,8,8,9,9,9-Tridecafluoro-1-(1-(pyridin-2-yl)piperidin-4-yl)nonan-1-one (3q). The title compound was obtained as a white solid in 68% yield (0.5 mmol scale, 182.3 mg). mp: 68-70 °C; ^{1}H NMR (500 MHz, $CDCl_3$): 8.15 (dd, J = 5.0, 1.5 Hz, 1H), 7.45-7.41 (m, 1H), 6.64 (d, J = 9.0 Hz, 1H), 6.57 (dd, J = 35.5, 5.0 Hz, 1H), 4.30 (dd, J = 10.5, 3.0 Hz, 2H), 2.91-2.85 (m, 2H), 2.79-2.76 (m, 2H), 2.62-2.56 (m, 1H), 2.46-2.35 (m, 2H), 1.93-1.91 (m, 2H), 1.72-1.63 (m, 2H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 208.8, 159.5, 148.2, 137.7, 120.6-120.3 (m, 1C), 118.9-118.3 (m, 1C), 116.8-116.0 (m, 1C), 113.6-112.4 (m, 2C), 111.5-110.2 (m, 1C), 109.1-108.6 (m, 1C), 107.5, 49.2, 45.2, 31.4, 27.4, 25.3 (t, J = 22.0 Hz, 1C); ^{19}F NMR (470.7 MHz, $CDCl_3$): -80.7 (t, J = 9.5 Hz, 3F), -114.2 (t, J = 14.0 Hz, 2F), -121.8 (s, 2F), -122.8 (s, 2F), -123.4 (s, 2F), 126.1 (dd, J = 14.0, 9.5 Hz, 2F); FT-IR (neat): 1710, 1600, 1486, 1318, 1206, 1128, 735 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{19}H_{18}N_2OF_{13}$ $[M+H]^+$ 537.1212, found 537.1230.

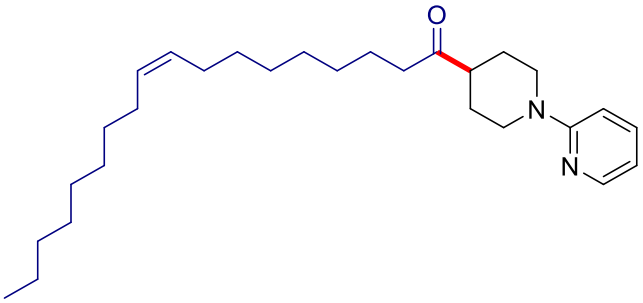


Benzyl (*S*)-3-((*tert*-Butoxycarbonyl)amino)-5-cyclohexyl-5-oxopentanoate (3r). The title compound was obtained as a liquid in 87% yield (0.5 mmol scale, 175.5 mg). ^{1}H NMR (500 MHz, $CDCl_3$): 7.30-7.25 (m, 5H), 5.25 (s, 1H), 5.10-5.04 (m, 2H), 4.26-4.19 (m, 1H), 2.81-2.58 (m, 4H), 2.24-2.19 (m, 1H), 1.72-1.59 (m, 5H), 1.38 (s, 9H), 1.25-1.14 (m, 5H); ^{13}C NMR (125.8 MHz,

$CDCl_3$): δ 212.2, 171.3, 155.2, 136.1, 128.7, 128.4, 79.5, 66.5, 51.3, 44.7, 43.6, 38.5, 28.5, 28.5, 28.4, 26.0, 25.8, 25.7; FT-IR (neat): 3351, 1726, 1698, 1686, 1523, 1273, 1169, 695 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{23}H_{34}NO_5$ $[M+H]^+$ 404.2437, found 404.2420.

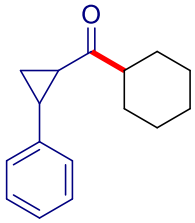


5-(Cyclohexanecarbonyl)tricyclo[2.2.1.0^{2,6}]heptan-3-one (3s). The title compound was obtained as a liquid in 83% yield (0.5 mmol scale, 90.5 mg). ^{1}H NMR (500 MHz, $CDCl_3$): 3.11 (s, 1H), 2.40-2.34 (m, 1H), 2.26-2.14 (m, 3H), 1.84-1.80 (m, 2H), 1.72-1.60 (m, 5H), 1.41 (t, *J* = 5.5 Hz, 1H), 1.37-1.12 (m, 5H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 211.8, 210.5, 51.4, 49.3, 41.3, 29.2, 28.7, 28.2, 25.9, 25.8, 25.5, 21.4, 19.9, 19.8; FT-IR (neat): 1756, 1700, 1239, 943, 832 cm^{-1}; HRMS (CI+) m/z calcd. for $C_{14}H_{19}O_2$ $[M+H]^+$ 219.1385, found 219.1382.



(*Z*)-1-(1-(Pyridin-2-yl)piperidin-4-yl)octadec-9-en-1-one (3t). The title compound was obtained as a liquid in 57% yield (0.5 mmol scale, 121.6 mg). ^{1}H NMR (500 MHz, $CDCl_3$): 8.14 (dd, *J* = 5.0, 1.5 Hz, 1H), 7.43-7.40 (m, 1H), 6.62 (d, *J* = 8.5 Hz, 1H), 6.55 (dd, *J* = 7.0, 5.0 Hz, 1H), 5.33-5.31 (m, 2H), 4.27 (dd, *J* = 13.0, 3.5 Hz, 2H), 2.89-2.84 (m, 2H), 2.54-2.49 (m, 1H), 2.44 (t, *J* =

7.5 Hz, 2H), 2.01-1.97 (m, 3H), 1.89-1.86 (m, 2H), 1.68-1.60 (m, 2H), 1.56-1.53 (m, 2H), 1.27-1.23 (m, 21H), 0.87-0.84 (m, 3H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 212.7, 159.5, 148.1, 137.6, 130.2, 129.9, 113.1, 107.4, 49.1, 45.3, 40.7, 32.1, 30.0, 29.9, 29.7, 29.6, 29.5, 29.5, 29.5, 29.3, 27.5, 27.4, 23.8, 22.9, 14.3; FT-IR (neat): 2922, 2852, 1708, 1594, 1481, 1436, 769 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{28}H_{47}N_2O$ $[M+H]^+$ 427.3688, found 427.3692.



Cyclohexyl(2-phenylcyclopropyl)methanone (3u). The title compound was obtained as a liquid in 81% yield (0.5 mmol scale, 92.4 mg). ^{1}H NMR (500 MHz, $CDCl_3$): 7.30-7.27 (m, 2H), 7.22-7.19 (m, 1H), 7.11-7.09 (m, 2H), 2.55-2.49 (m, 1H), 2.47-2.43 (m, 1H), 2.25-2.22 (m, 1H), 1.95-1.91 (m, 2H), 1.82-1.76 (m, 2H), 1.69-1.63 (m, 2H), 1.44-1.18 (m, 6H); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ 211.9, 140.8, 128.7, 126.6, 126.3, 51.8, 31.1, 29.1, 28.6, 28.5, 26.1, 26.0, 25.9, 18.7; FT-IR (neat): 1689, 1398, 1067, 749, 695 cm^{-1}; HRMS (ES+) m/z calcd. for $C_{16}H_{21}O$ $[M+H]^+$ 229.1592, found 229.1570.

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2) For information on these reactors and their construction see the supporting information of: (a) Patel, N. R.; Kelly, C. B.; Jouffroy, M.; Molander, G. A. *Org. Lett*. **2016**, *18*, 764. (b) Jouffroy, M.; Kelly, C. B.; Molander, G. A. *Org. Lett*. **2016**, *18*, 876.

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7) Fujimura, T.; Aoki, S.; Nakamura, E. *J. Org. Chem*. **1991**, *56*, 2809.

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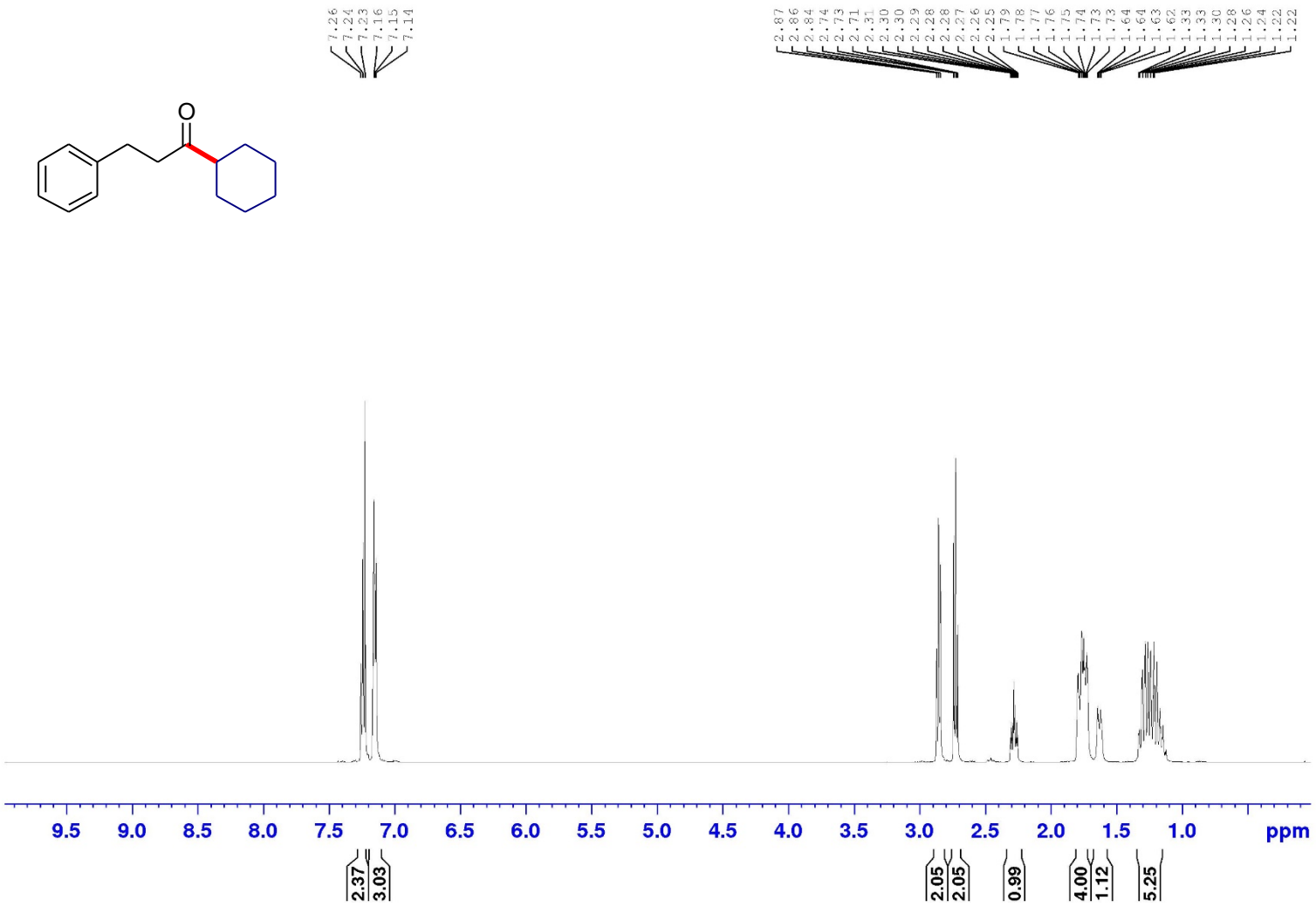
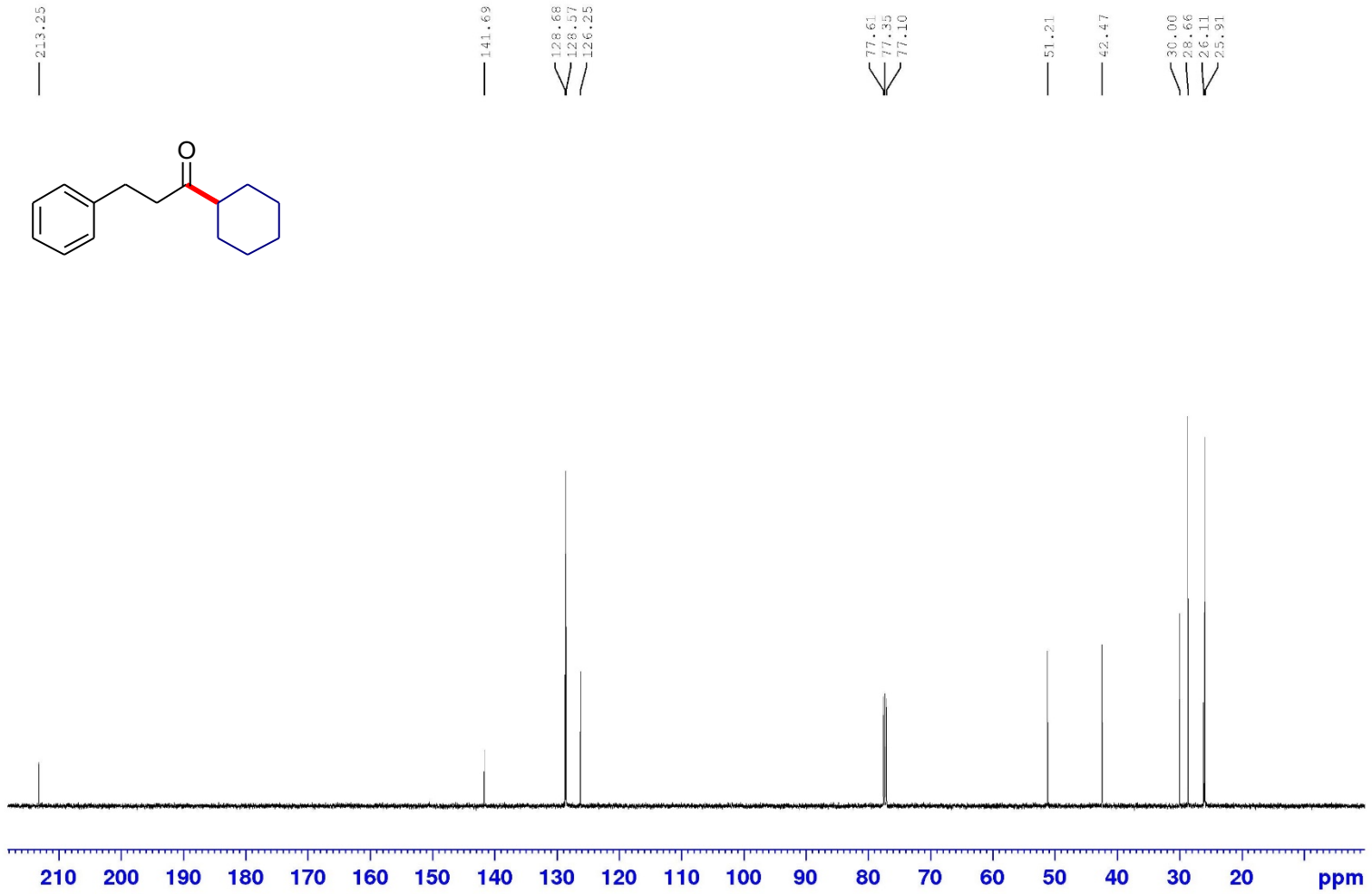
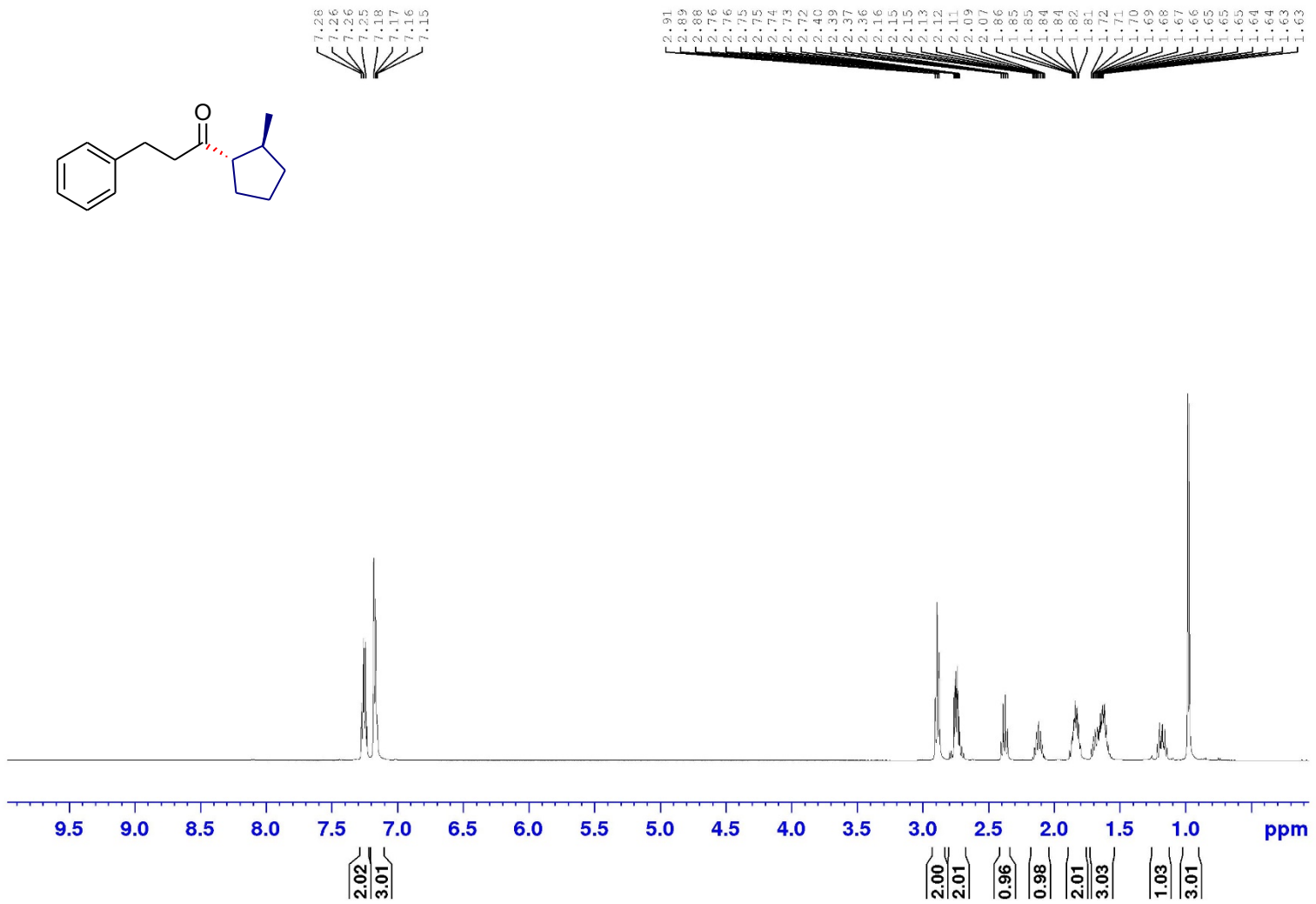


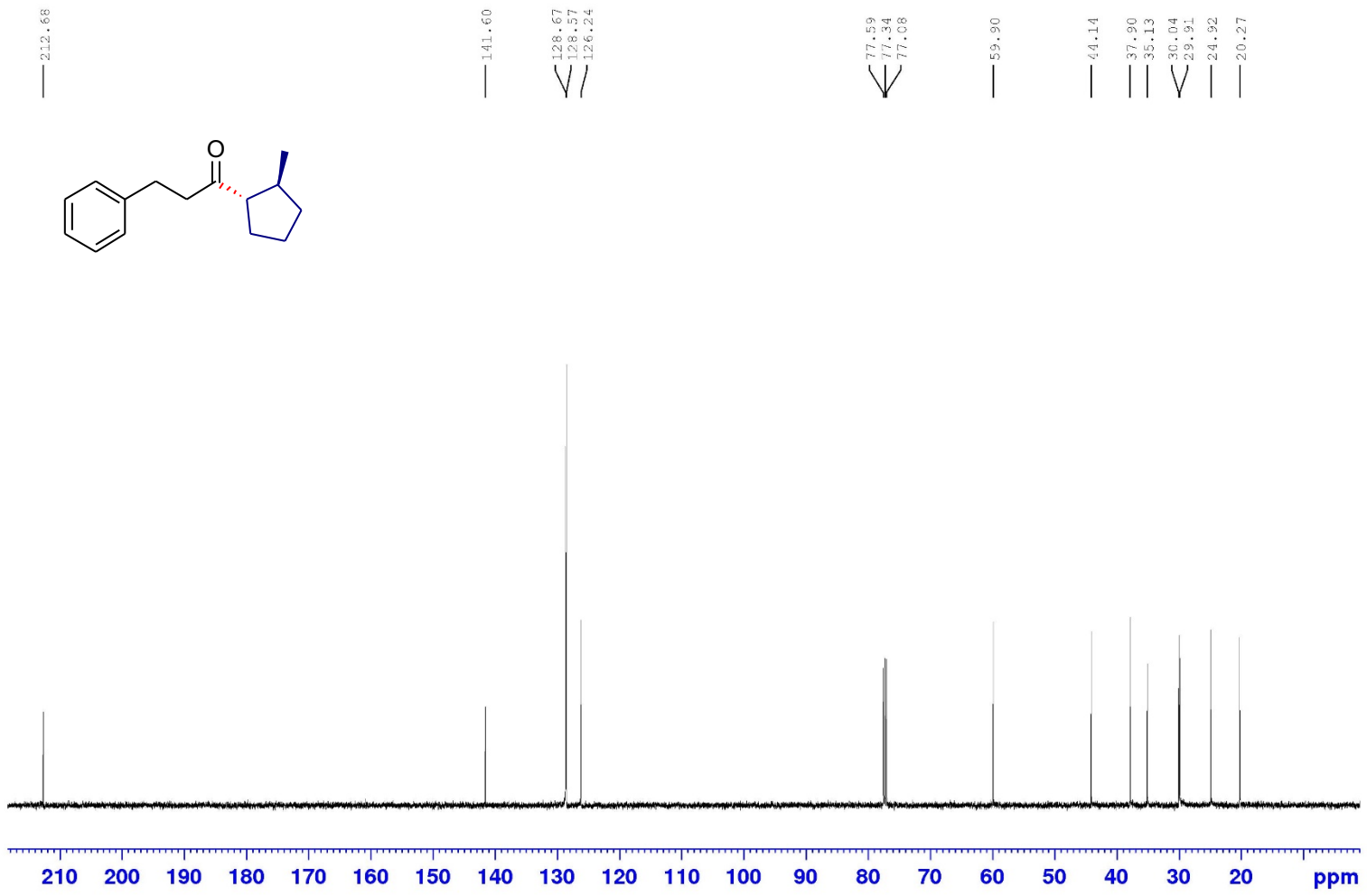
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 1-cyclohexyl-3-phenylpropan-1-one (2a)



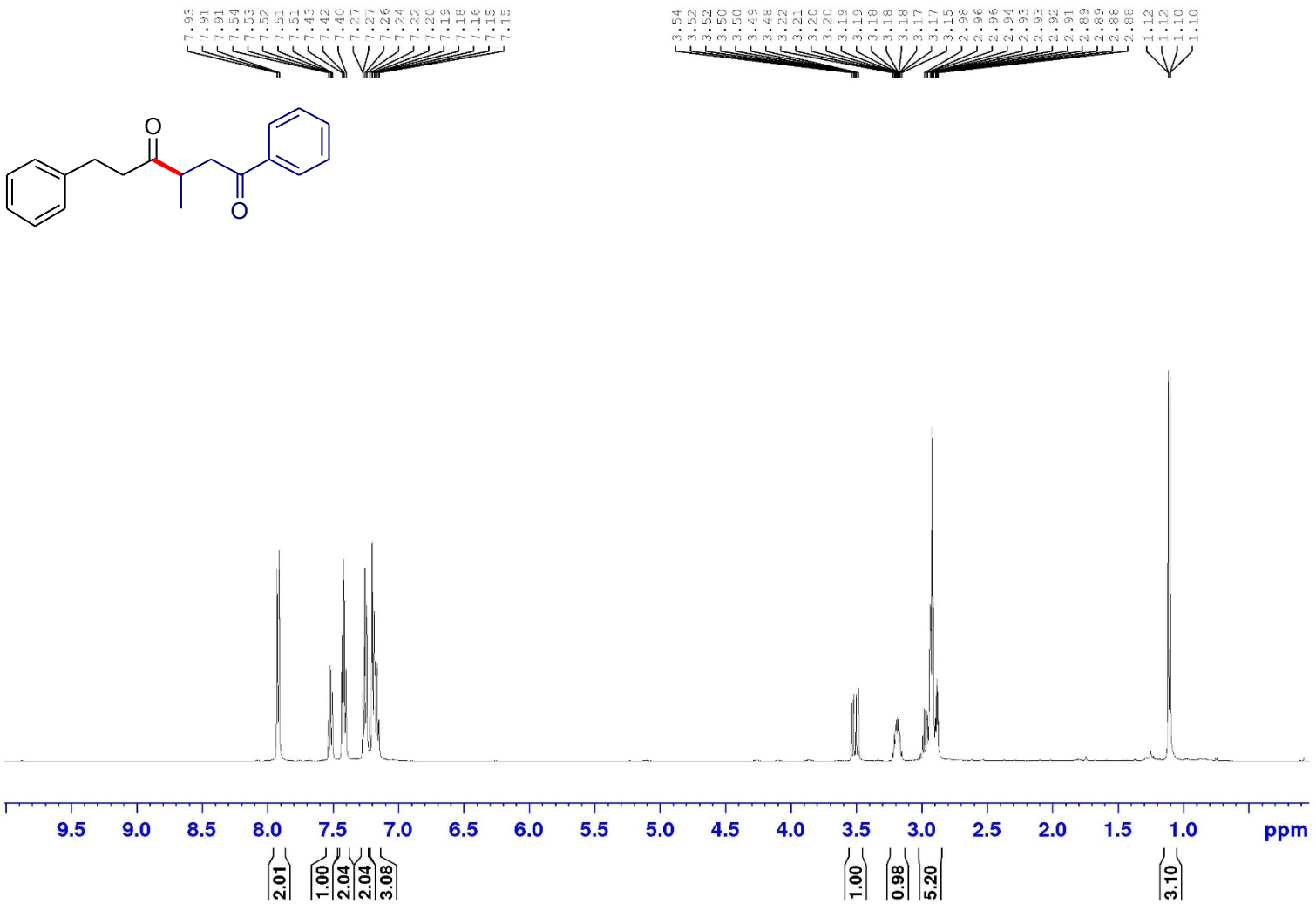
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of 1-cyclohexyl-3-phenylpropan-1-one (2a)



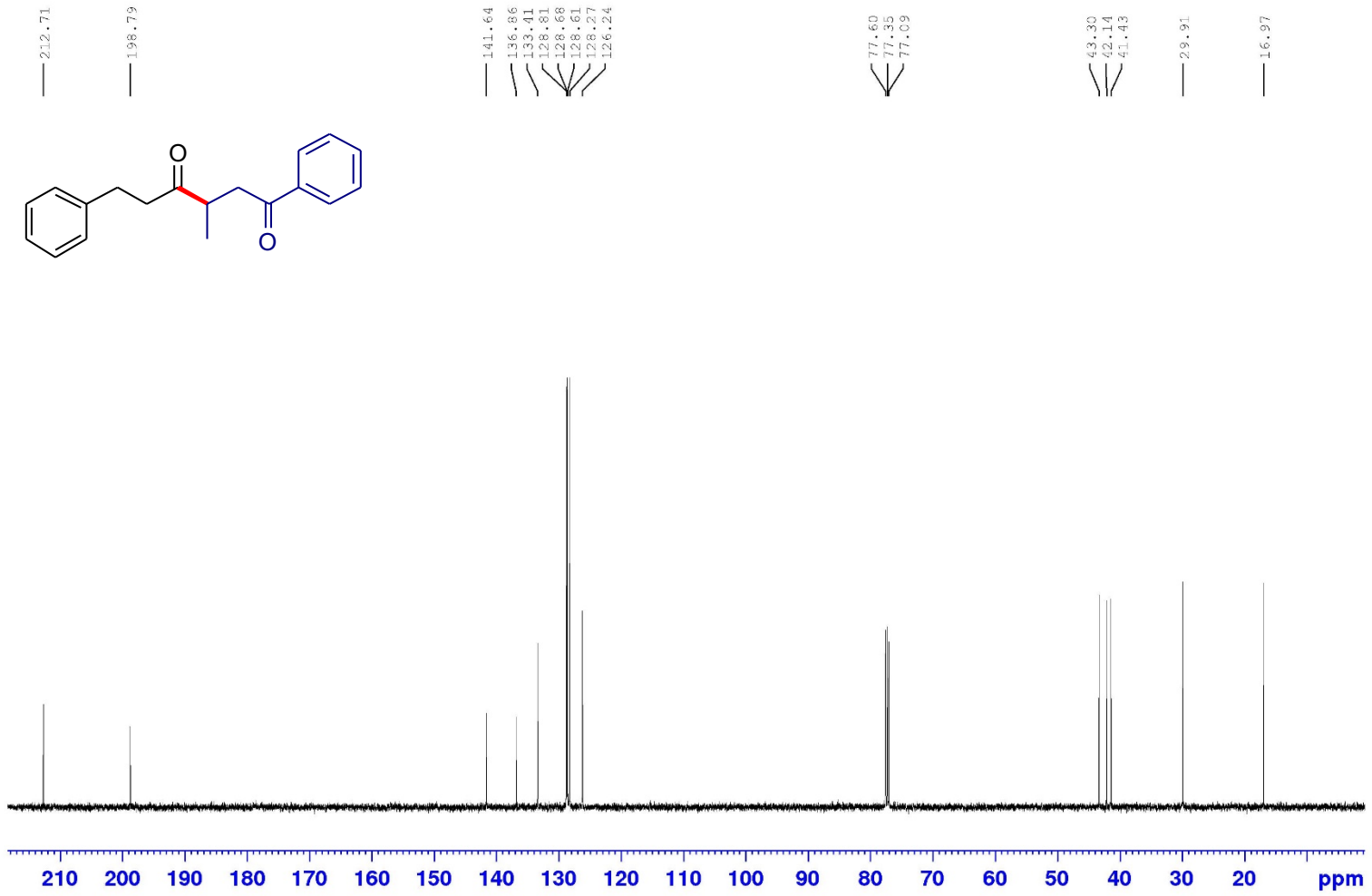
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 1-(2-Methylcyclopentyl)-3-phenylpropan-1-one (2b)



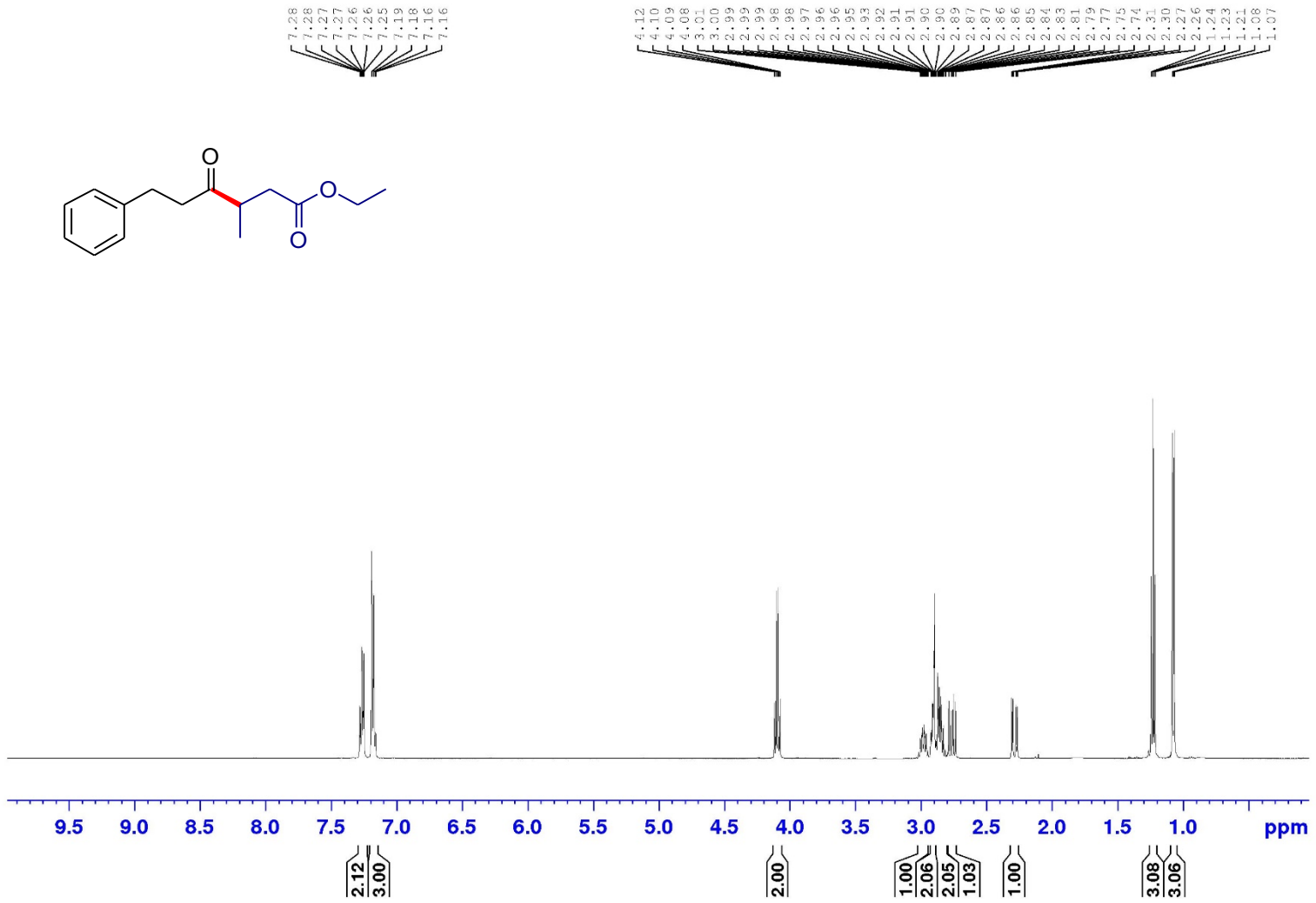
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of 1-(2-methylcyclopentyl)-3-phenylpropan-1-one (2b)



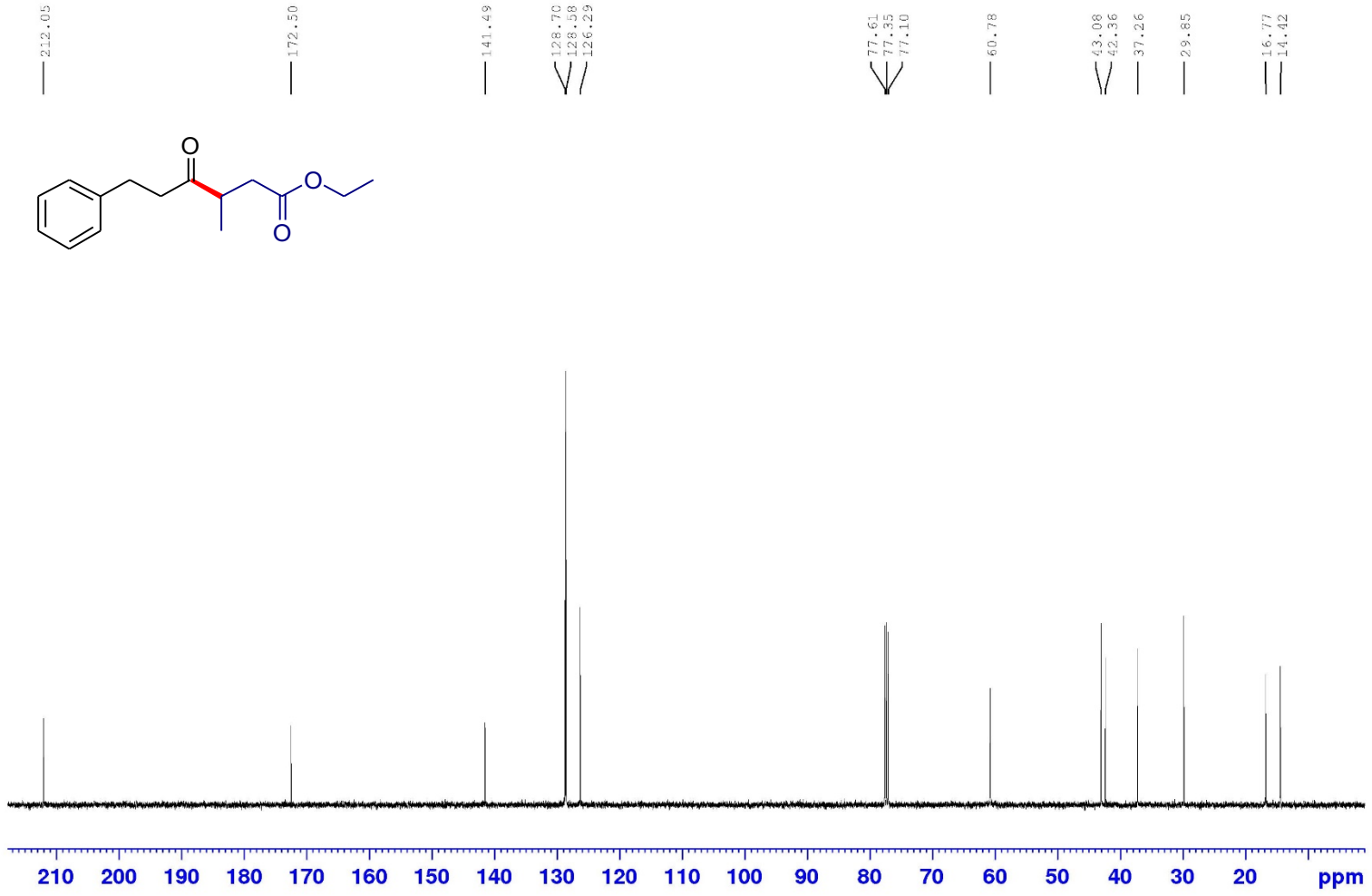
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 3-methyl-1,6-diphenylhexane-1,4-dione (2c)



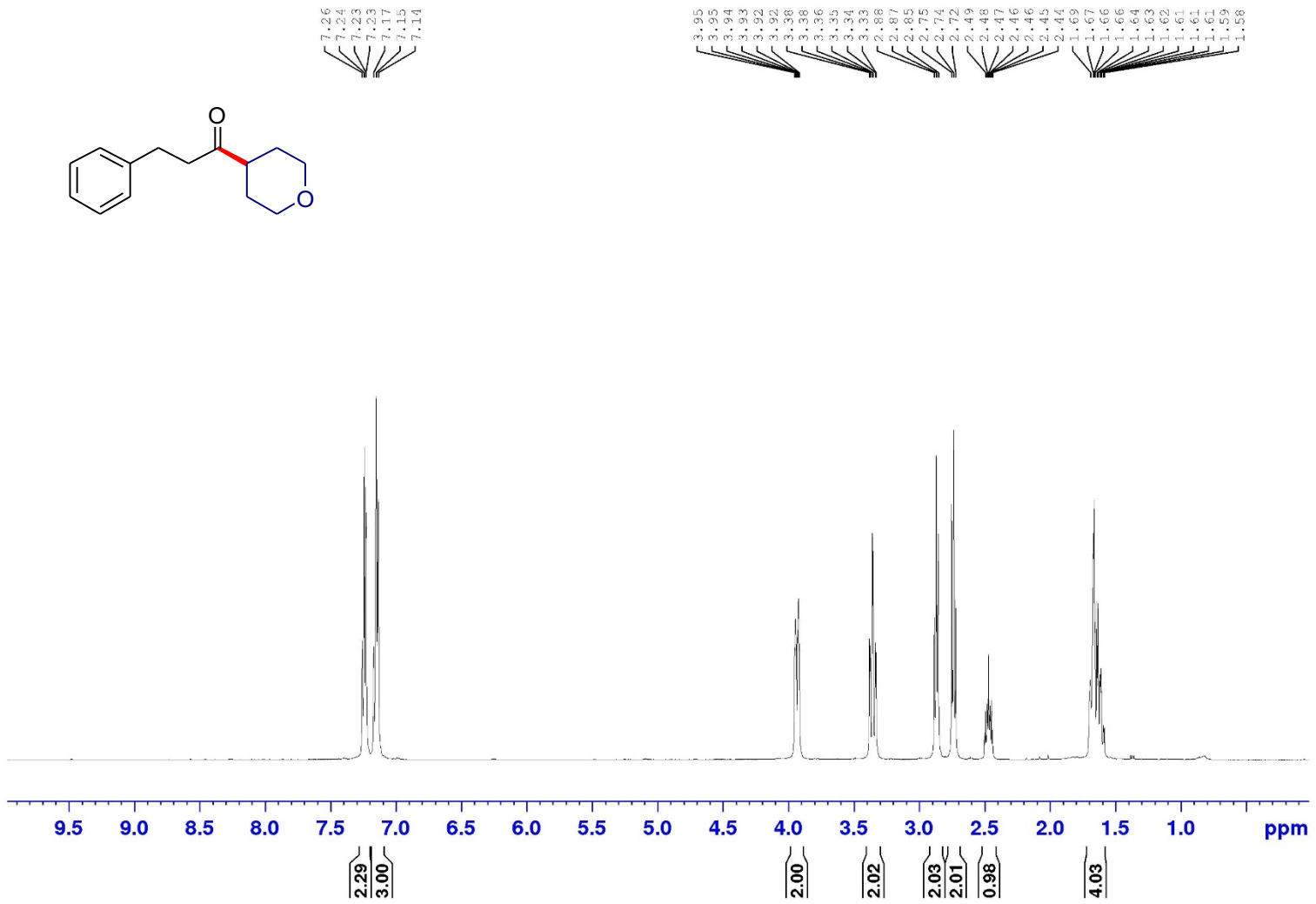
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of 3-methyl-1,6-diphenylhexane-1,4-dione (2c)



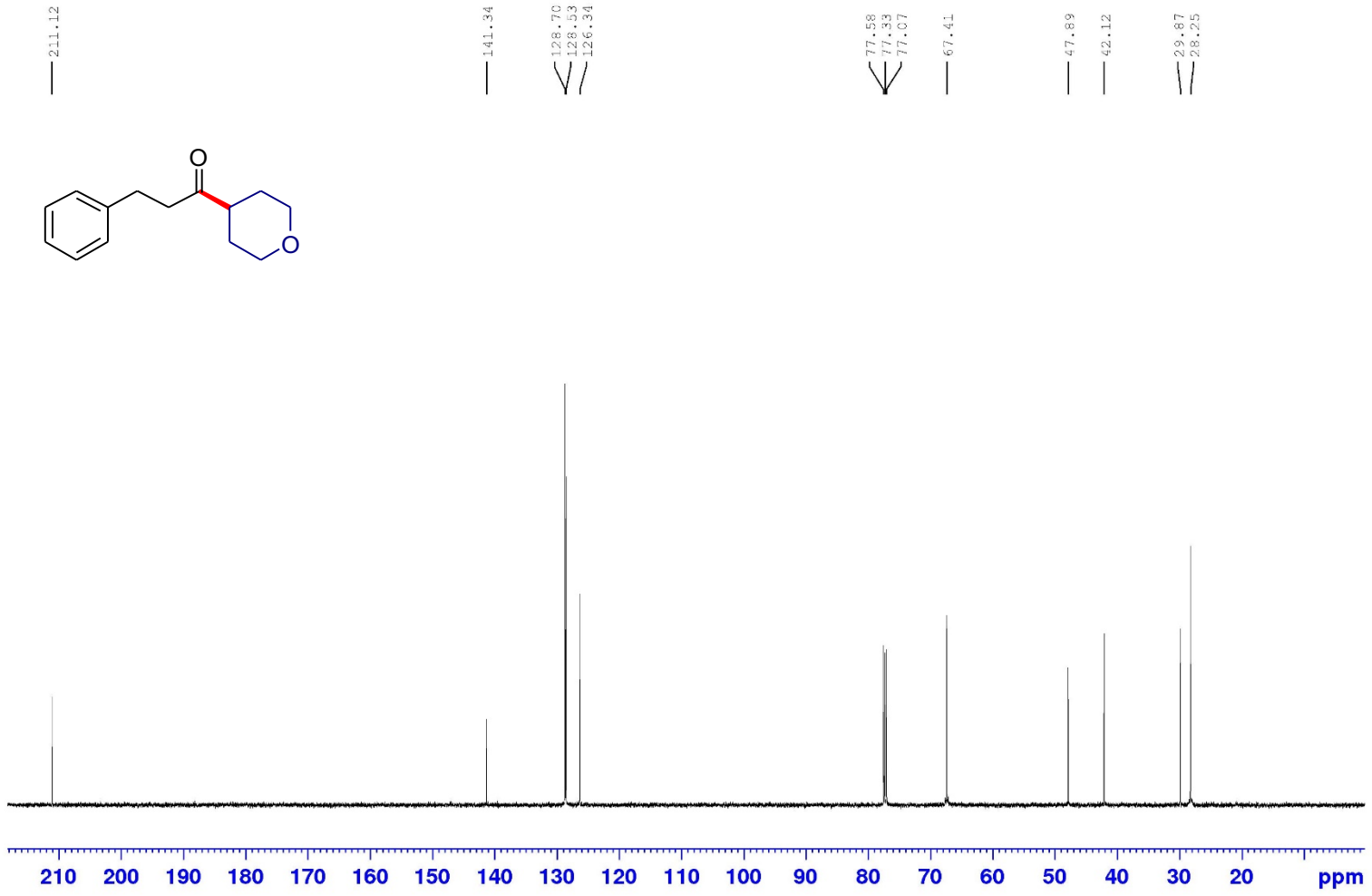
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of ethyl 3-Methyl-4-oxo-6-phenylhexanoate (2d)



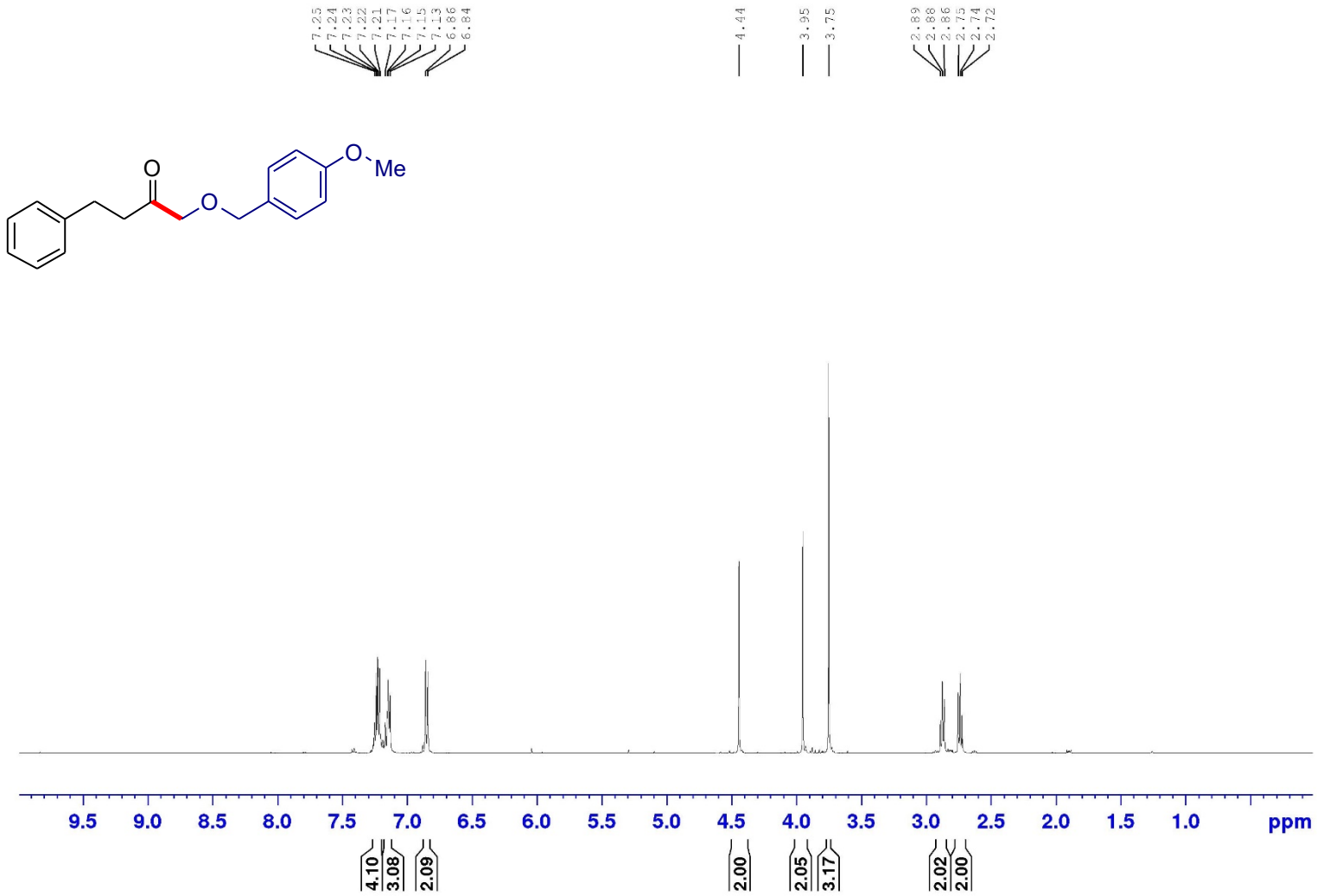
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of ethyl 3-Methyl-4-oxo-6-phenylhexanoate (2d)



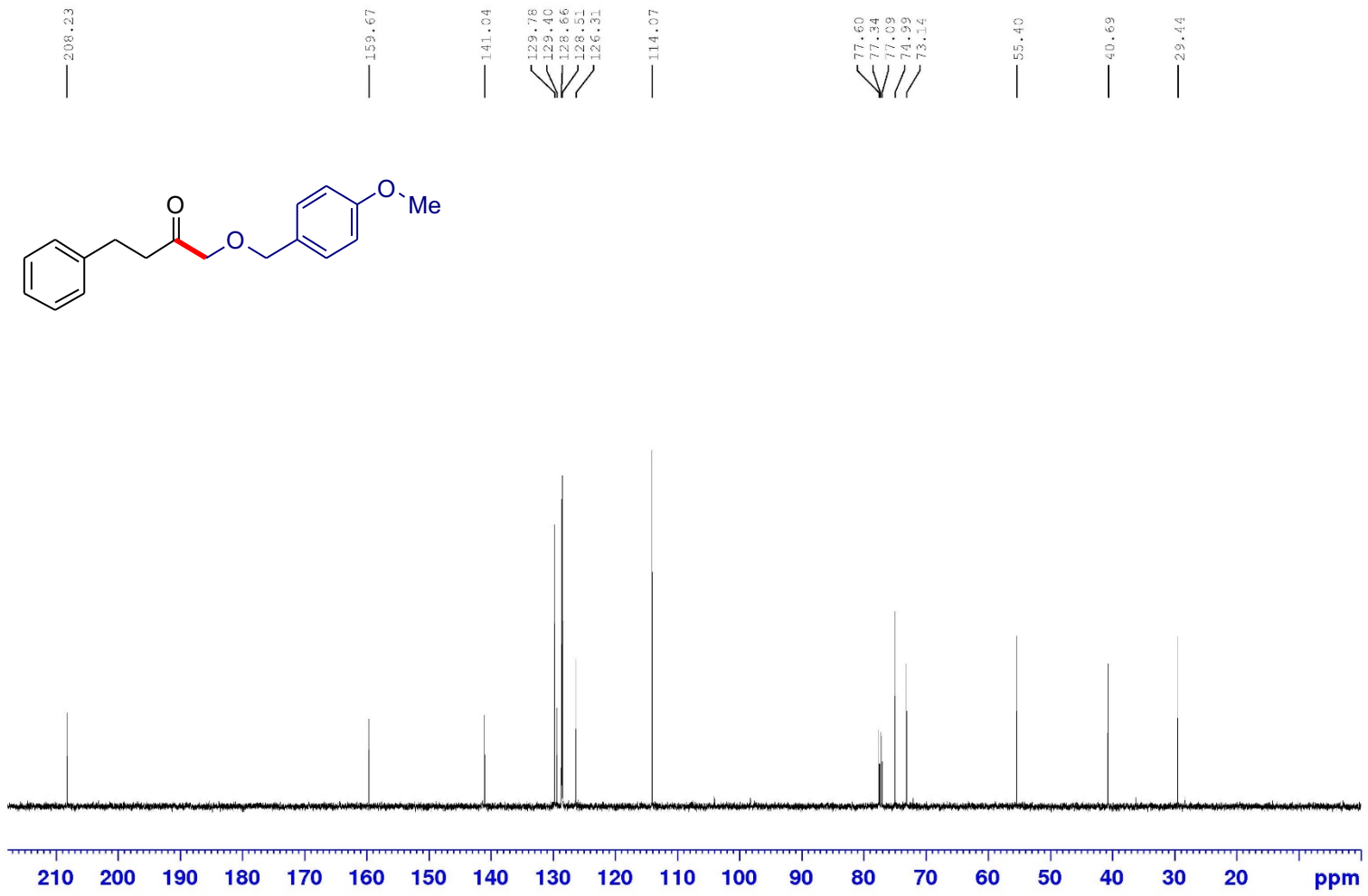
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 3-phenyl-1-(tetrahydro-2H-pyran-4-yl)propan-1-one (2e)



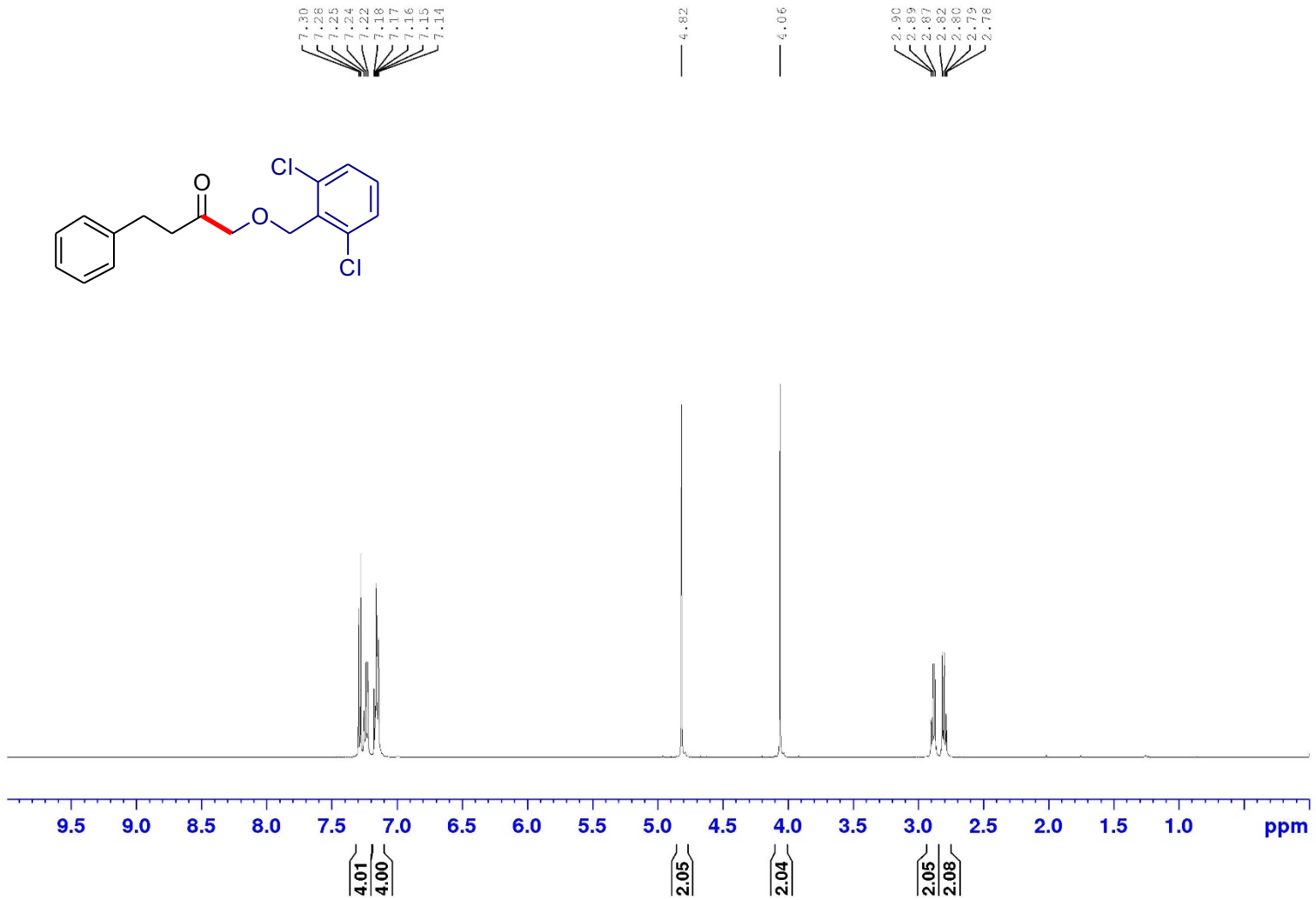
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of 3-phenyl-1-(tetrahydro-2H-pyran-4-yl)propan-1-one (2e)



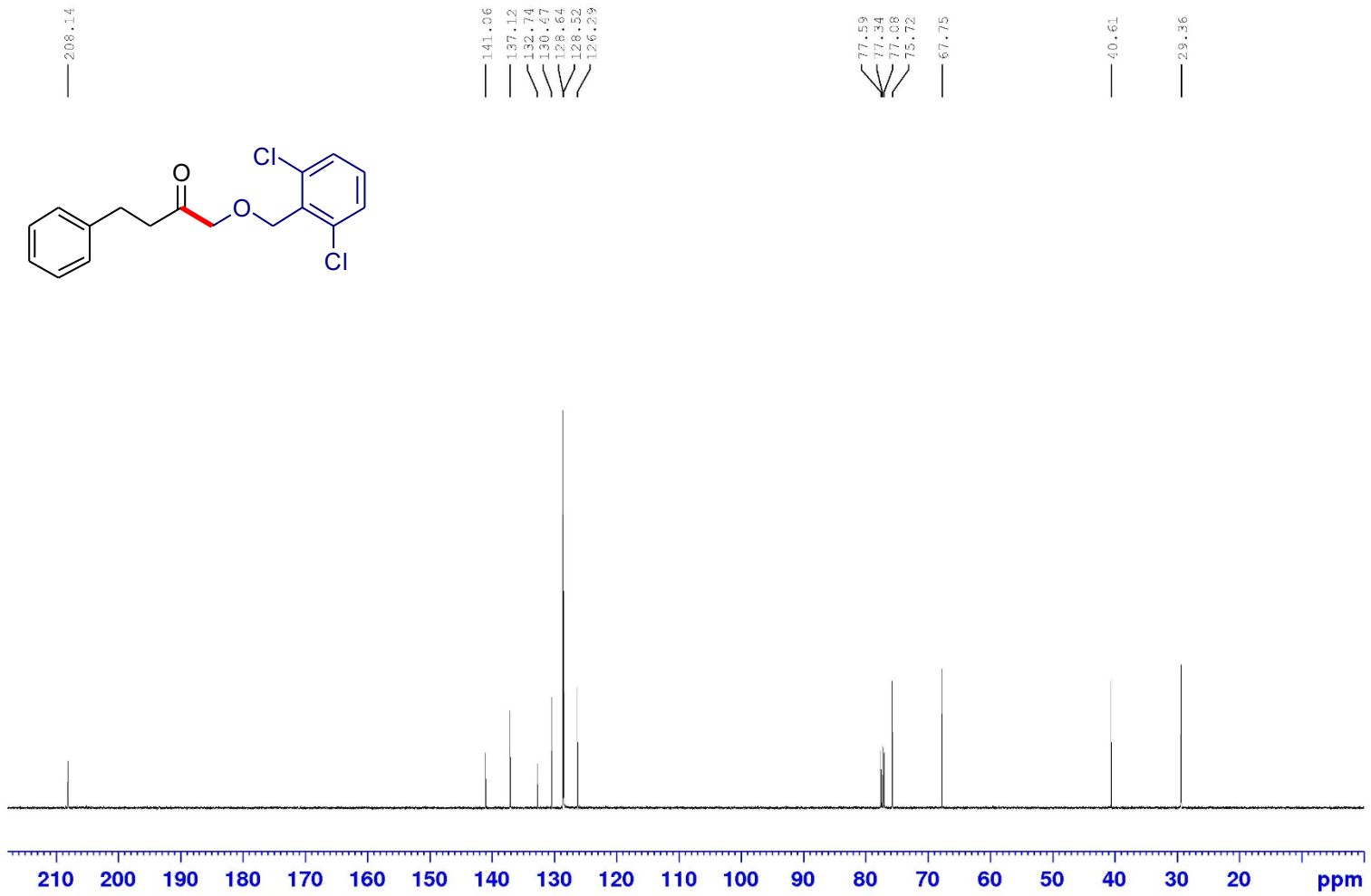
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 1-((4-methoxybenzyl)oxy)-4-phenylbutan-2-one (2f)



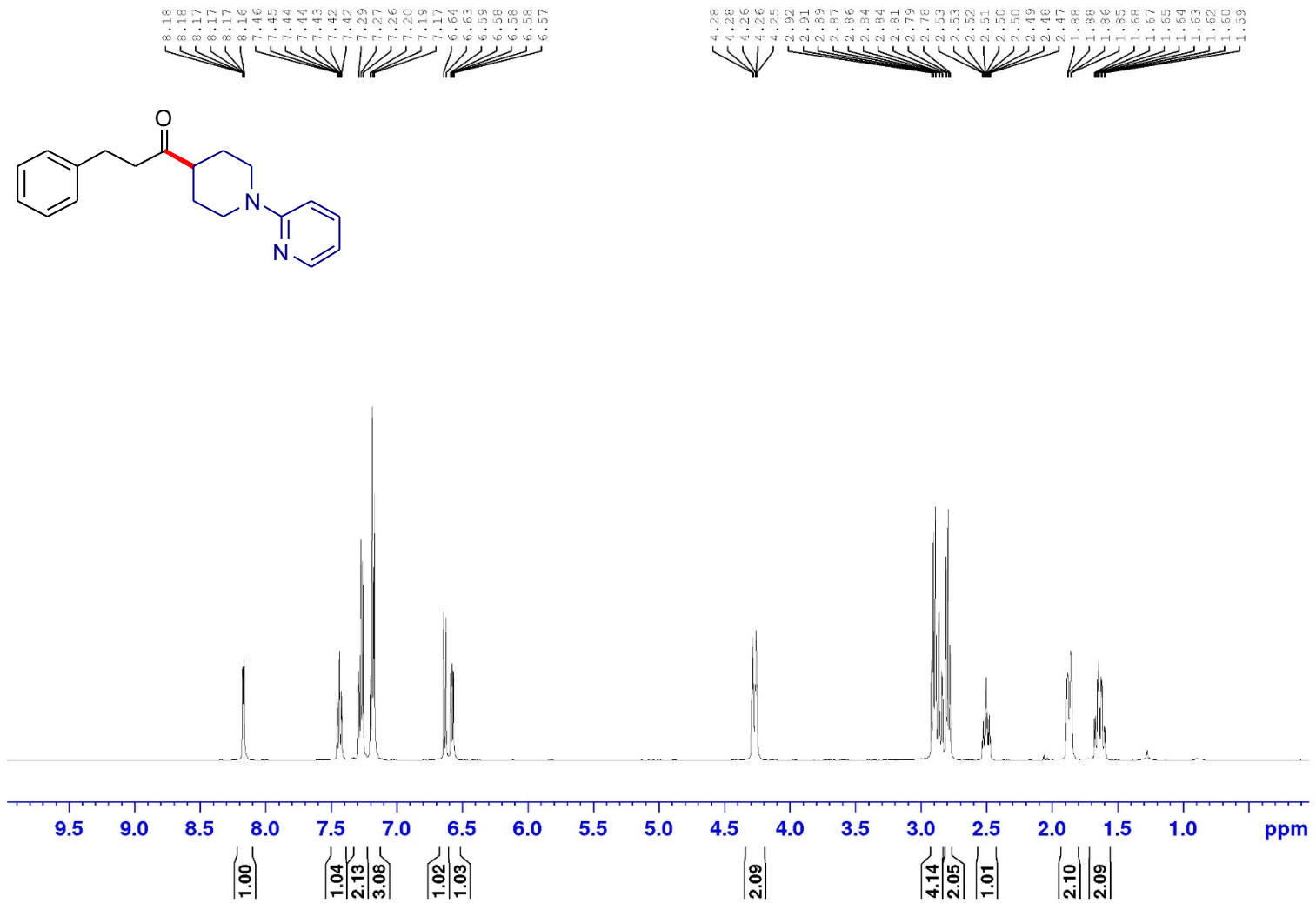
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of 1-((4-methoxybenzyl)oxy)-4-phenylbutan-2-one (2f)



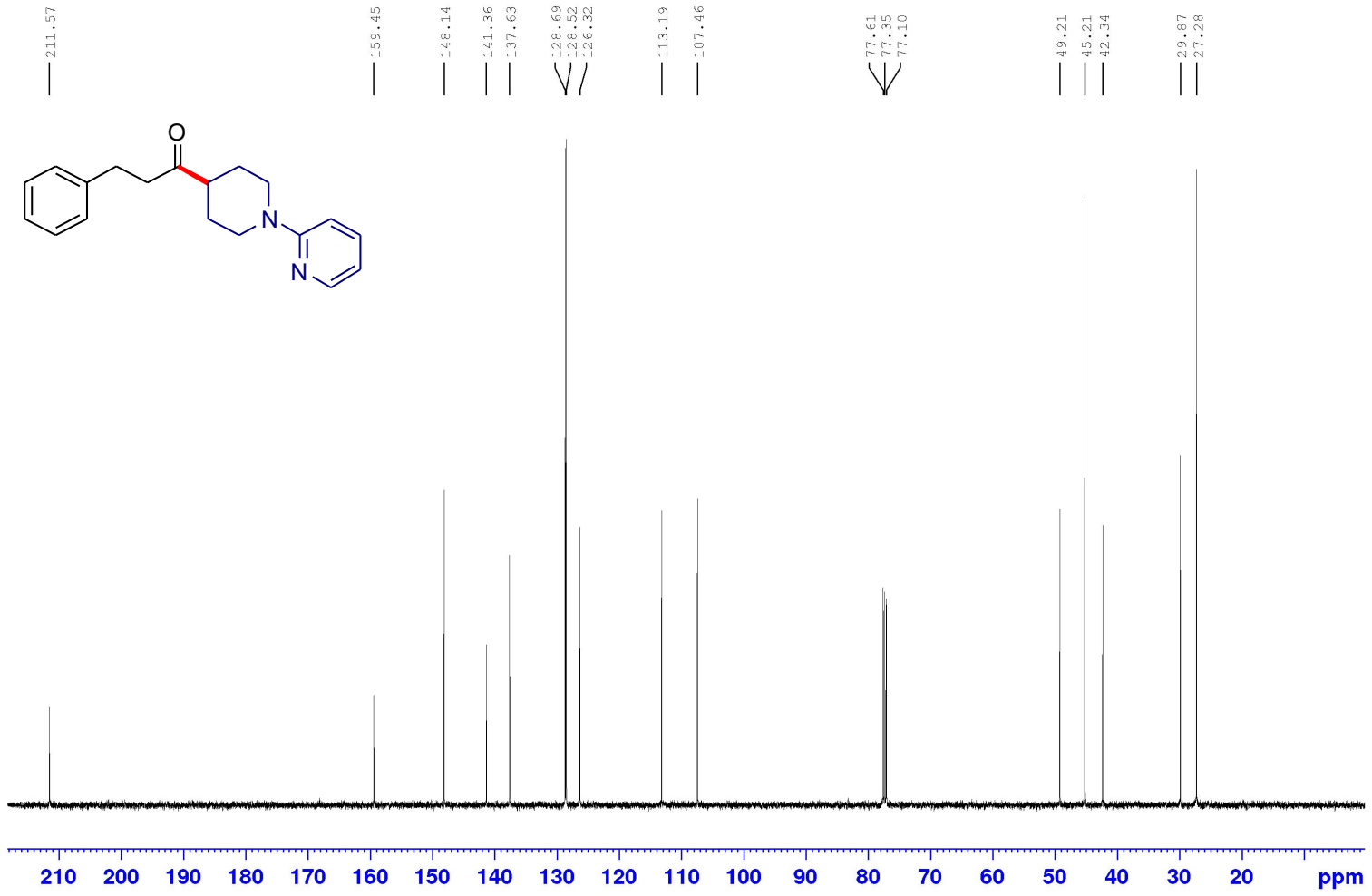
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 1-(2,6-dichlorobenzyloxy)-4-phenylbutan-2-one (2g)



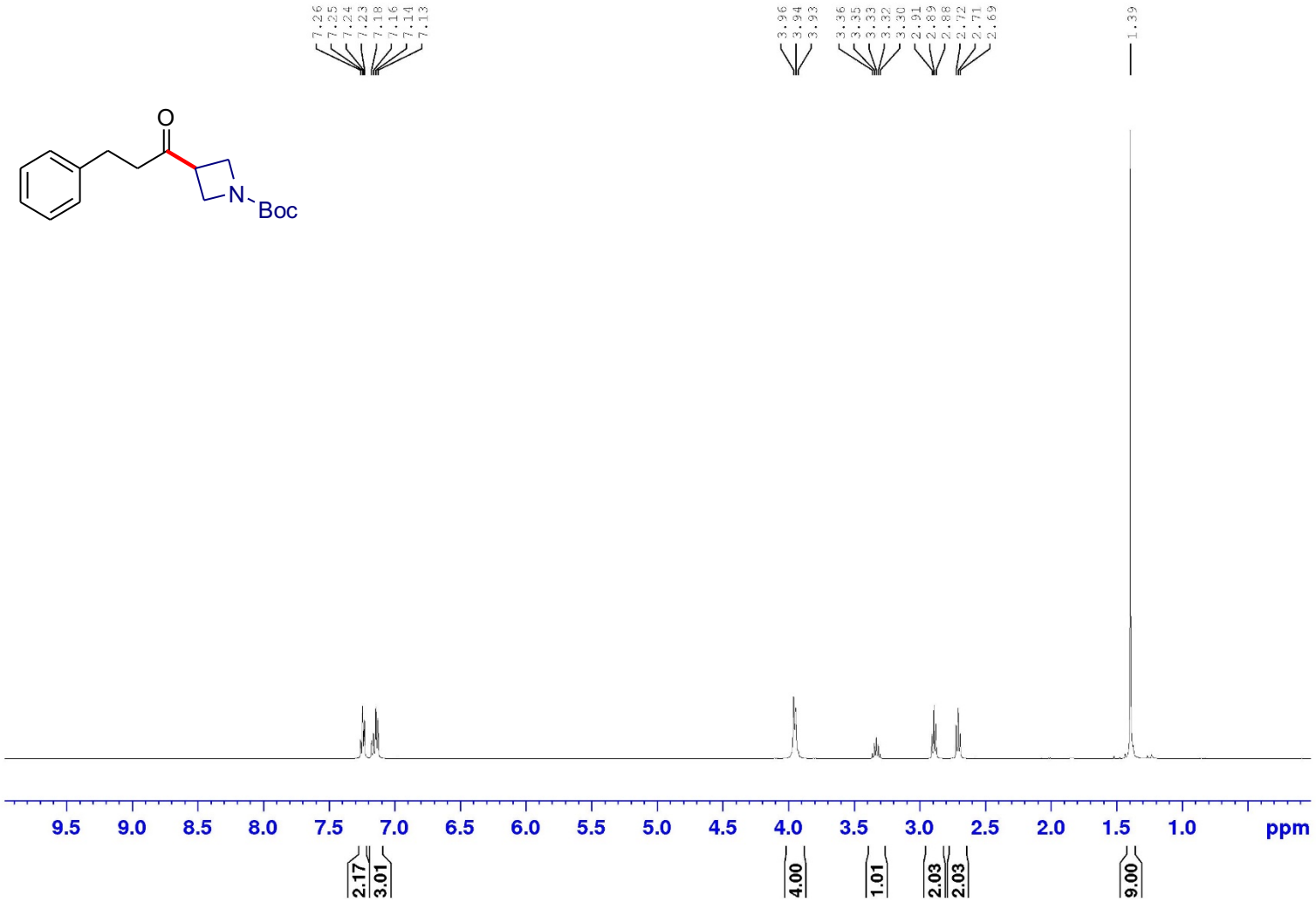
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of 1-(2,6-dichlorobenzyloxy)-4-phenylbutan-2-one (2g)



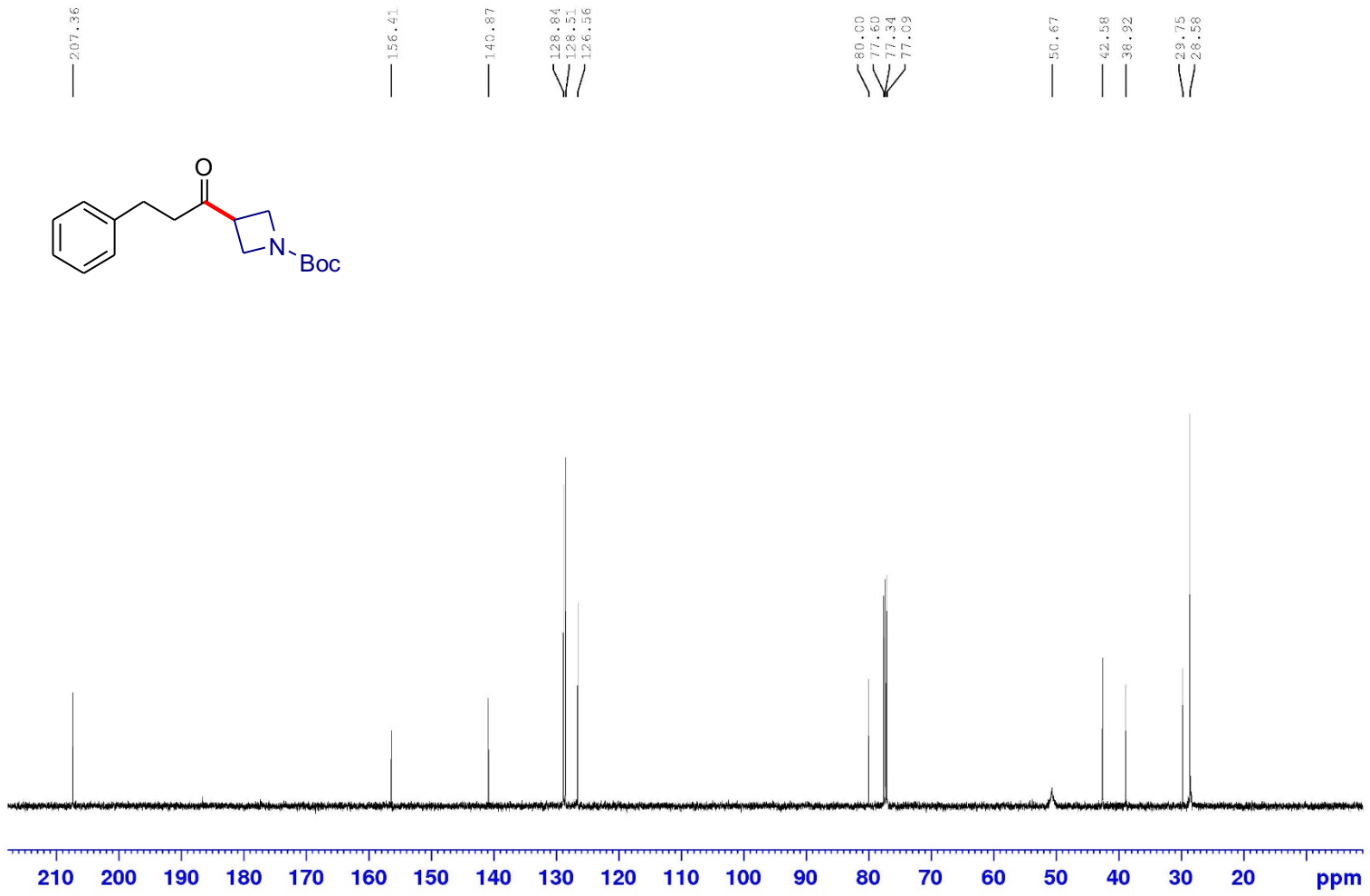
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 3-phenyl-1-(1-(pyridin-2-yl)piperidin-4-yl)propan-1-one (2h)



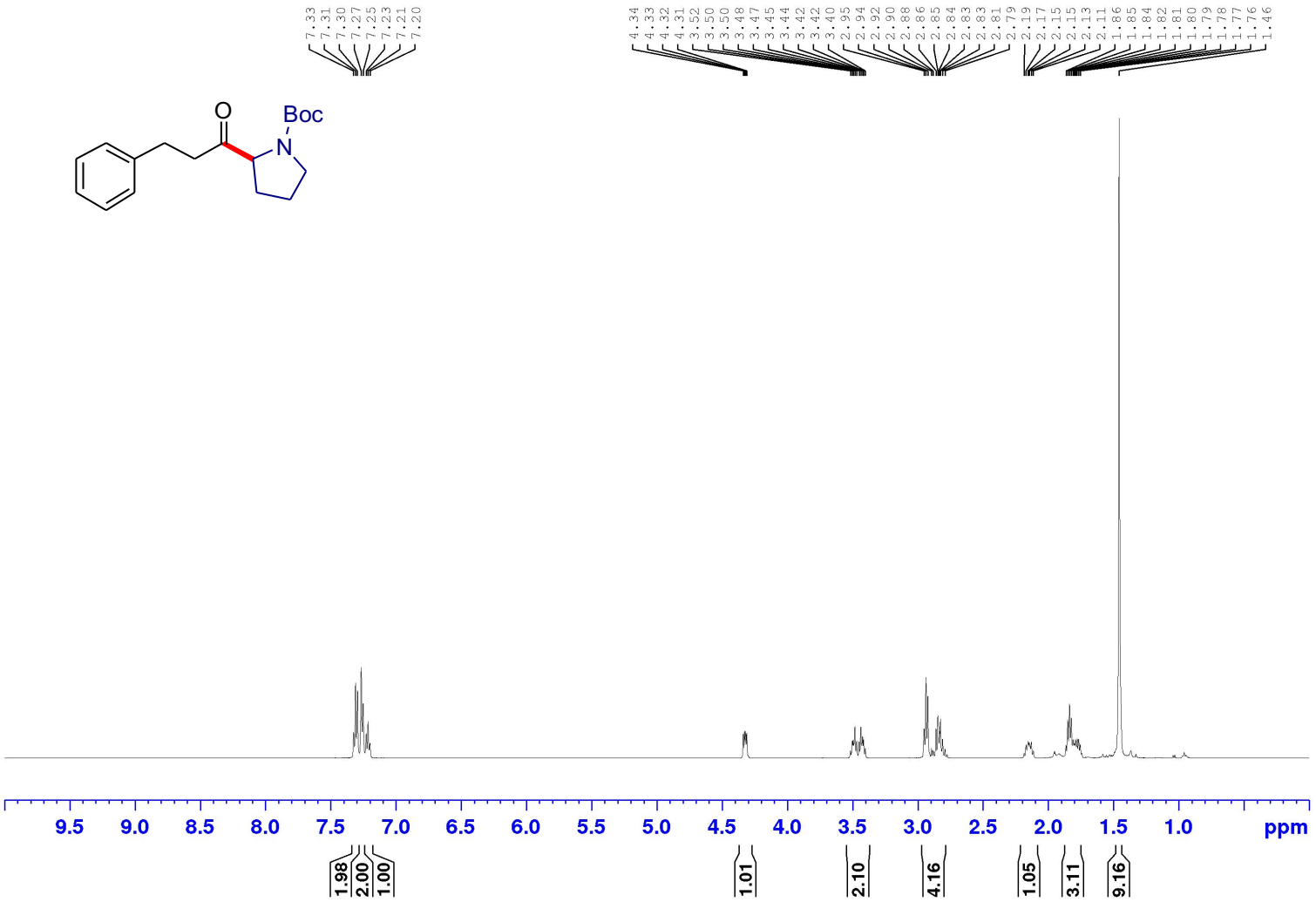
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of 3-phenyl-1-(1-(pyridin-2-yl)piperidin-4-yl)propan-1-one (2h)



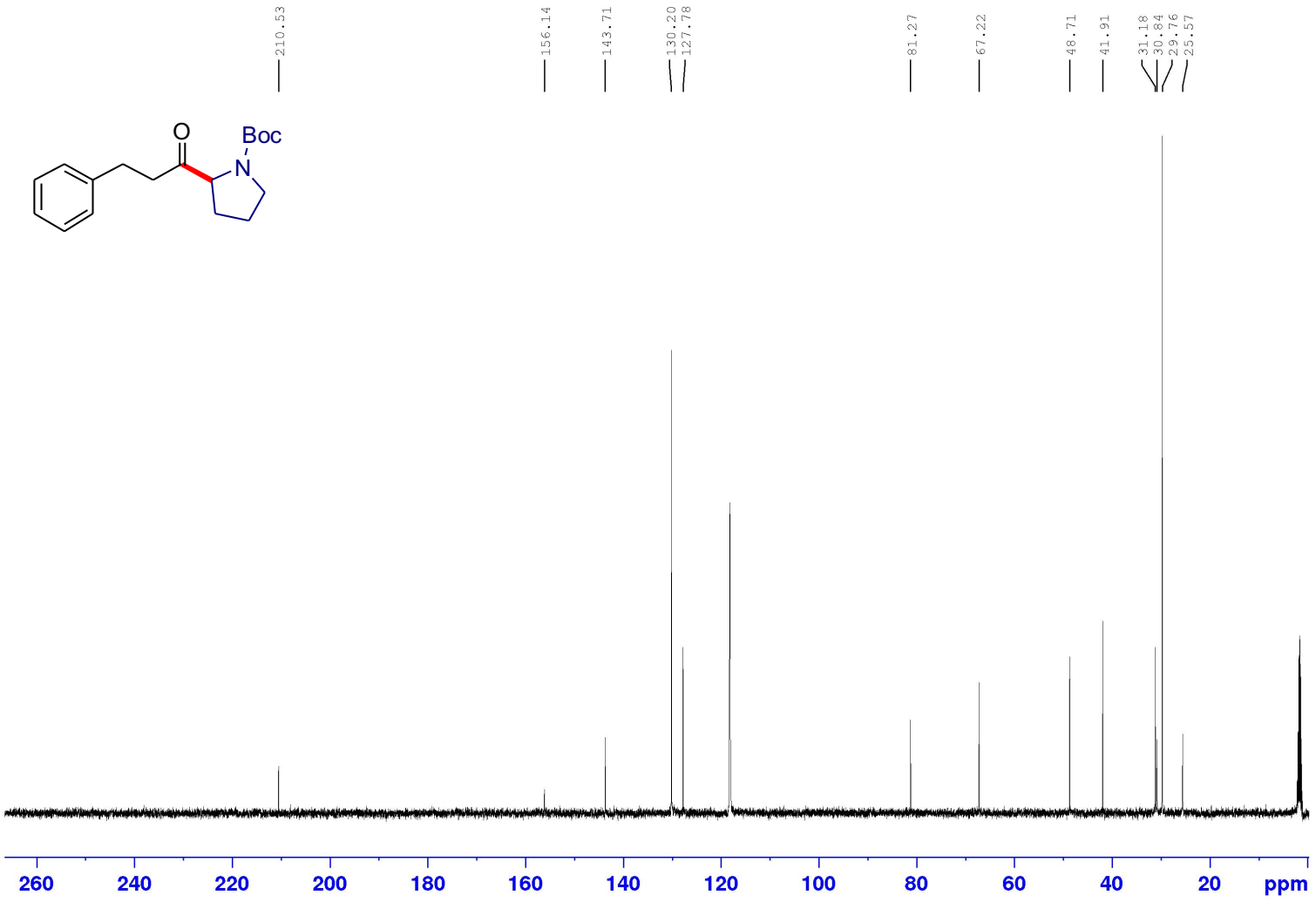
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of *tert*-butyl 3-(3-phenylpropanoyl)azetidine-1-carboxylate (2i)



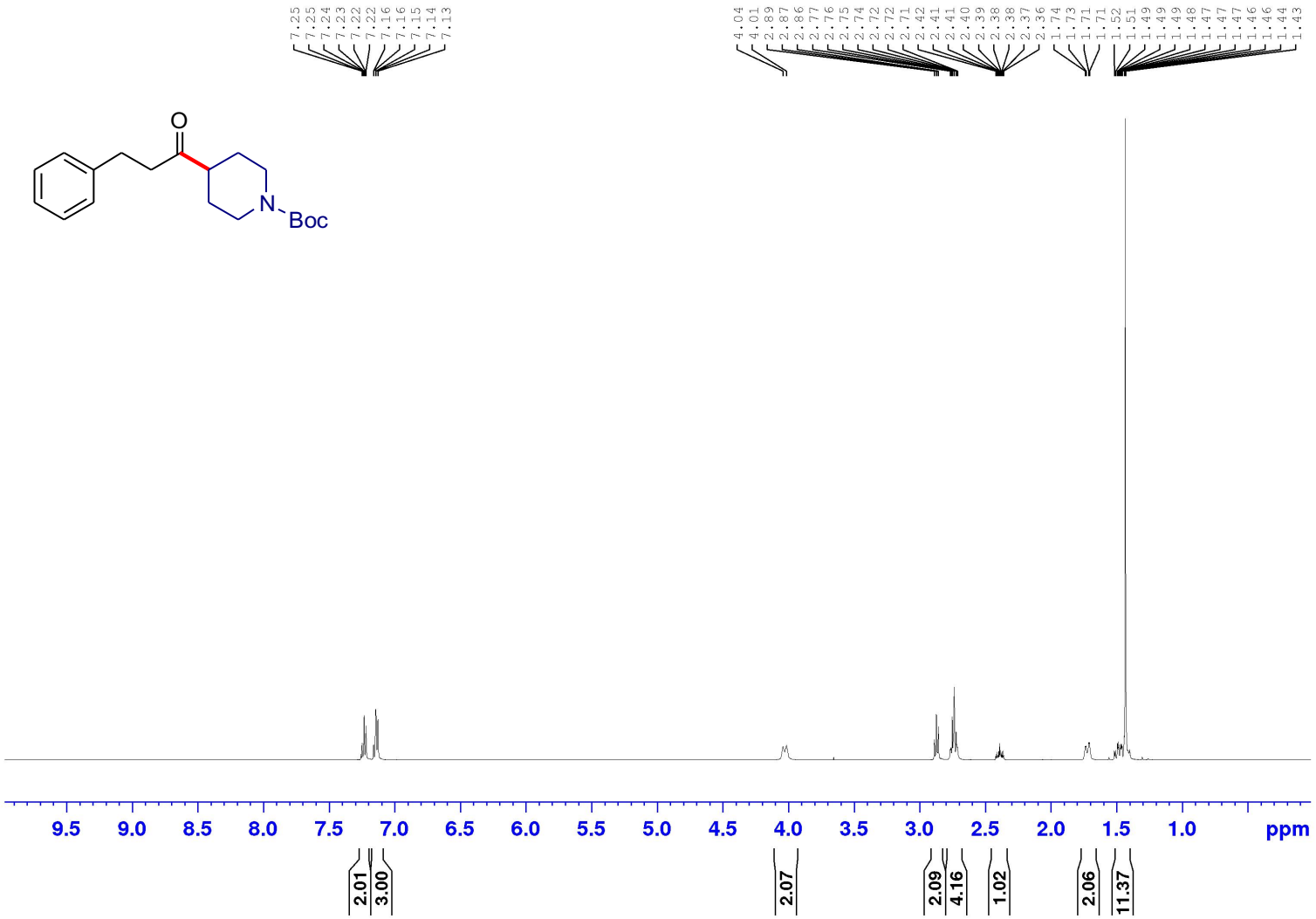
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of *tert*-butyl 3-(3-phenylpropanoyl)azetidine-1-carboxylate (2i)



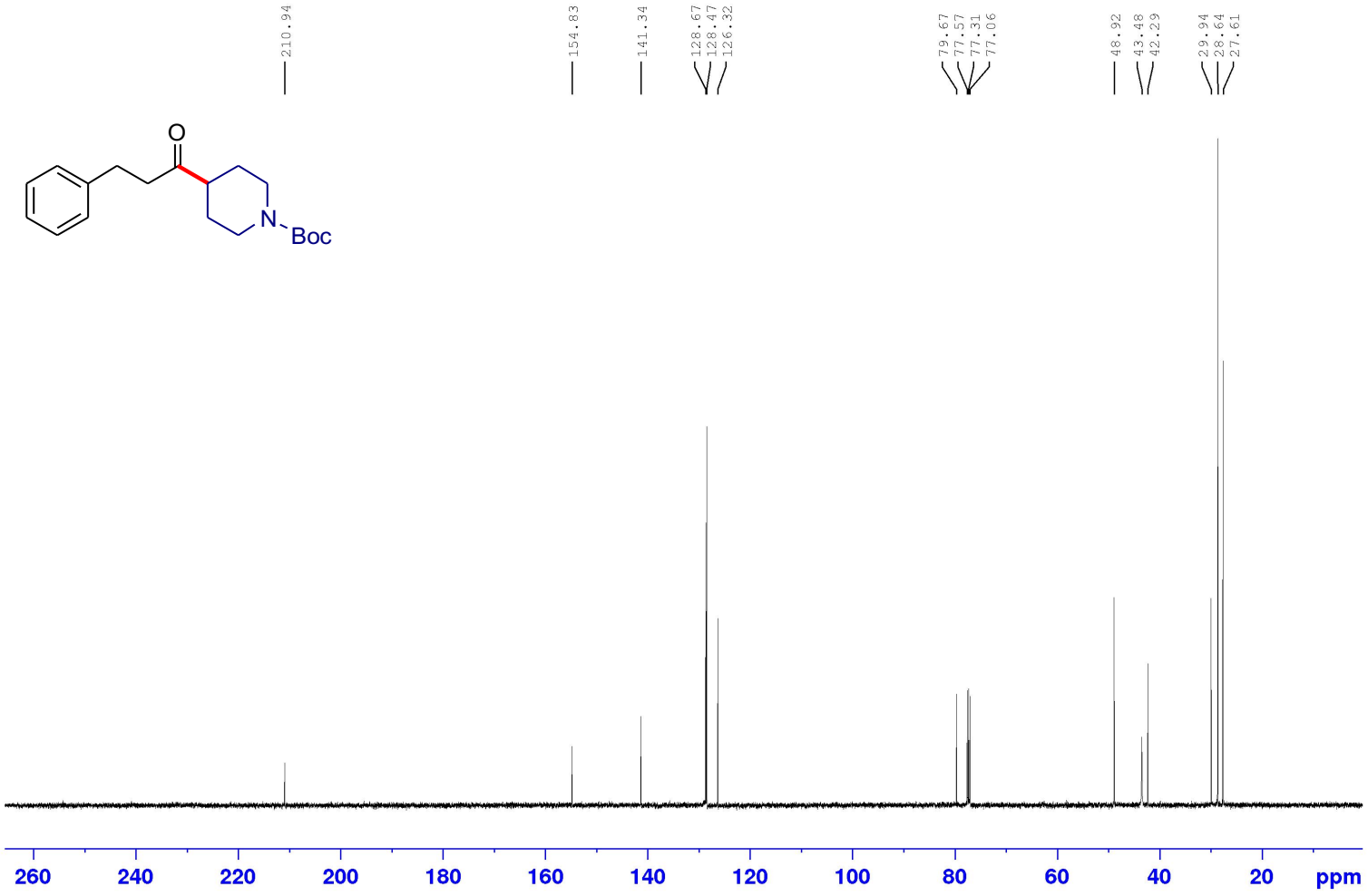
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of *tert*-butyl-2-(3-phenylpropanoyl)pyrrolidine-1-carboxylate (2j)



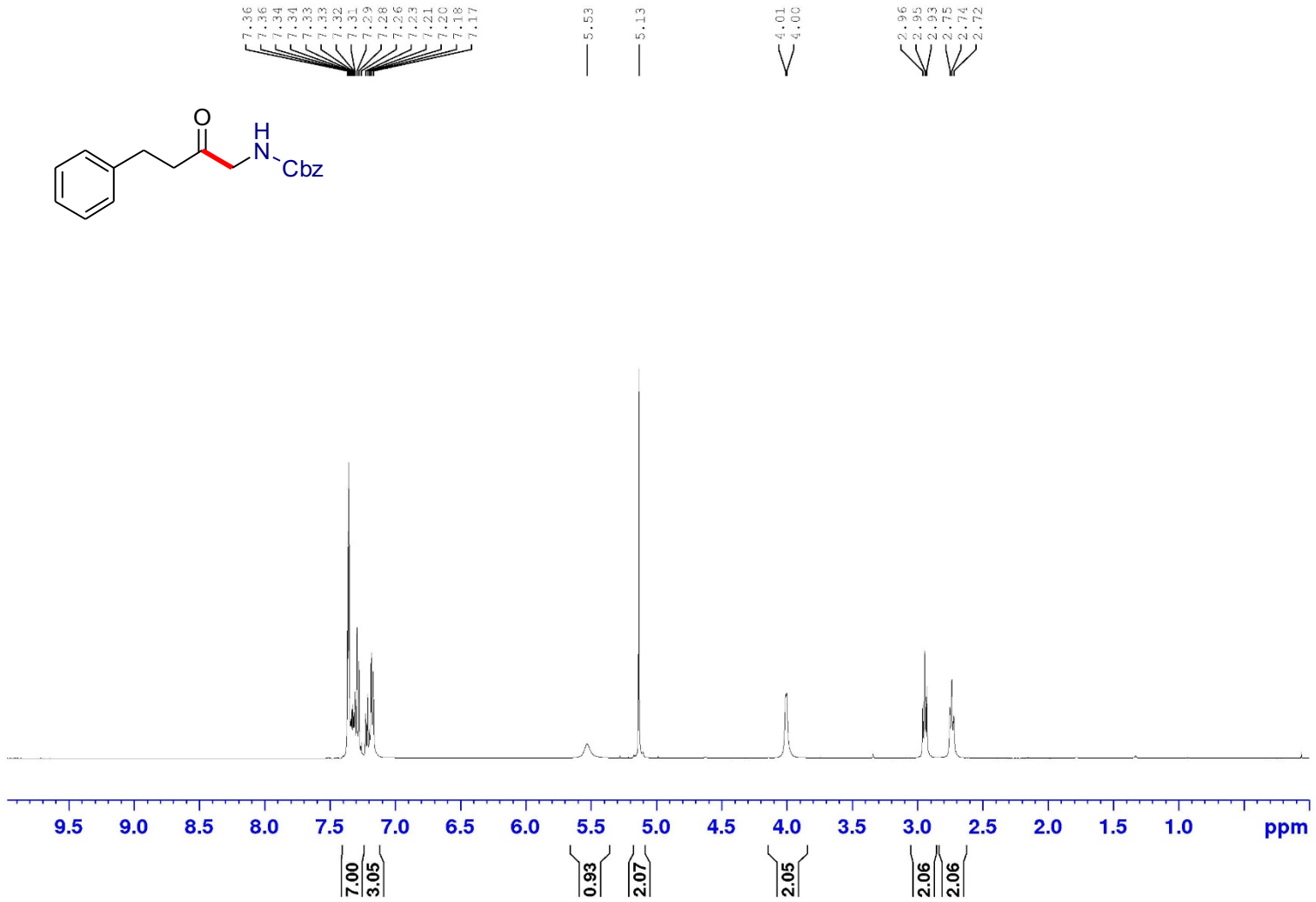
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of *tert*-butyl-2-(3-phenylpropanoyl)pyrrolidine-1-carboxylate (2j)



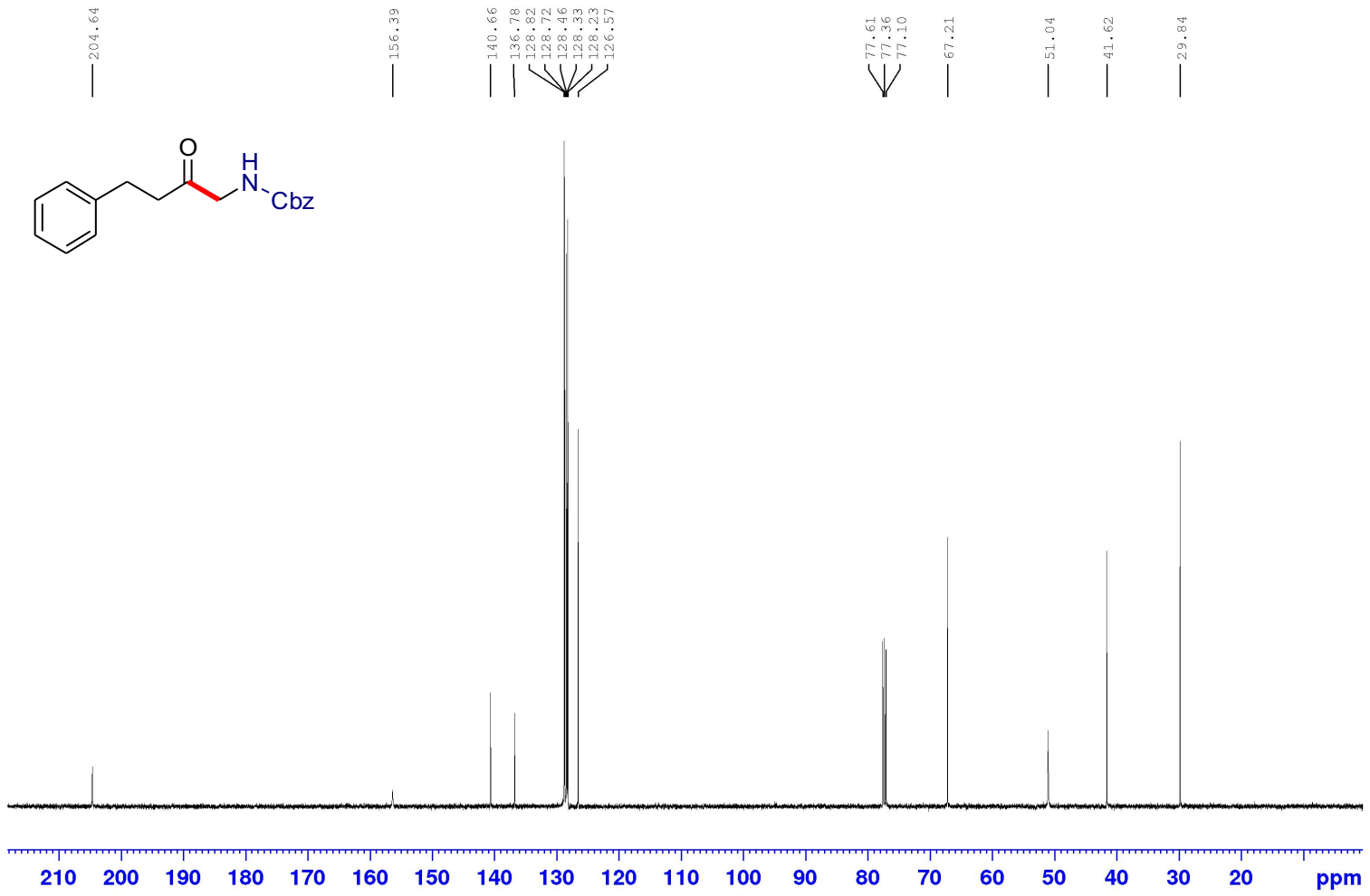
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of *tert*-butyl 4-(3-phenylpropanoyl)piperidine-1-carboxylate (2k)



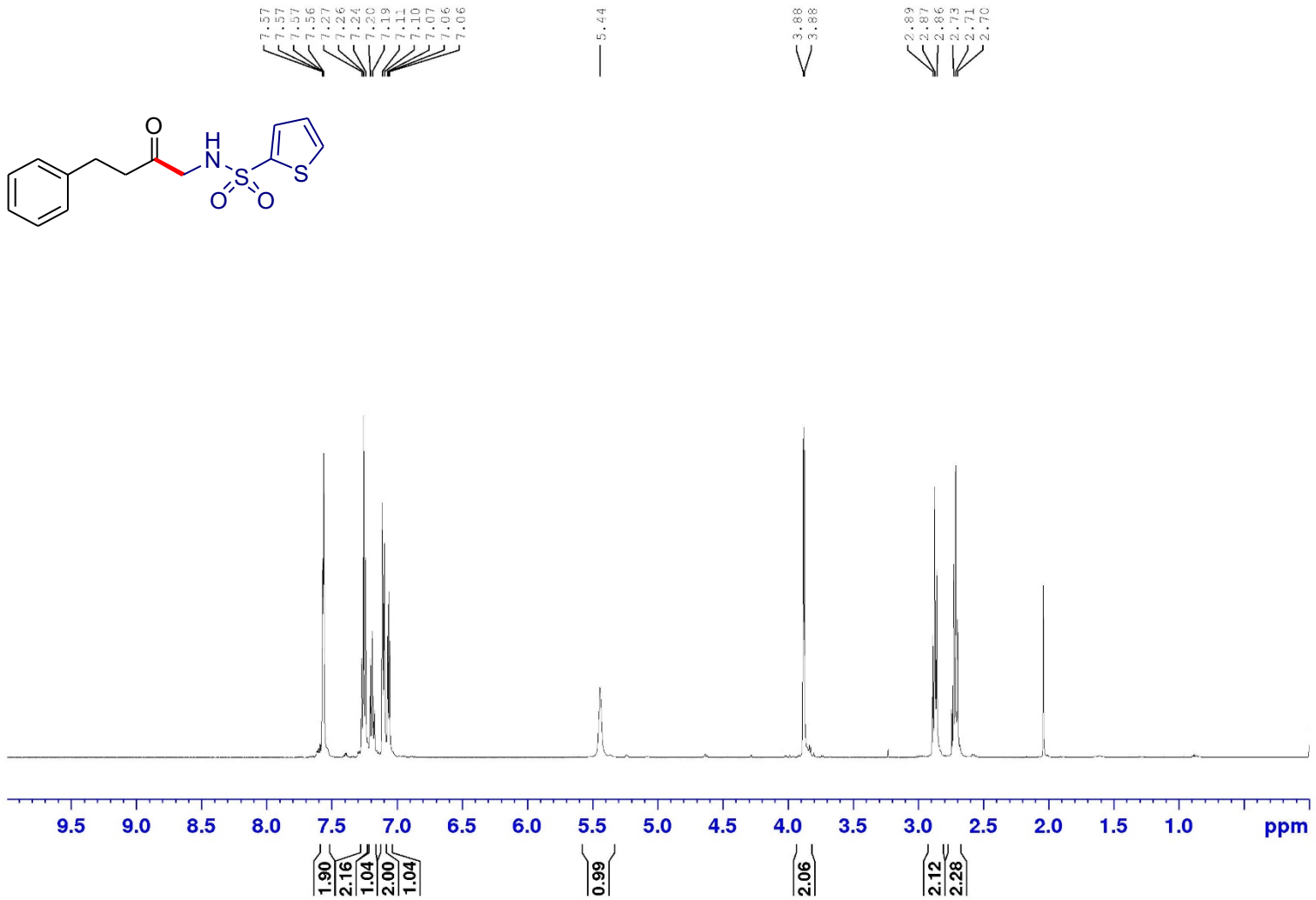
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of *tert*-butyl 4-(3-phenylpropanoyl)piperidine-1-carboxylate (2k)



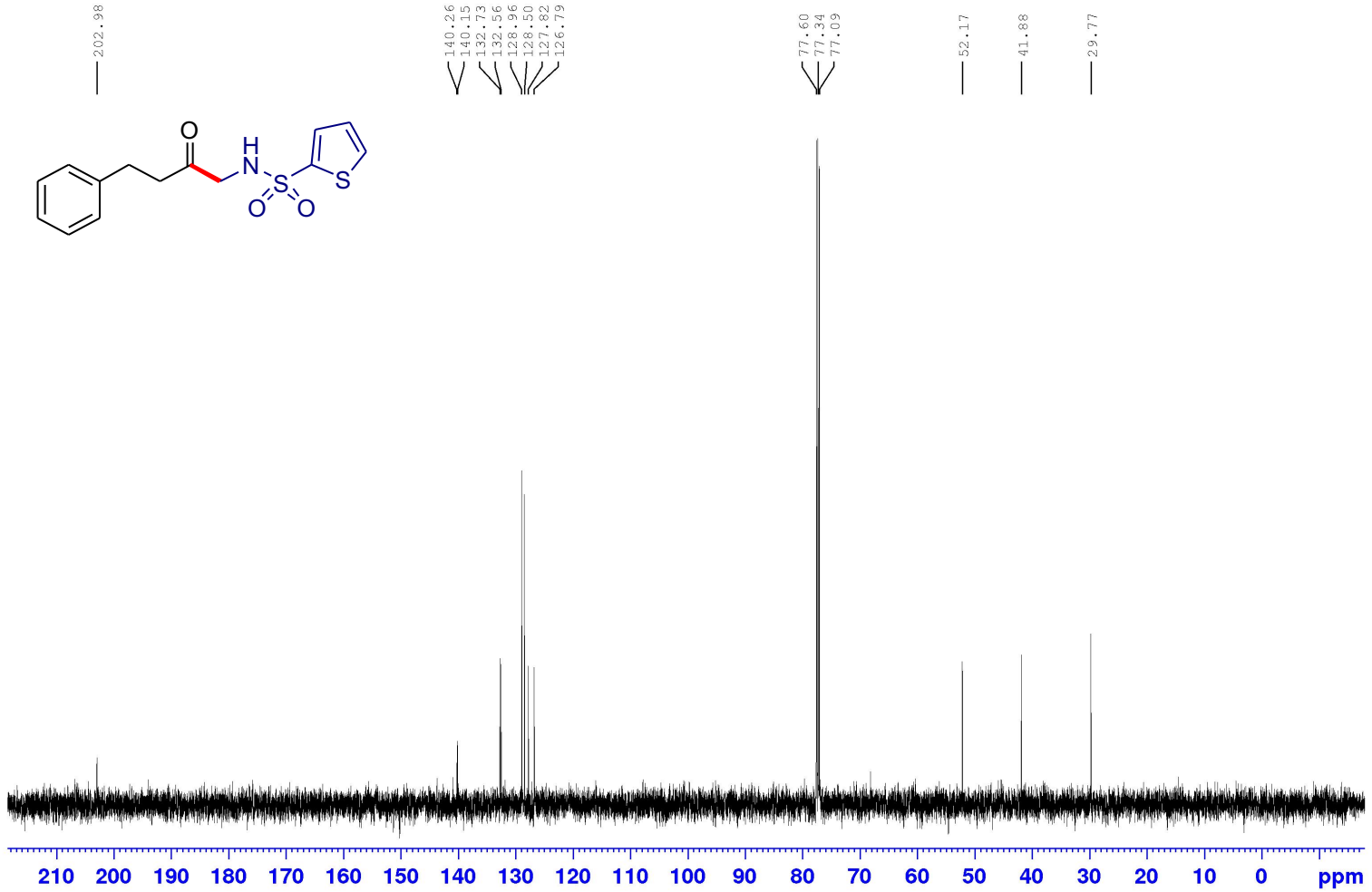
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of benzyl (2-oxo-4-phenylbutyl)carbamate (2l)



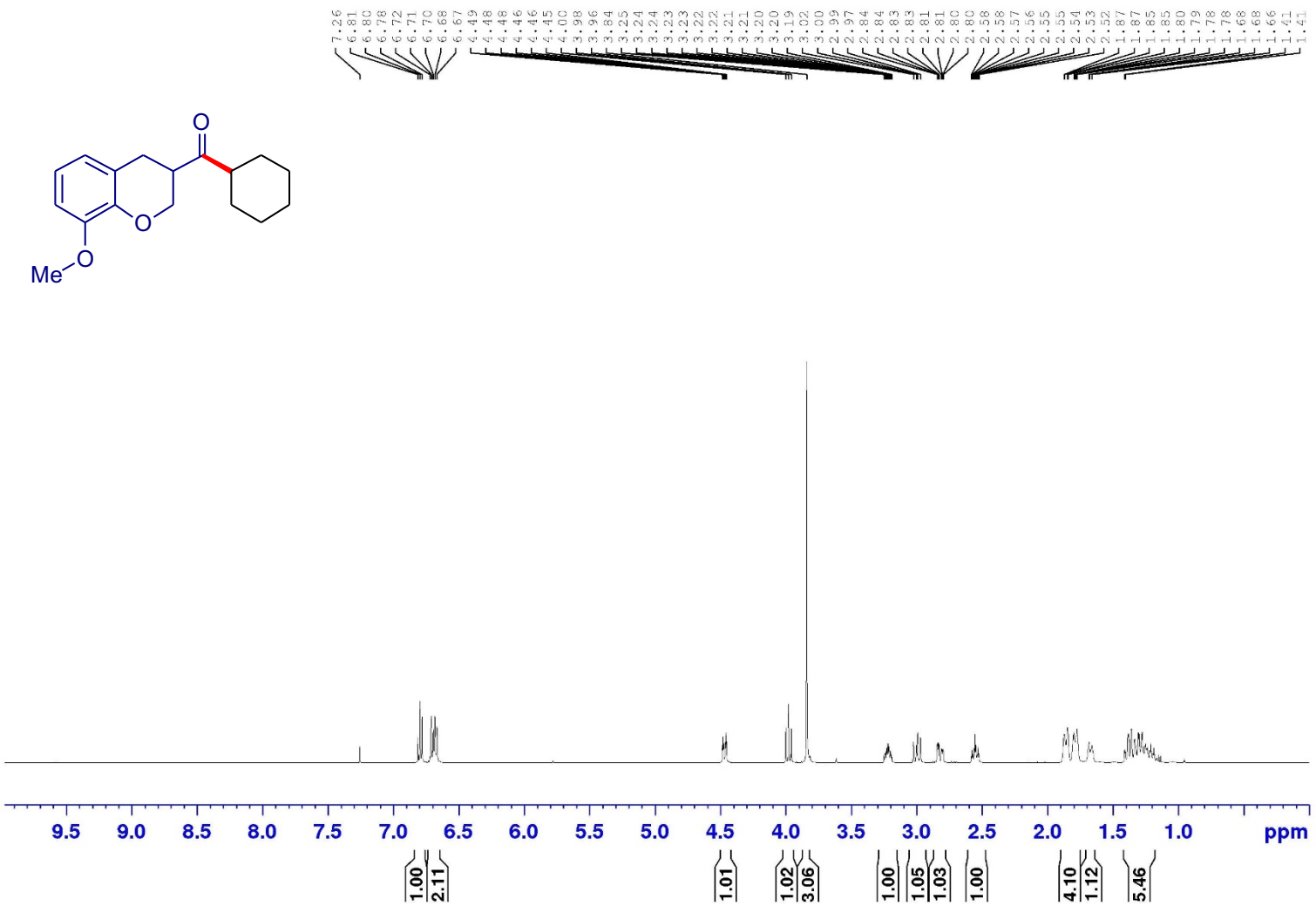
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of benzyl (2-oxo-4-phenylbutyl)carbamate (2l)



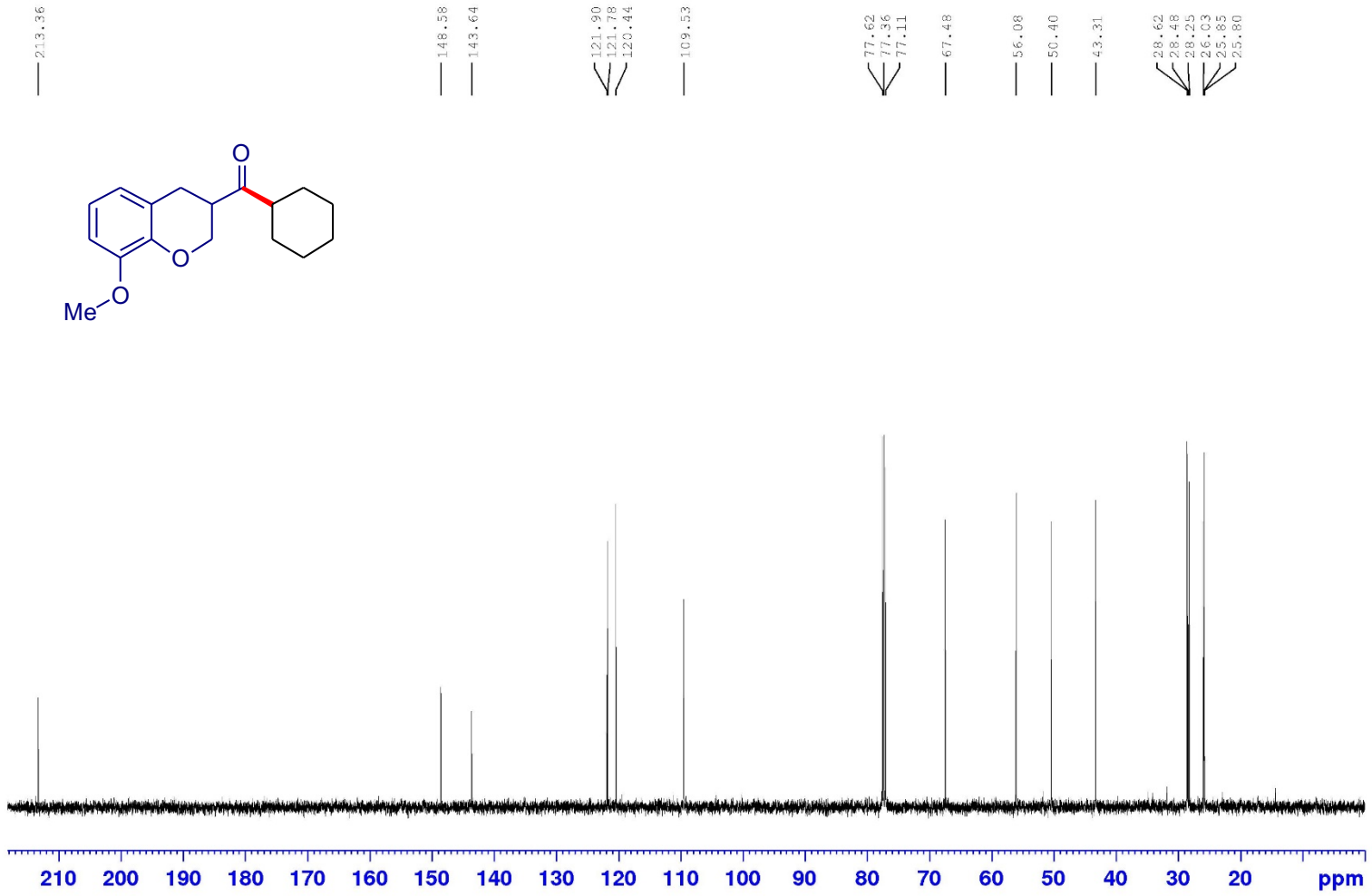
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of *N*-(2-oxo-4-phenylbutyl)thiophene-2-sulfonamide (2m)



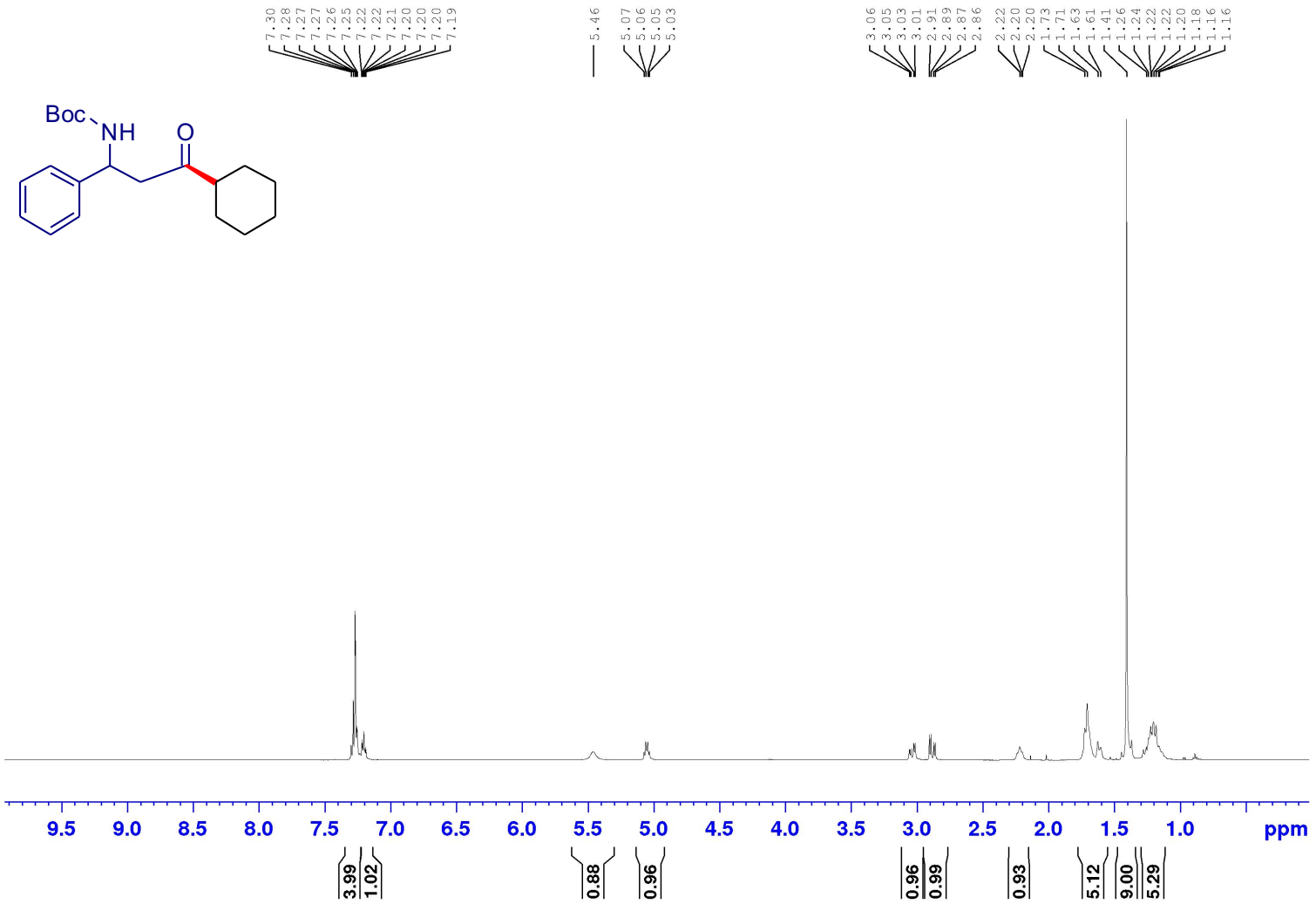
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of *N*-(2-oxo-4-phenylbutyl)thiophene-2-sulfonamide (2m)



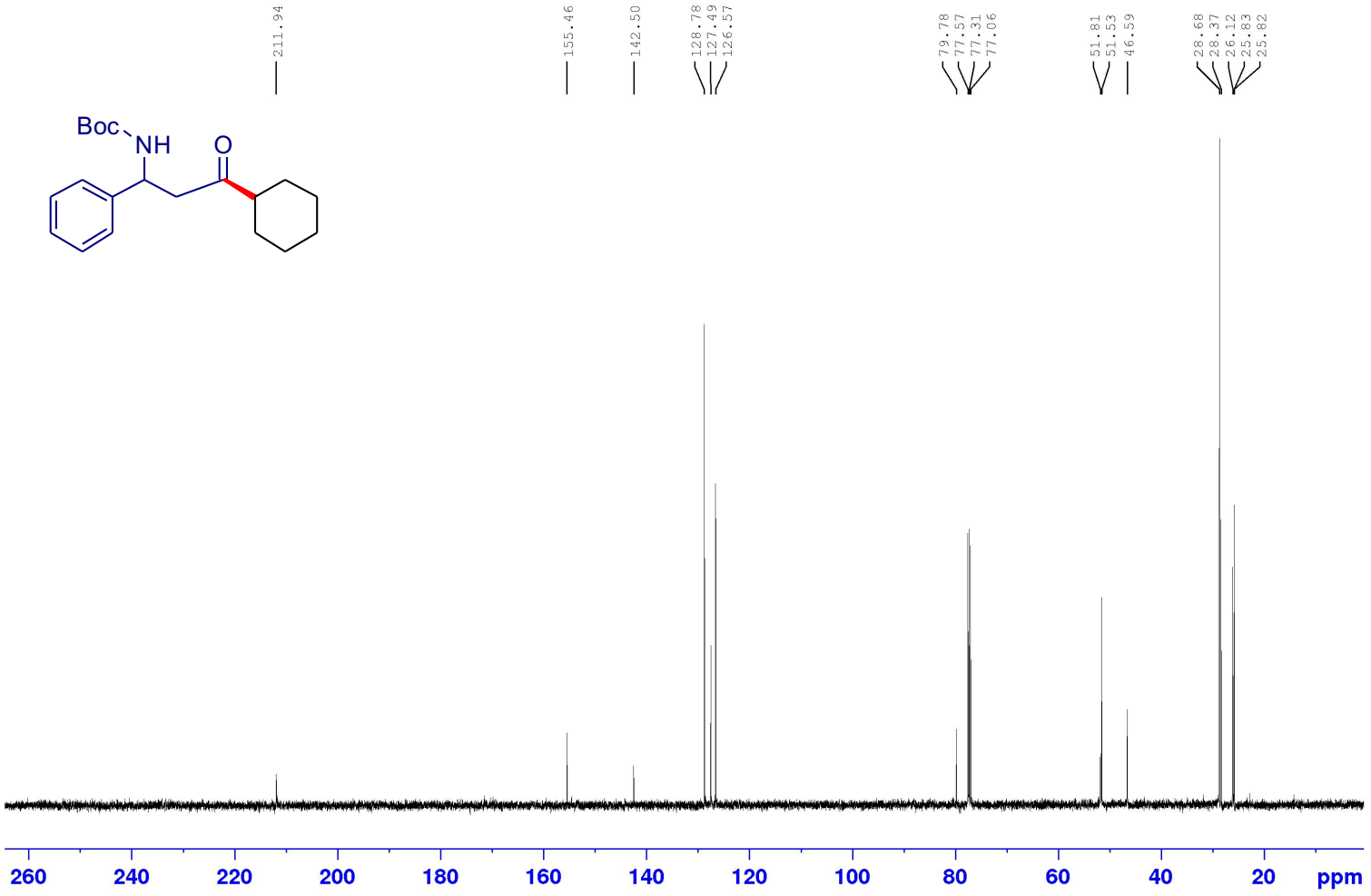
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of cyclohexyl(8-methoxychroman-3-yl)methanone (3a)



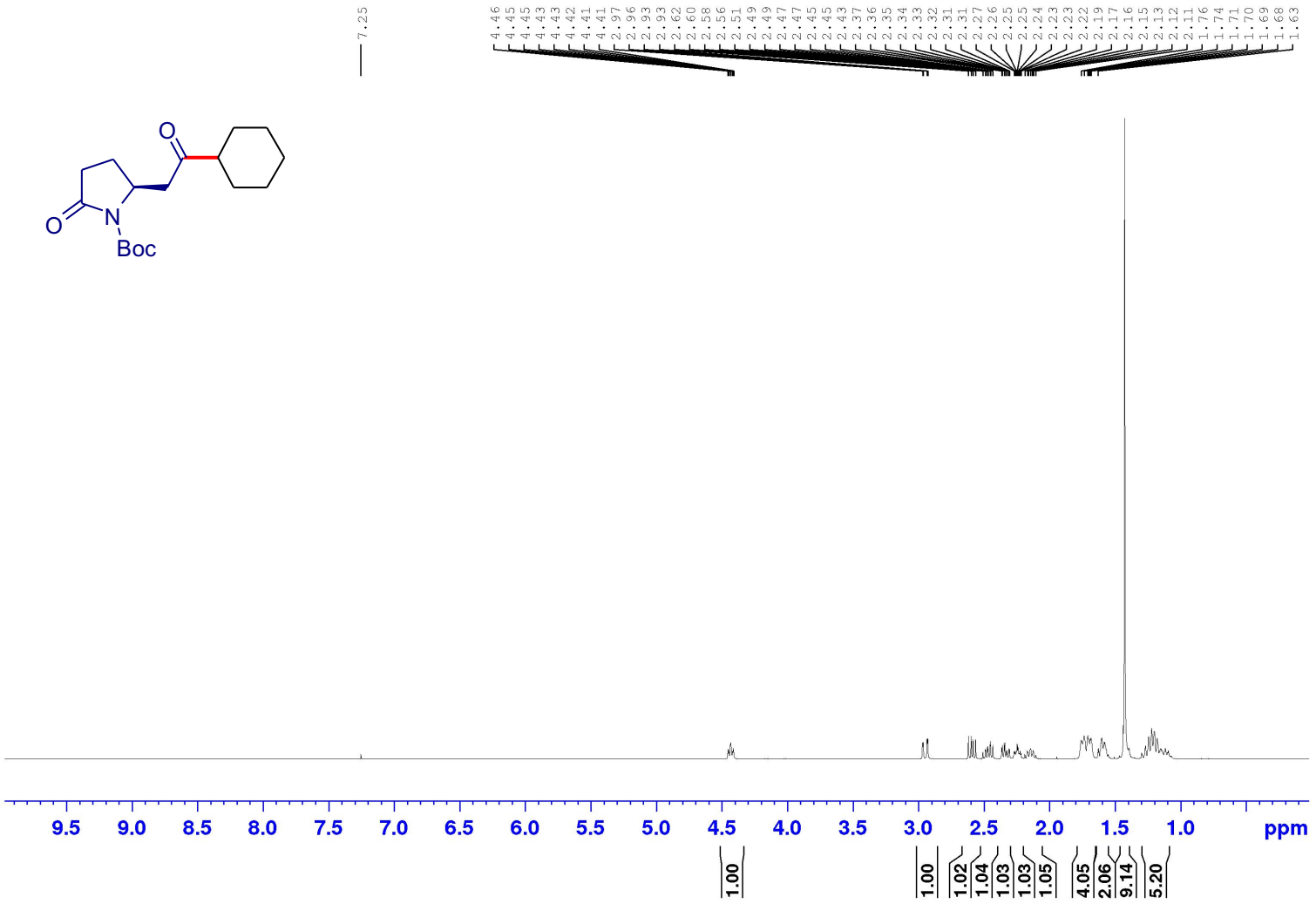
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of cyclohexyl(8-methoxychroman-3-yl)methanone (3a)



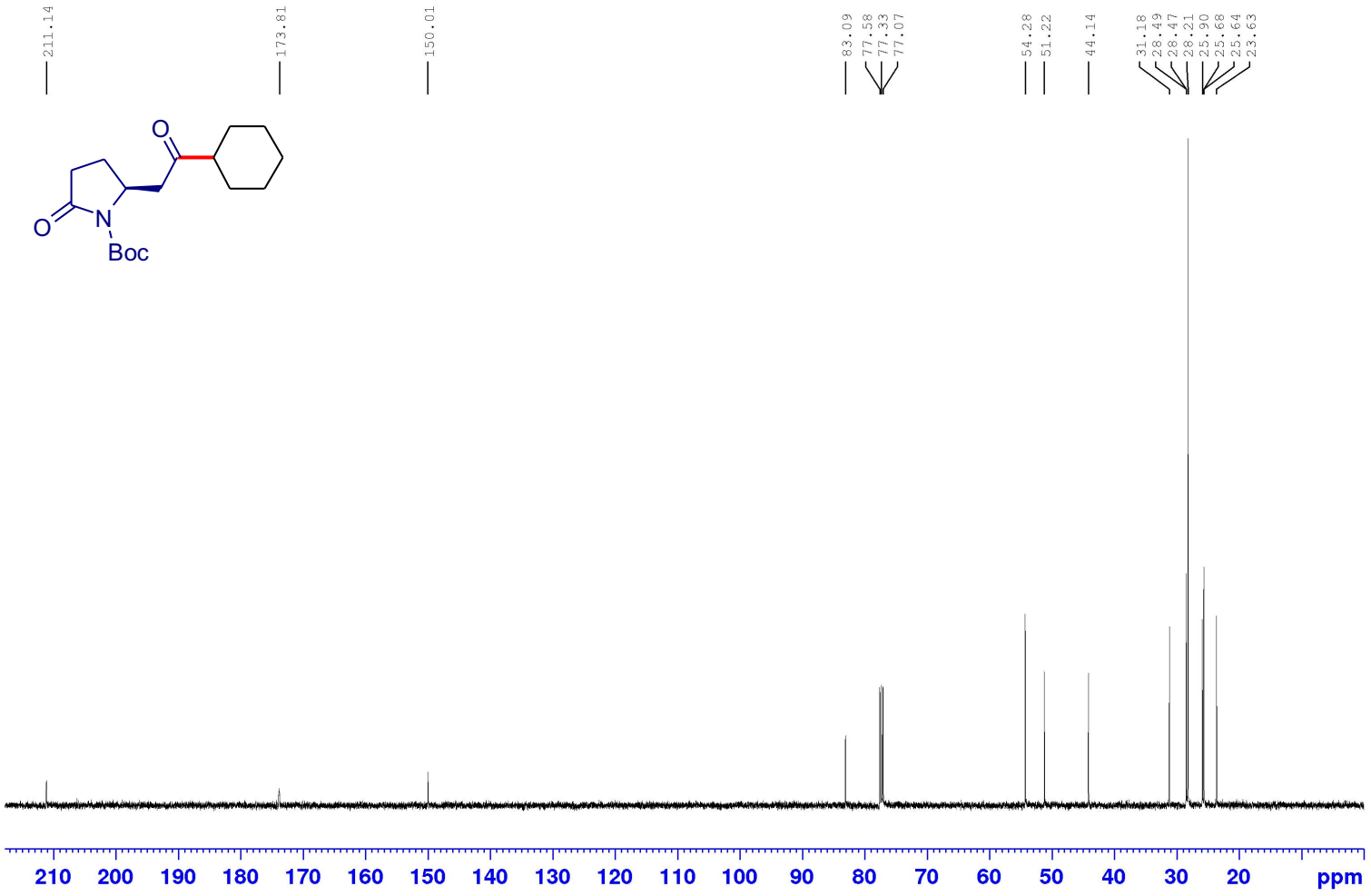
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of *tert*-butyl -(3-cyclohexyl-3-oxo-1-phenylpropyl)carbamate (3b)



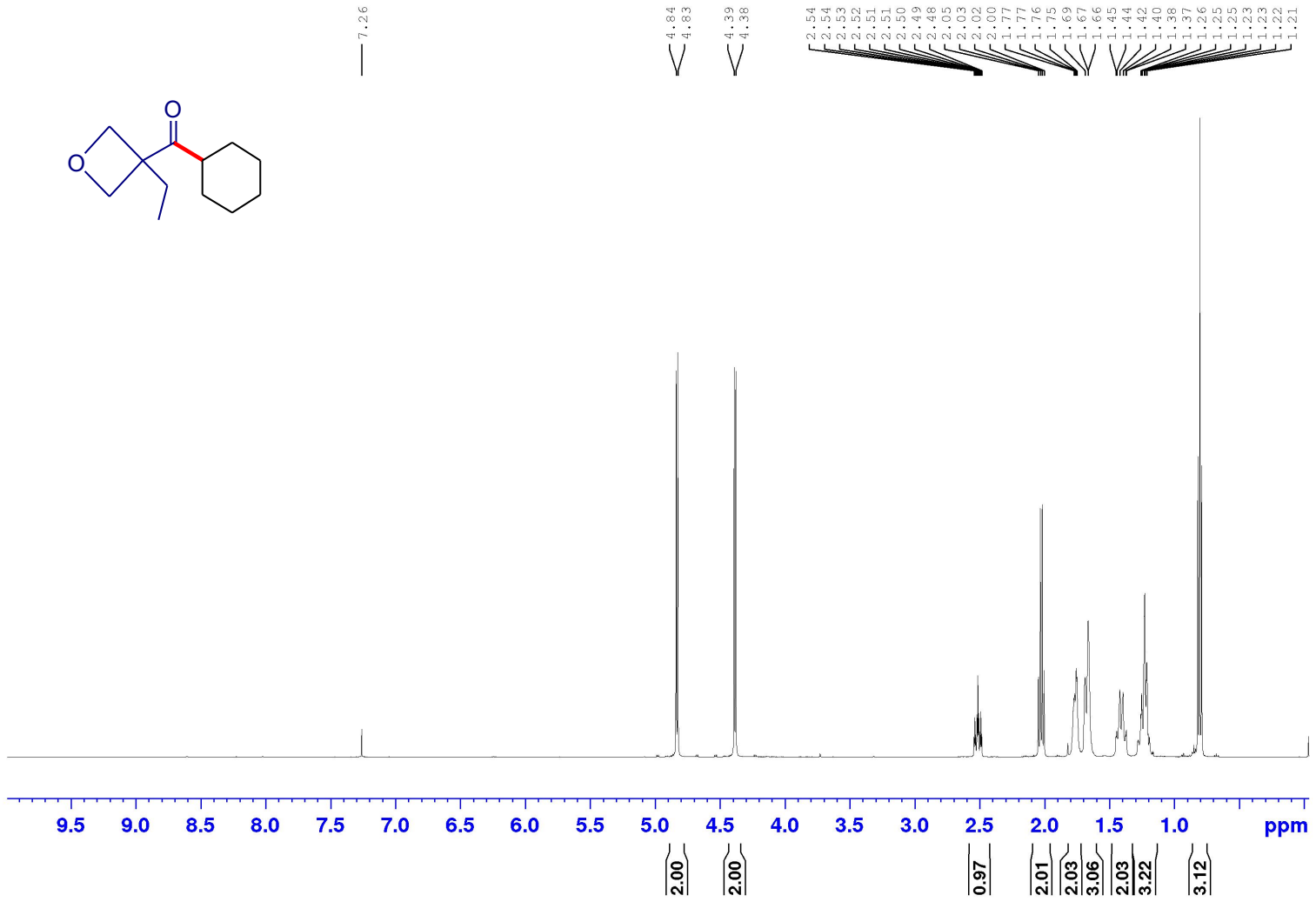
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of *tert*-butyl -(3-cyclohexyl-3-oxo-1-phenylpropyl)carbamate (3b)



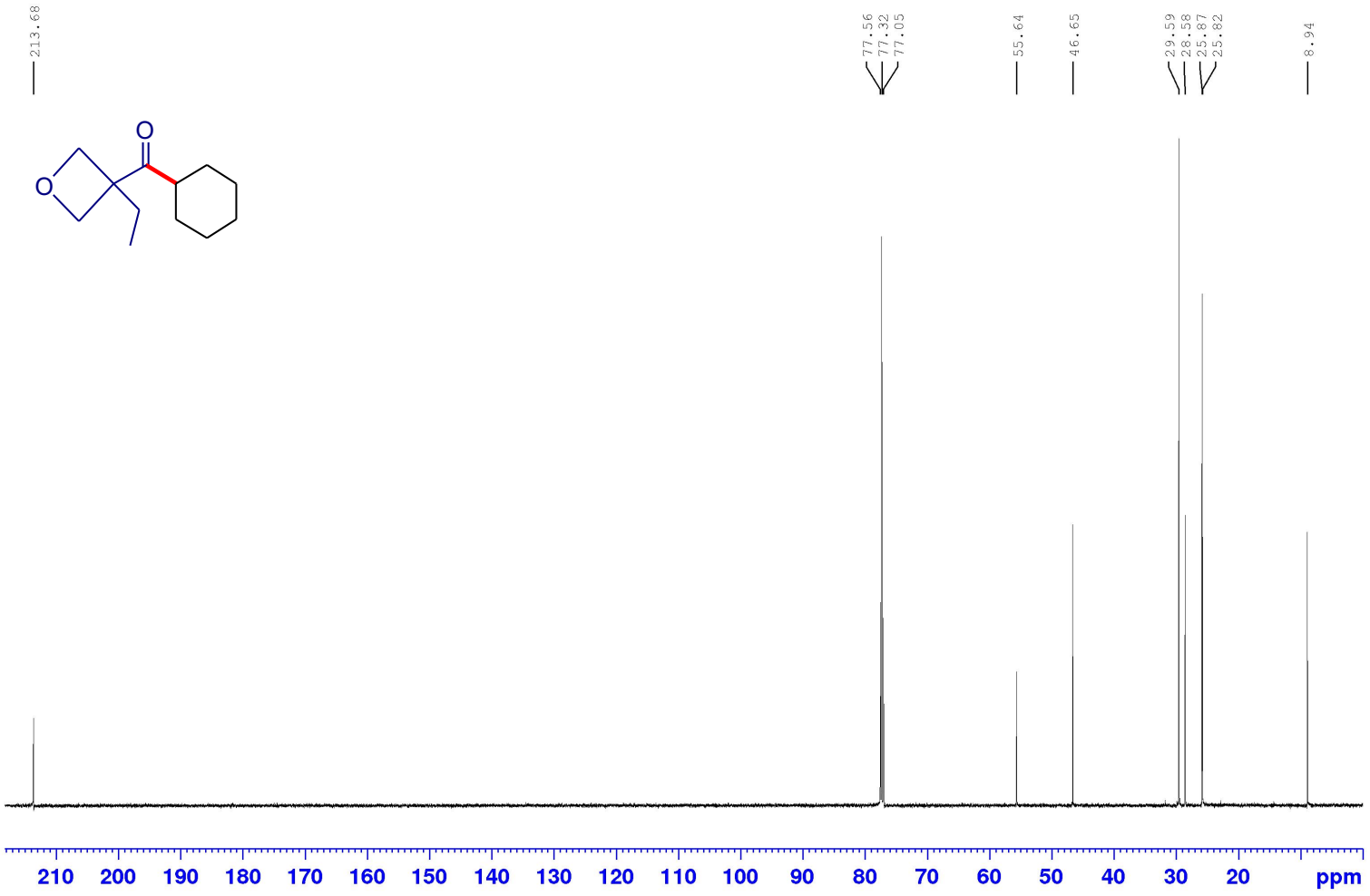
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 5-(2-cyclohexyl-2-oxoethyl)pyrrolidin-2-one (3c)



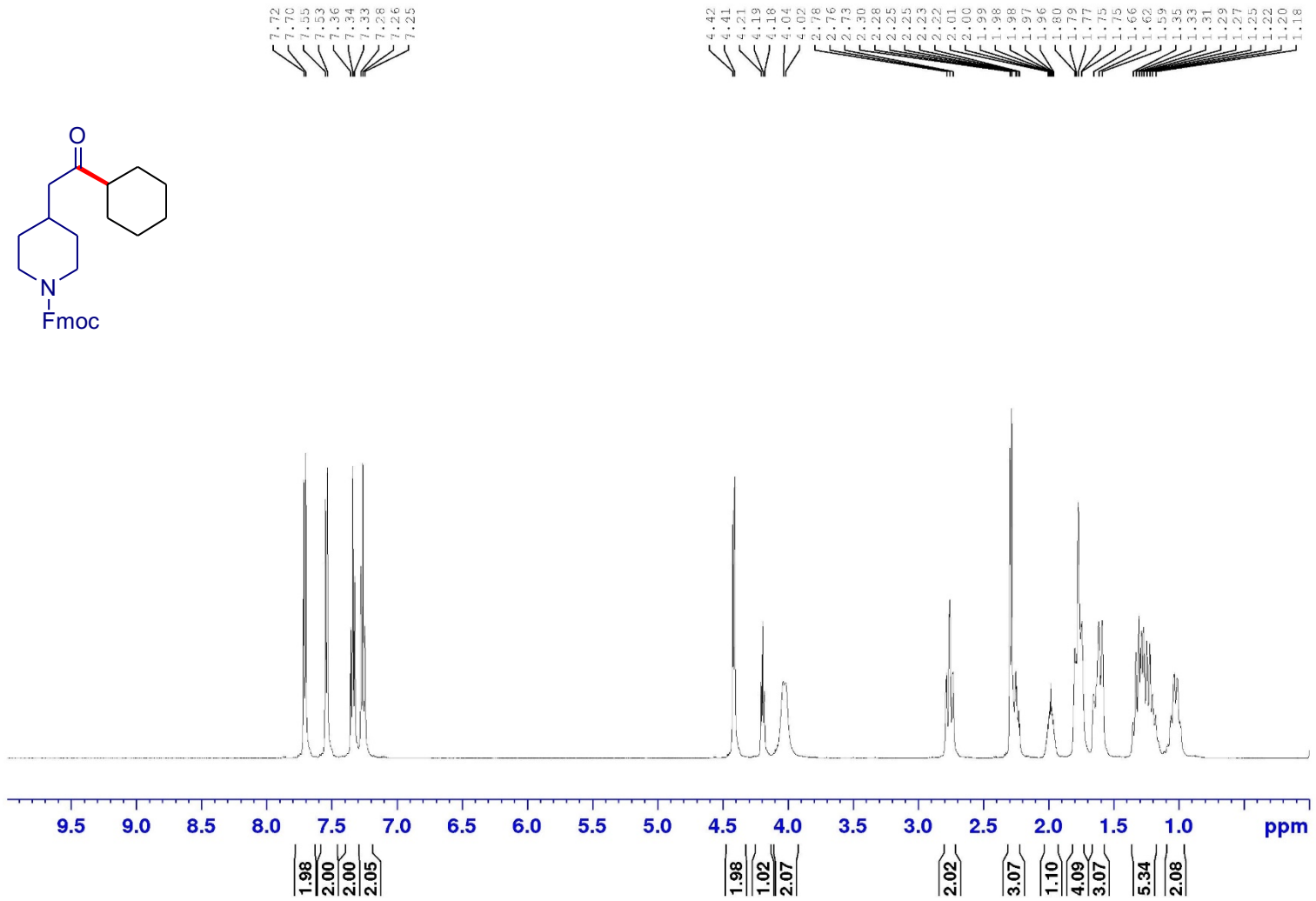
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of 5-(2-cyclohexyl-2-oxoethyl)pyrrolidin-2-one (3c)



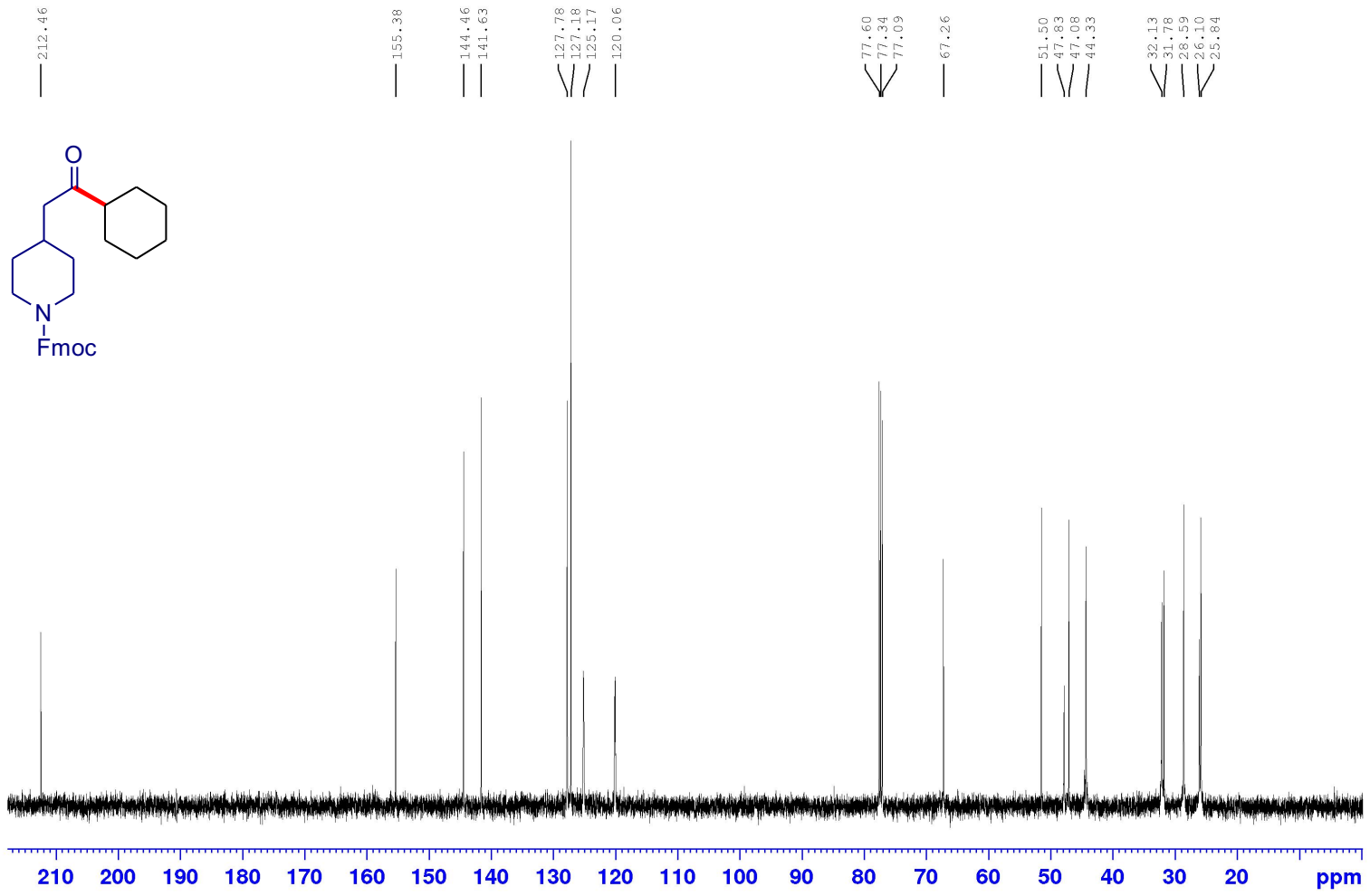
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of cyclohexyl(3-ethyloxetan-3-yl)methanone (3d)



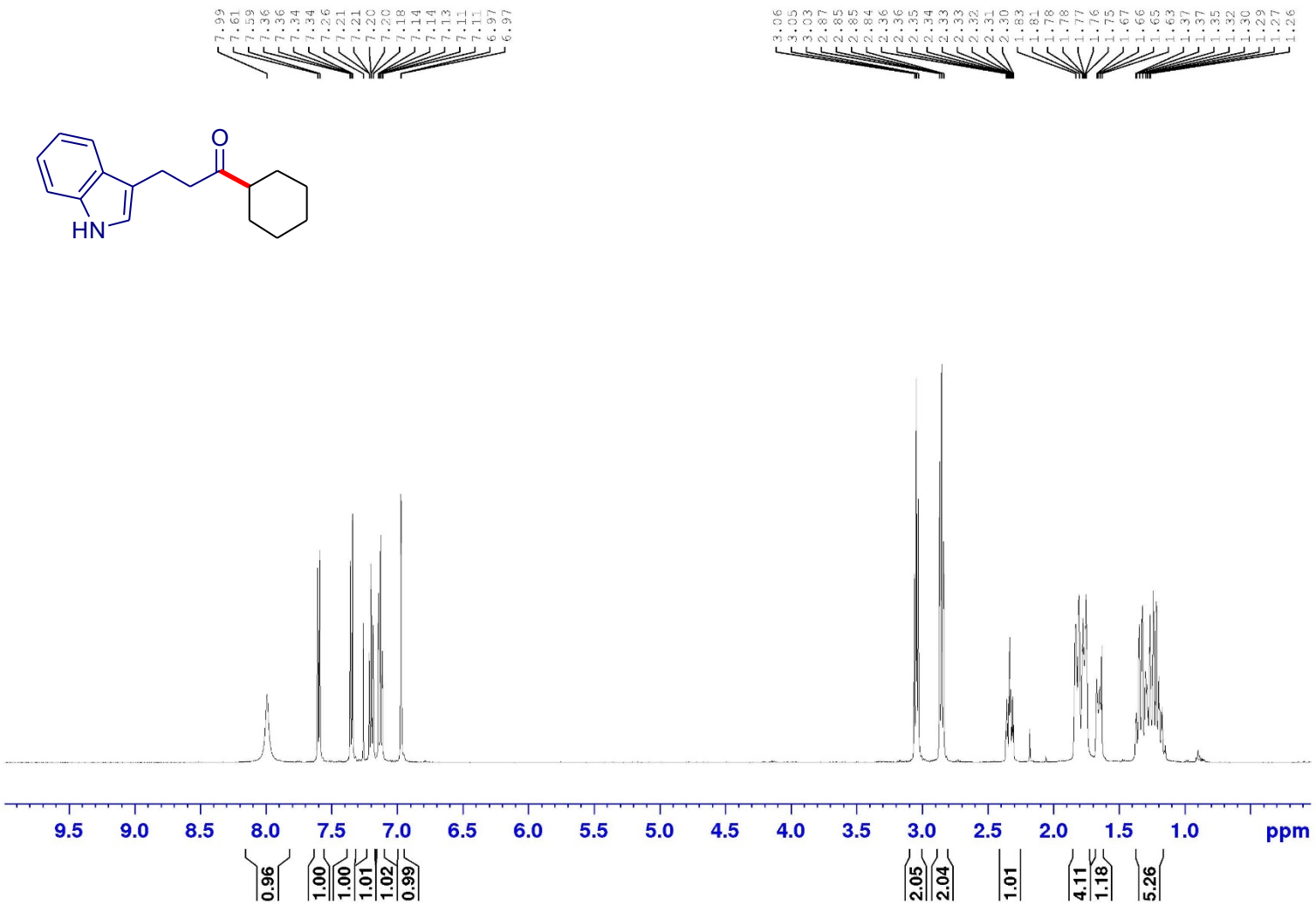
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of cyclohexyl(3-ethyloxetan-3-yl)methanone (3d)



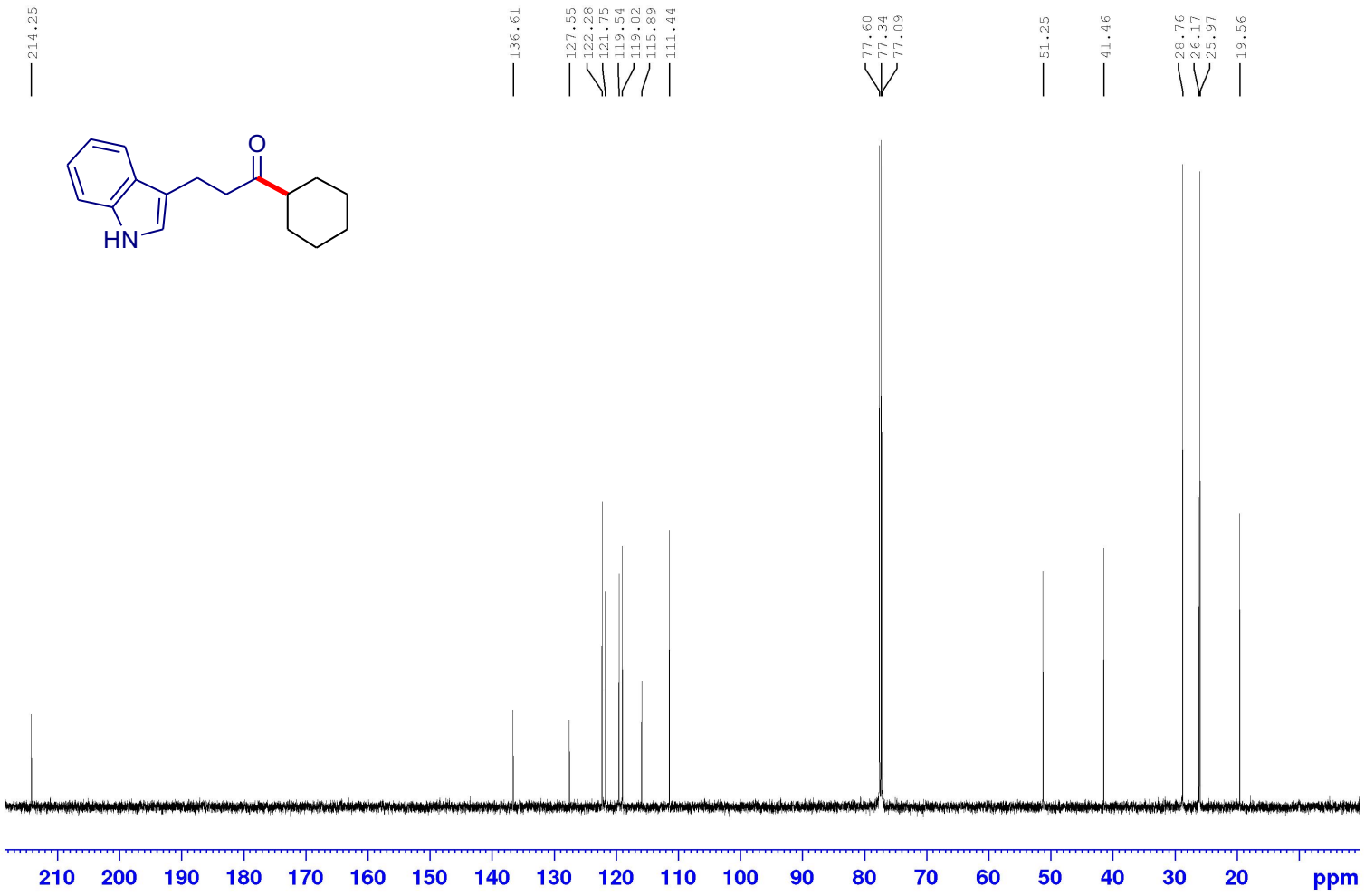
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of (9H-fluoren-9-yl)methyl 4-(2-cyclohexyl-2-oxoethyl)piperidine-1-carboxylate (3e)



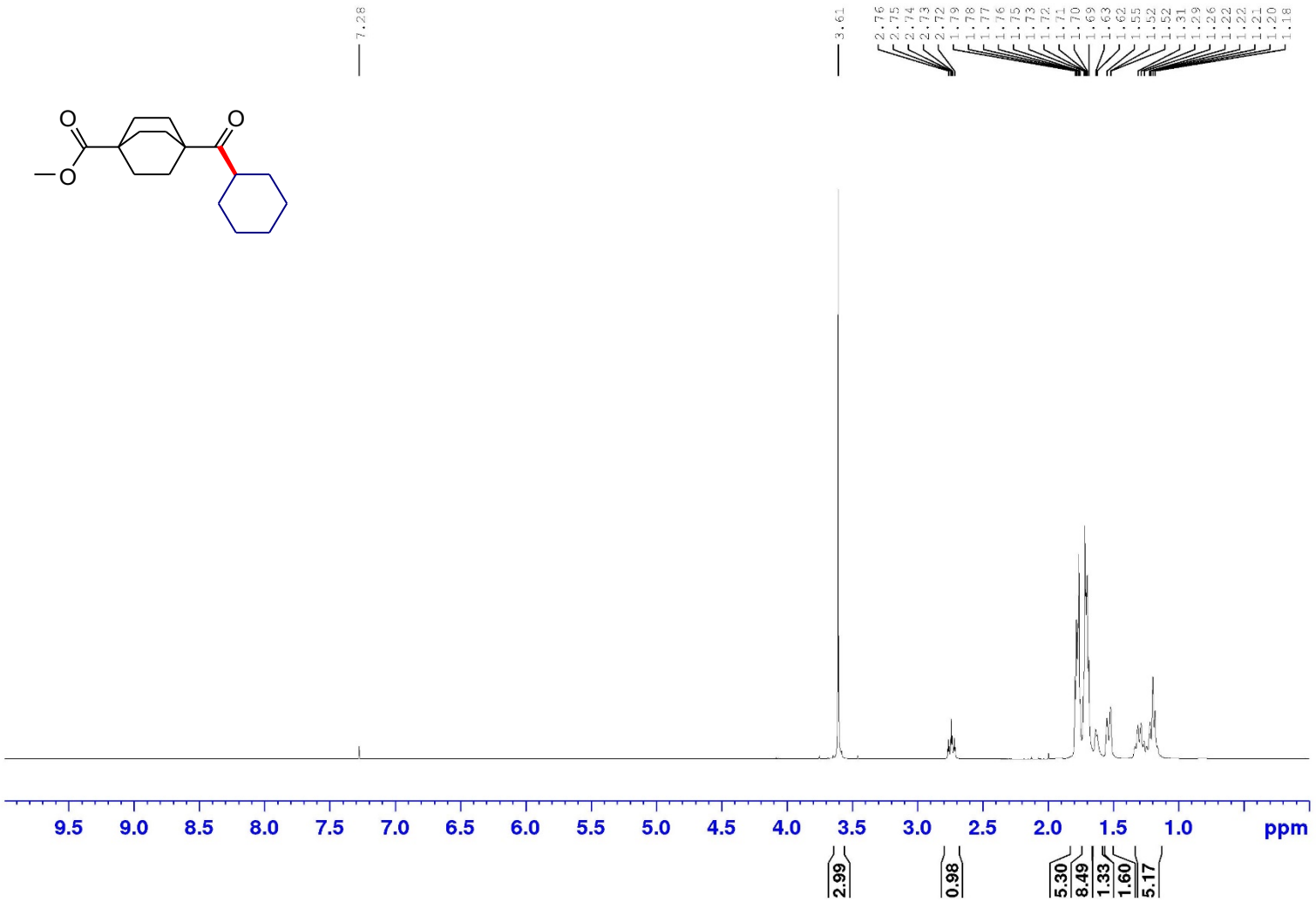
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of (9H-fluoren-9-yl)methyl 4-(2-cyclohexyl-2-oxoethyl)piperidine-1-carboxylate (3e)



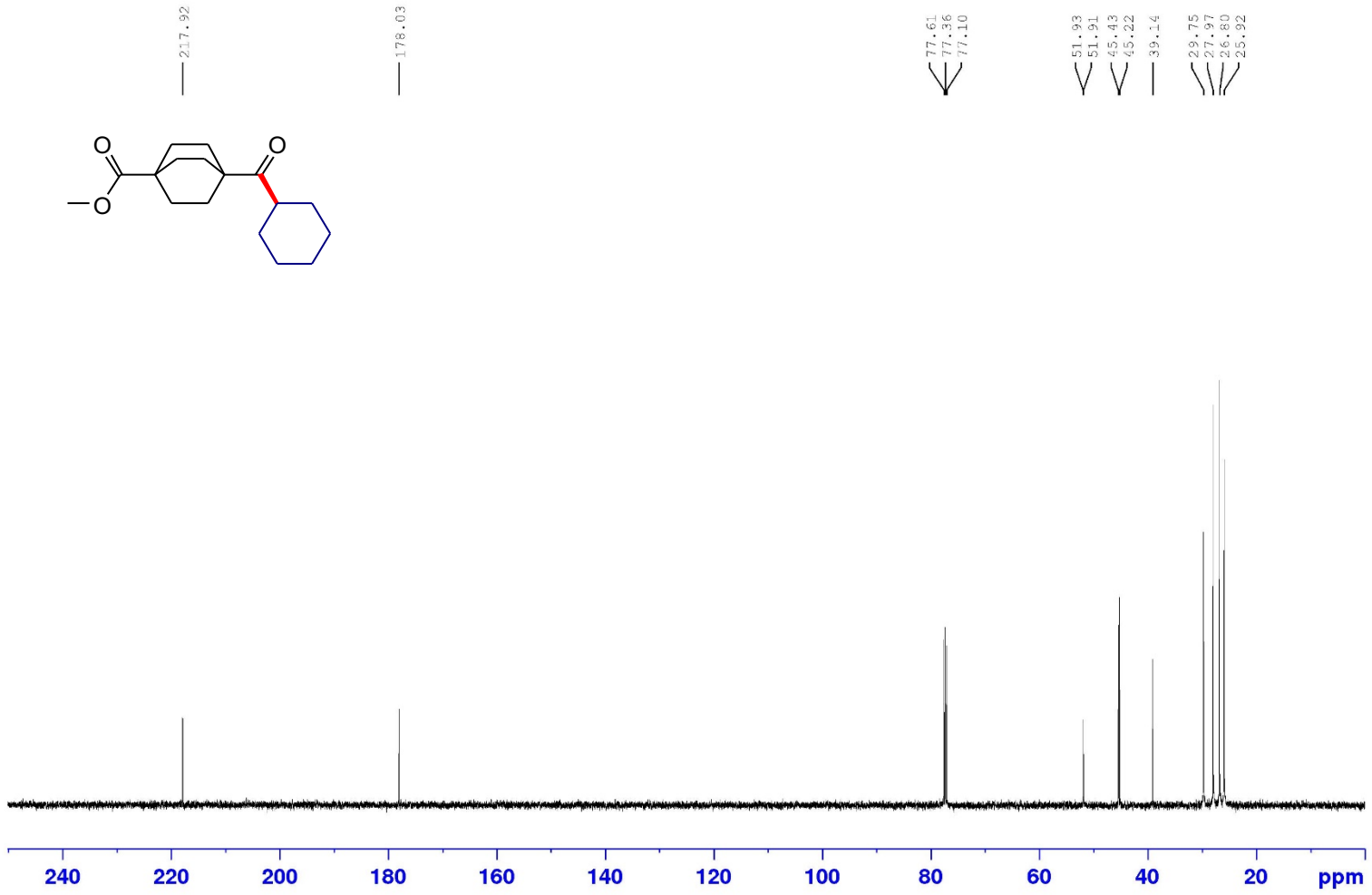
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 1-cyclohexyl-3-(1H-indol-3-yl)propan-1-one (3f)



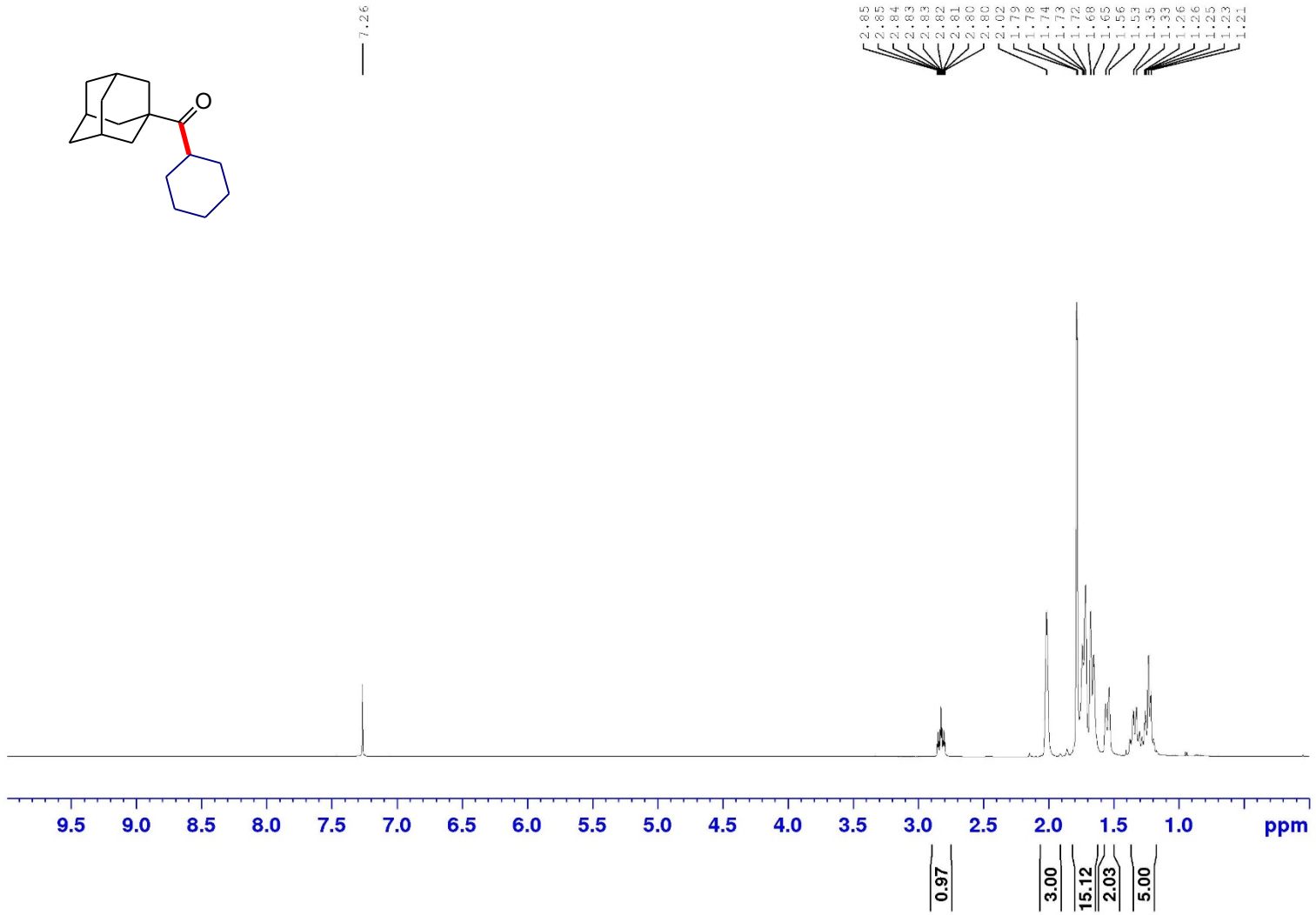
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of 1-cyclohexyl-3-(1H-indol-3-yl)propan-1-one (3f)



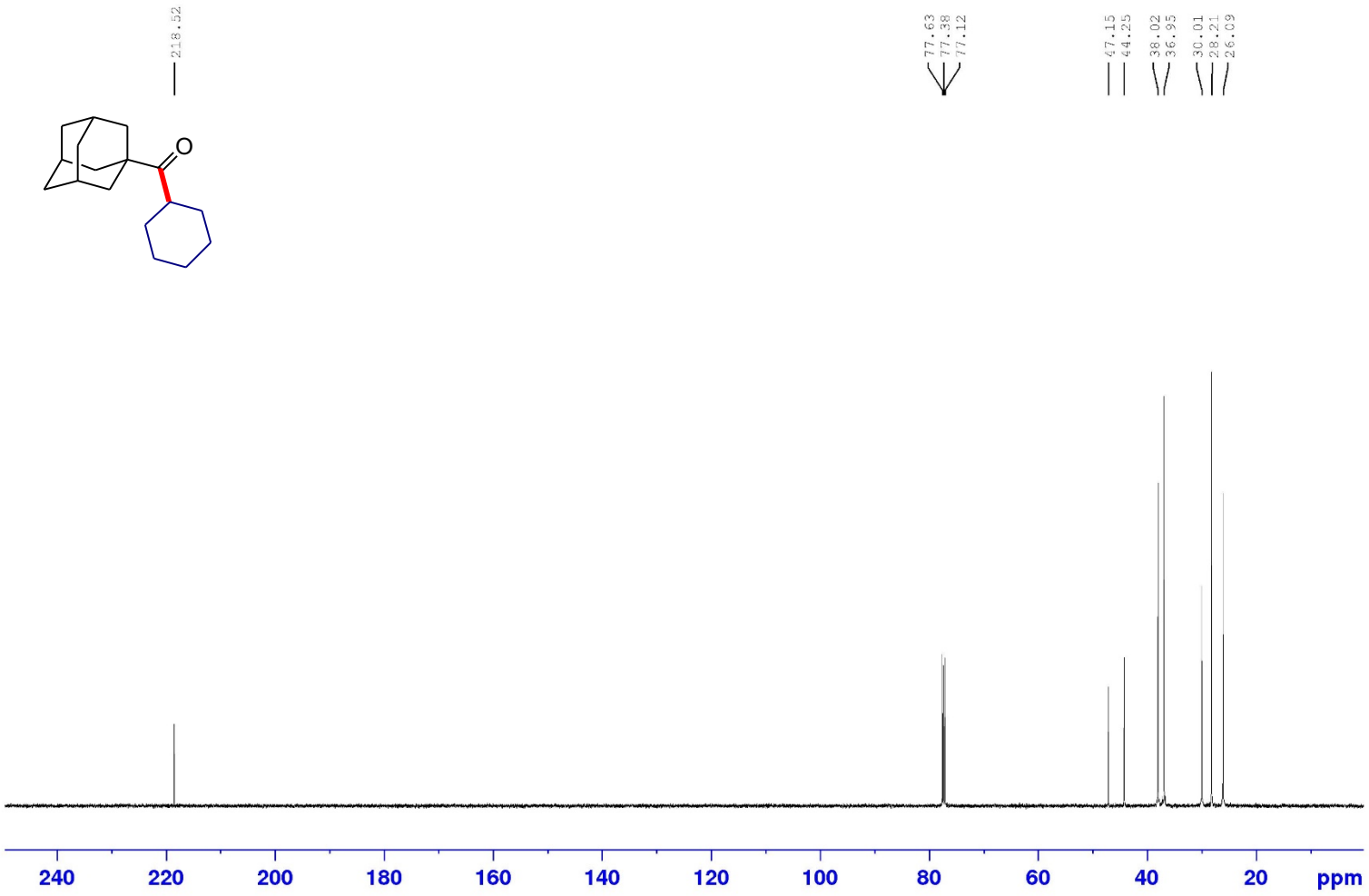
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of methyl 4-(cyclohexanecarbonyl)bicyclo[2.2.2]octane-1-carboxylate (3g)



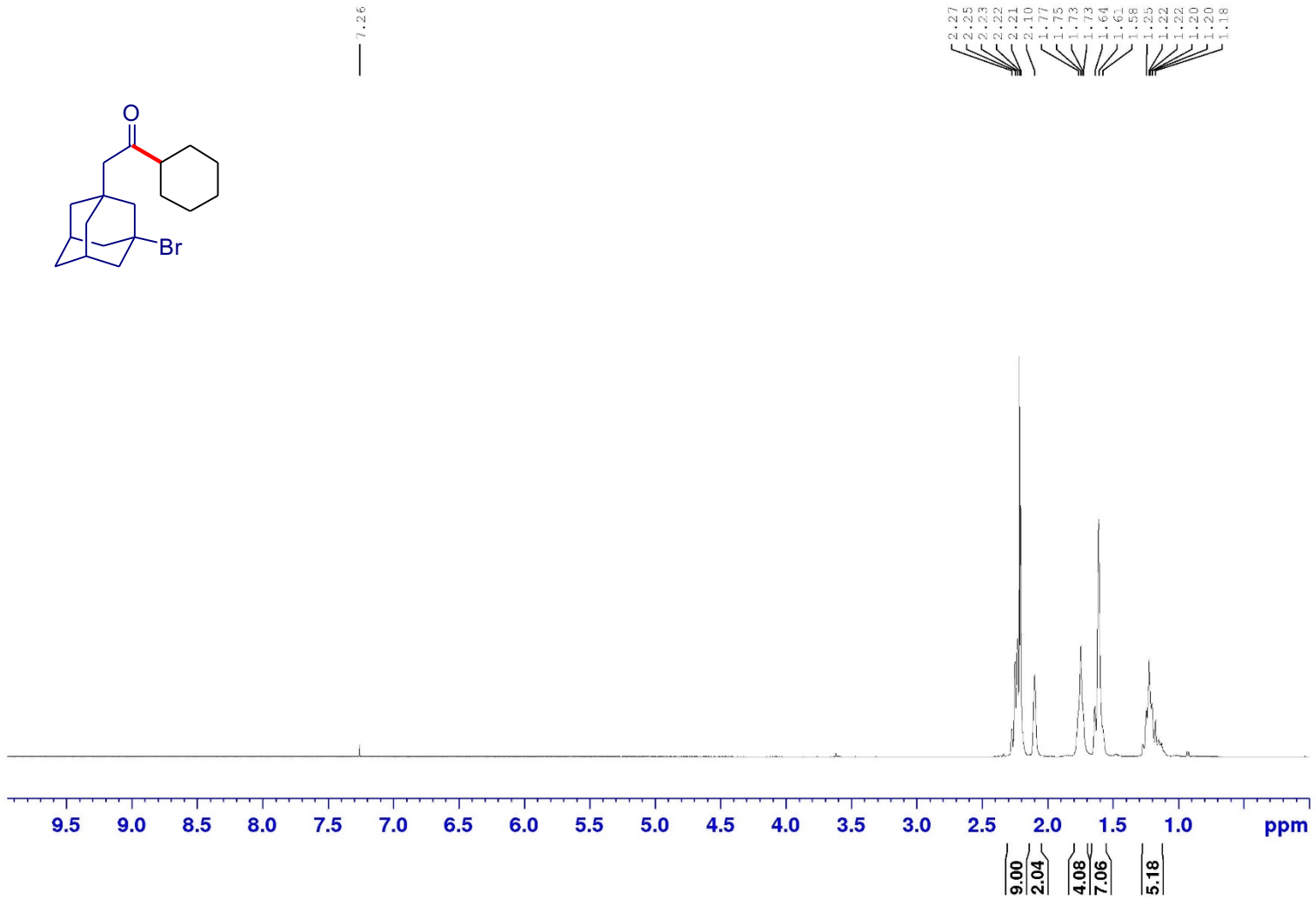
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of methyl 4-(cyclohexanecarbonyl)bicyclo[2.2.2]octane-1-carboxylate (3g)



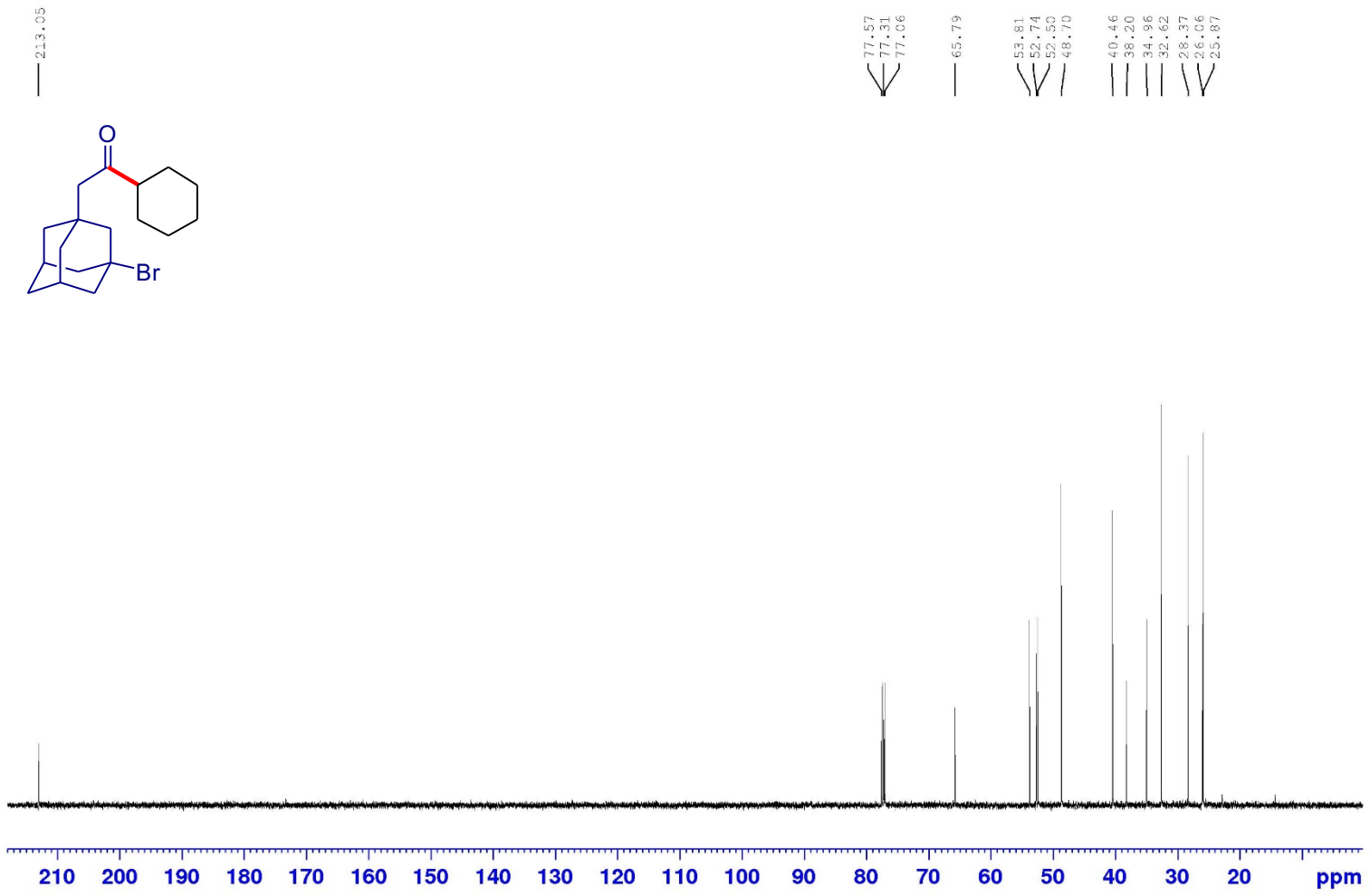
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of (adamantan-1-yl)(cyclohexyl)methanone (3h)



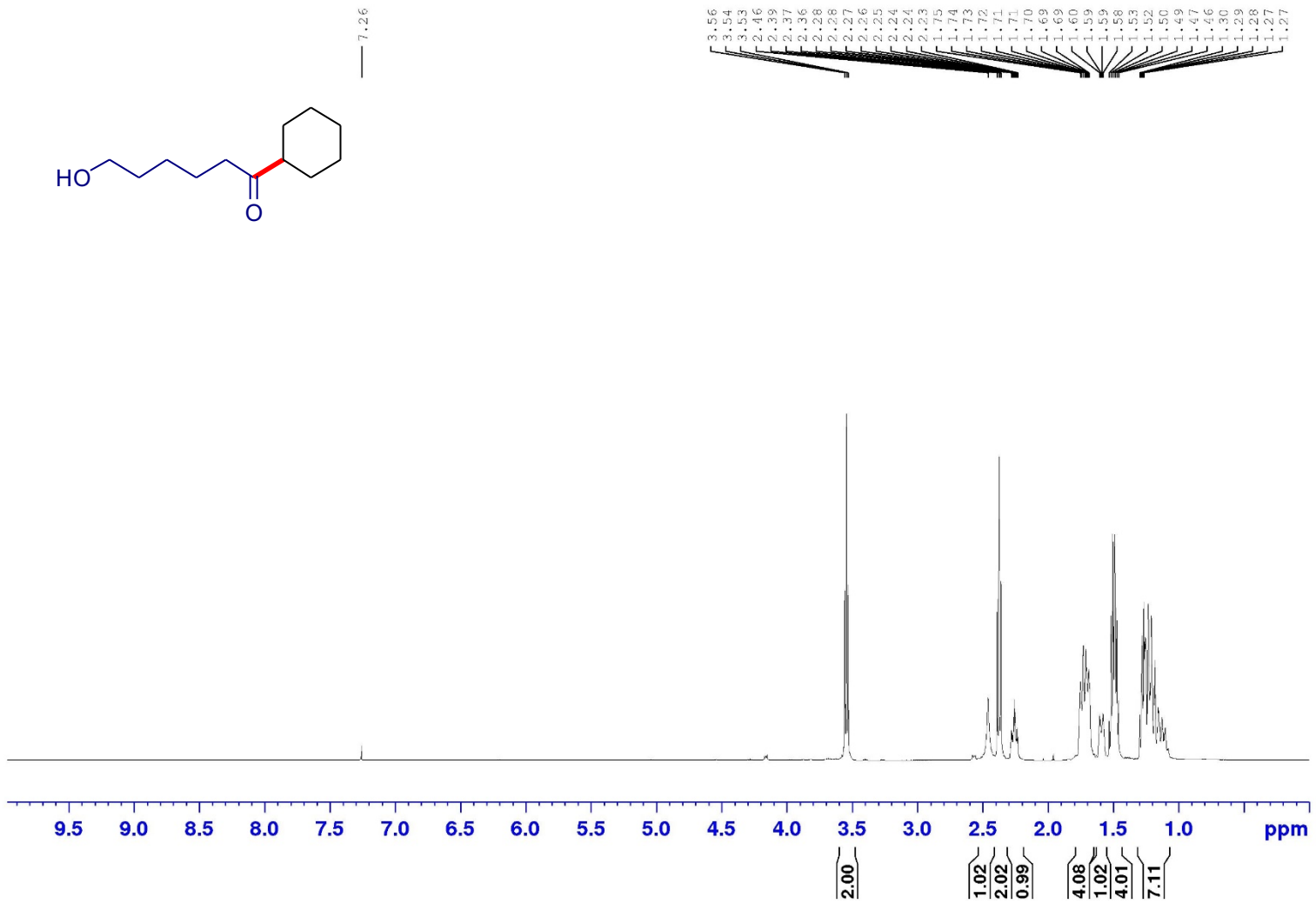
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of (adamantan-1-yl)(cyclohexyl)methanone (3h)



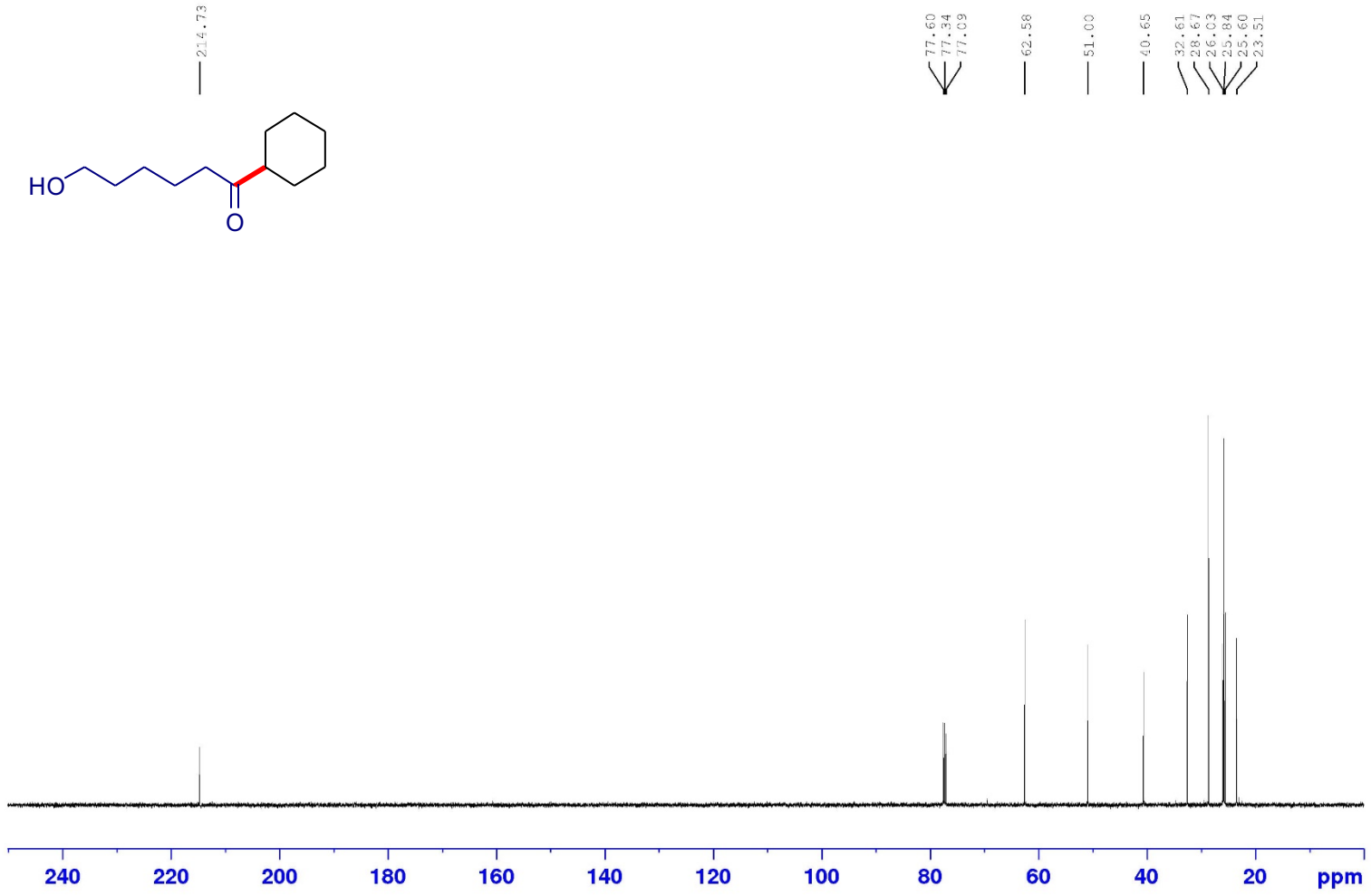
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 2-(3-bromoadamantan-1-yl)-1-cyclohexylethan-1-one (3i)



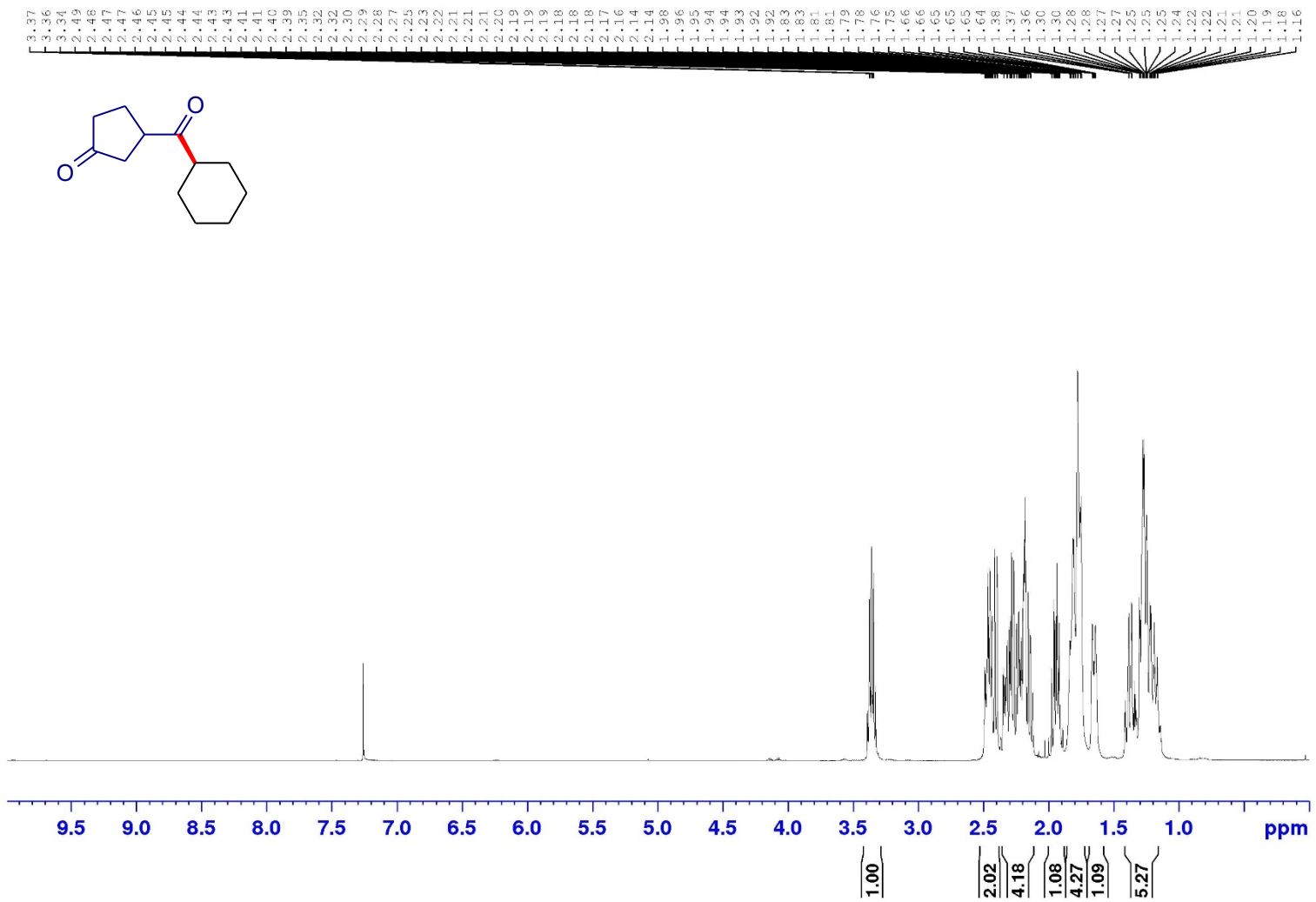
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of 2-(3-bromoadamantan-1-yl)-1-cyclohexylethan-1-one (3i)



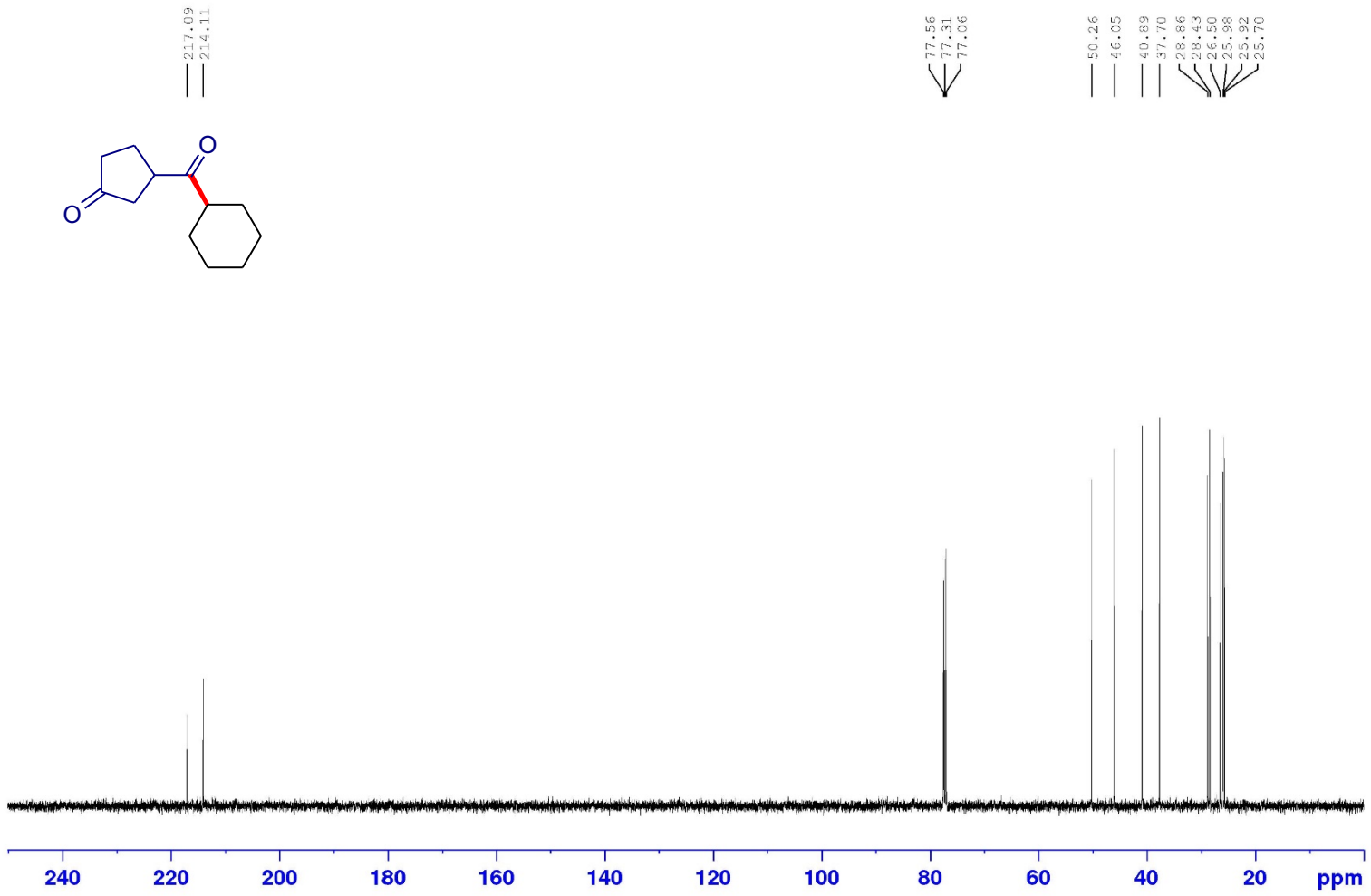
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 1-cyclohexyl-6-hydroxyhexan-1-one (3j)



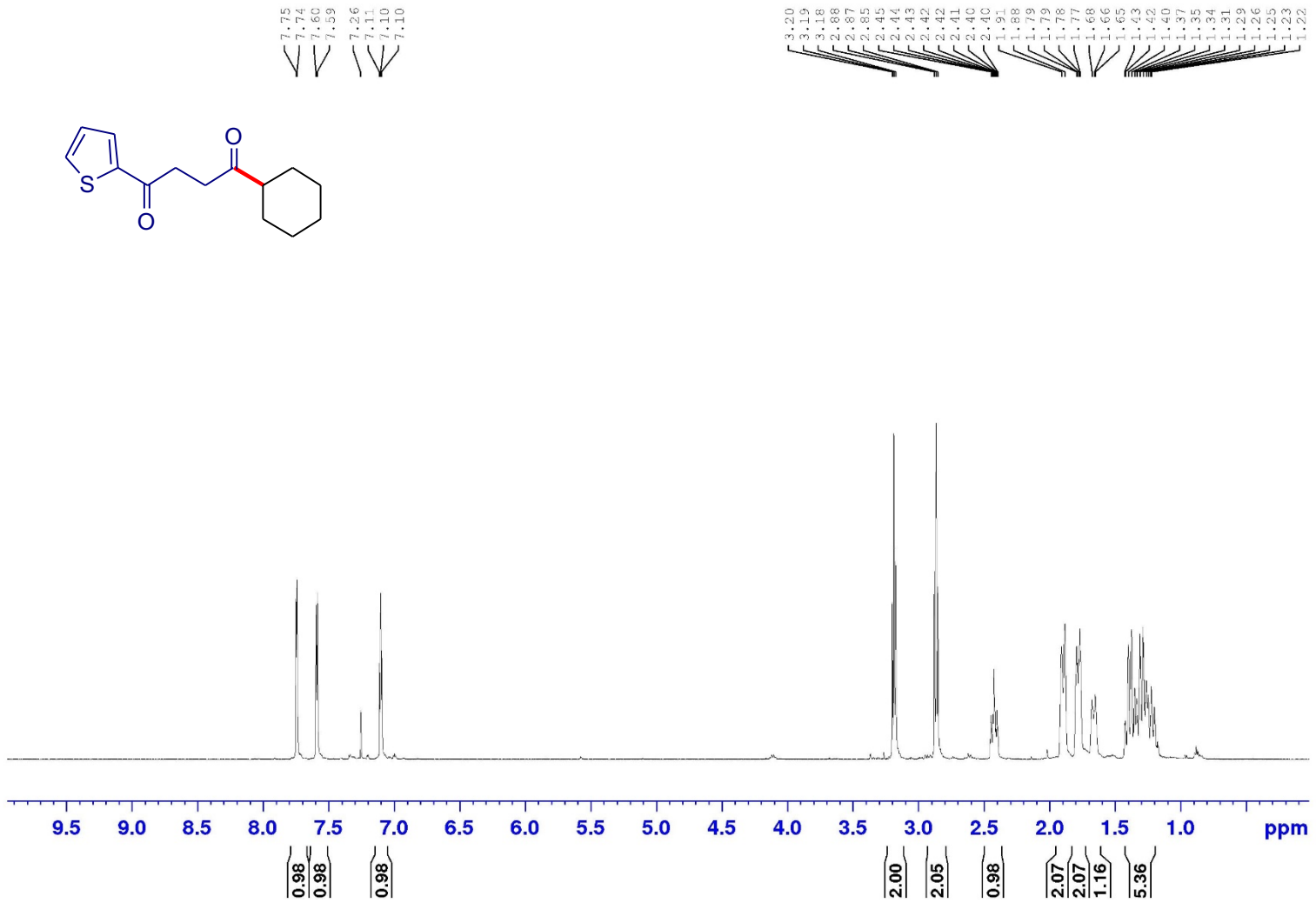
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of 1-cyclohexyl-6-hydroxyhexan-1-one (3j)



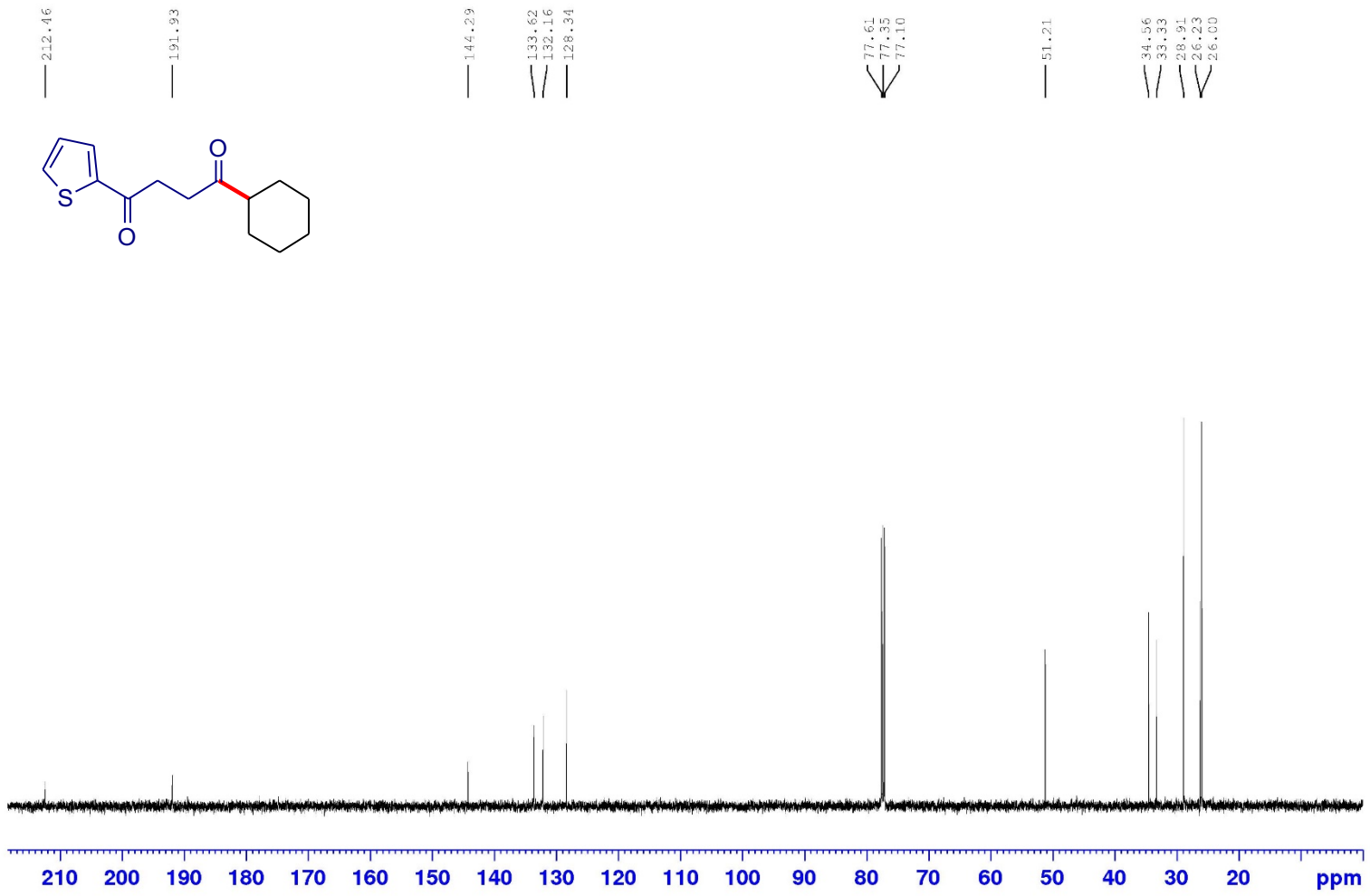
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 3-(cyclohexanecarbonyl)cyclopentan-1-one (3k)



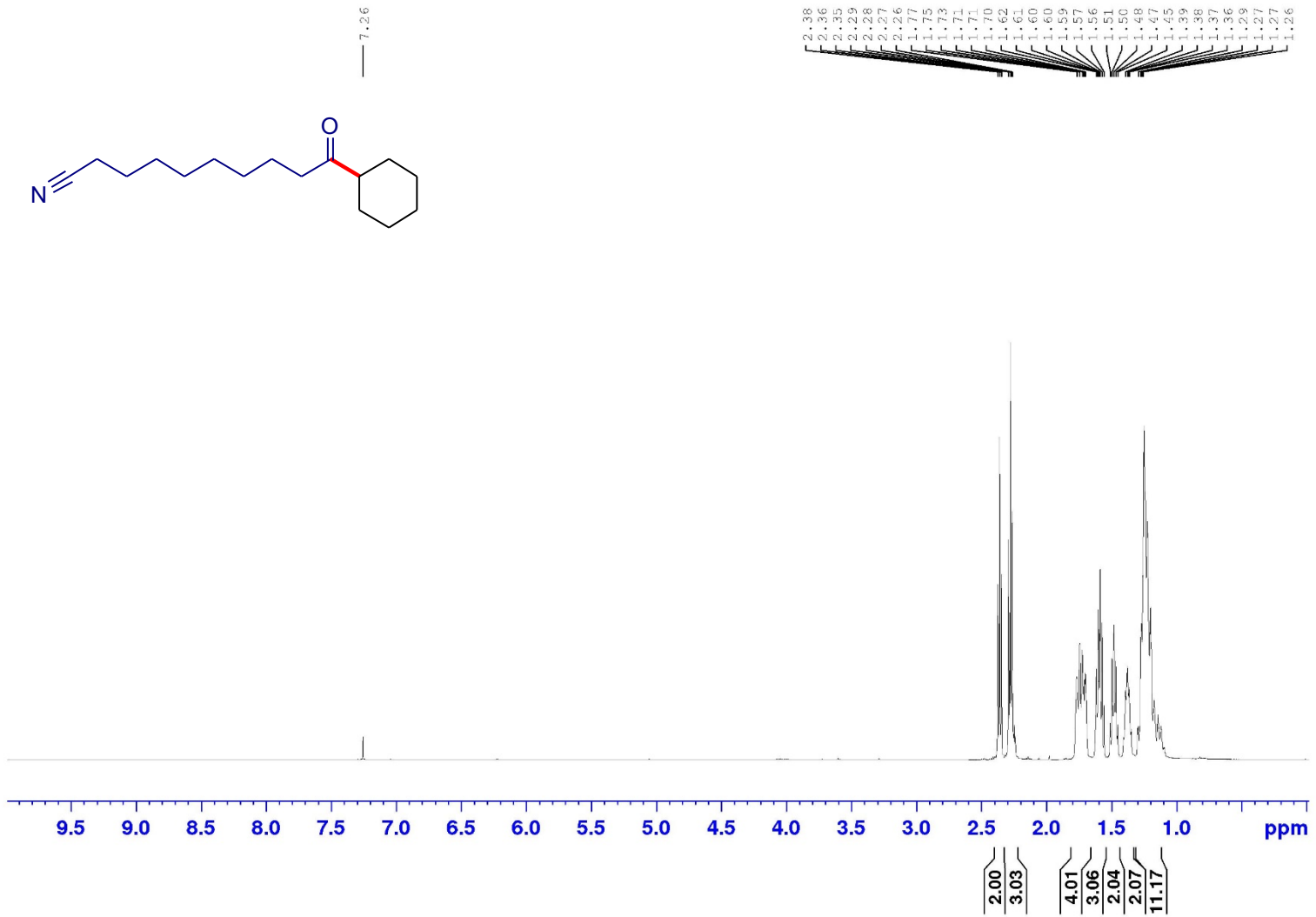
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of 3-(cyclohexanecarbonyl)cyclopentan-1-one (3k)



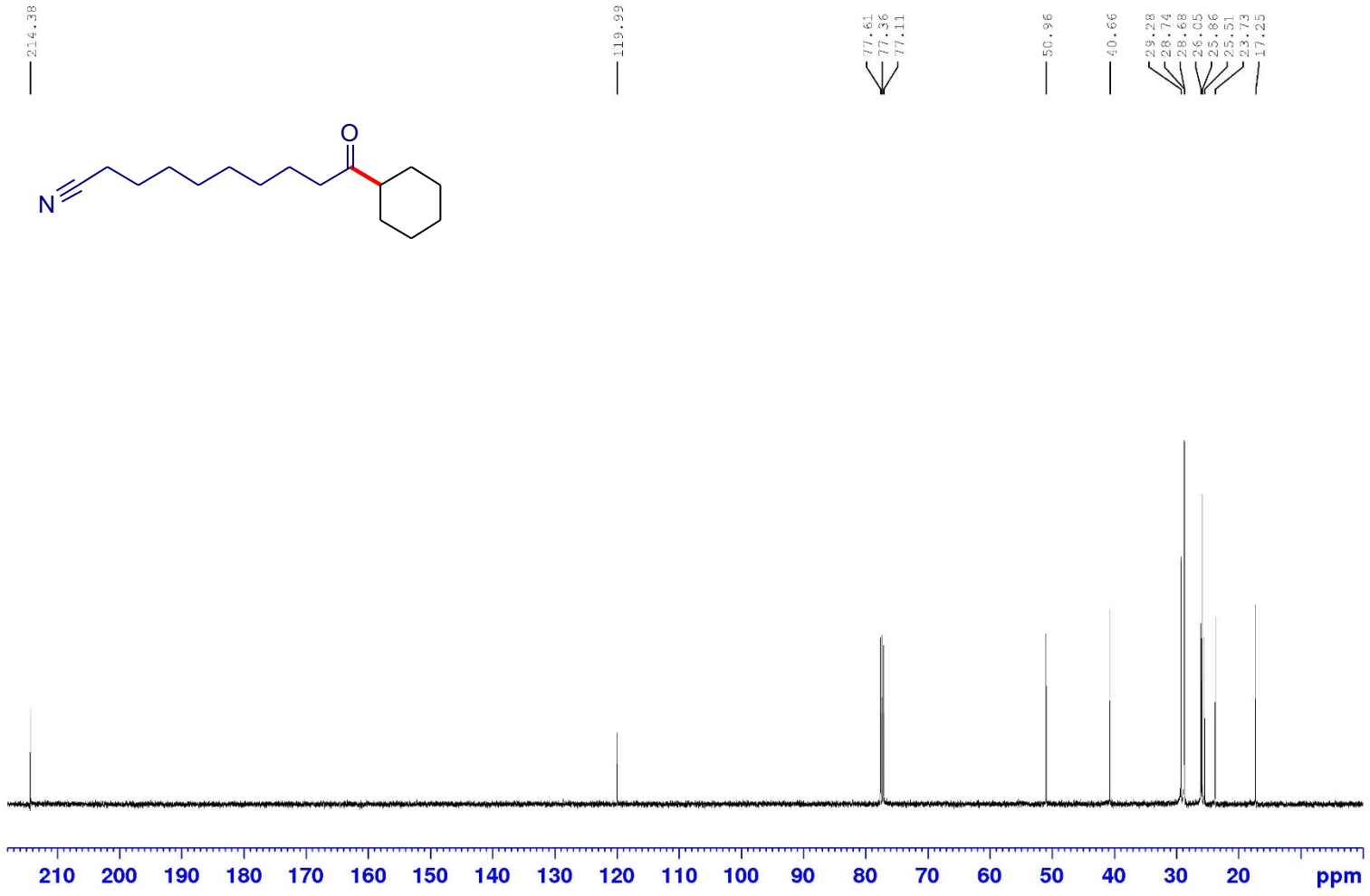
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 1-cyclohexyl-4-(thiophen-2-yl)butane-1,4-dione (3l)



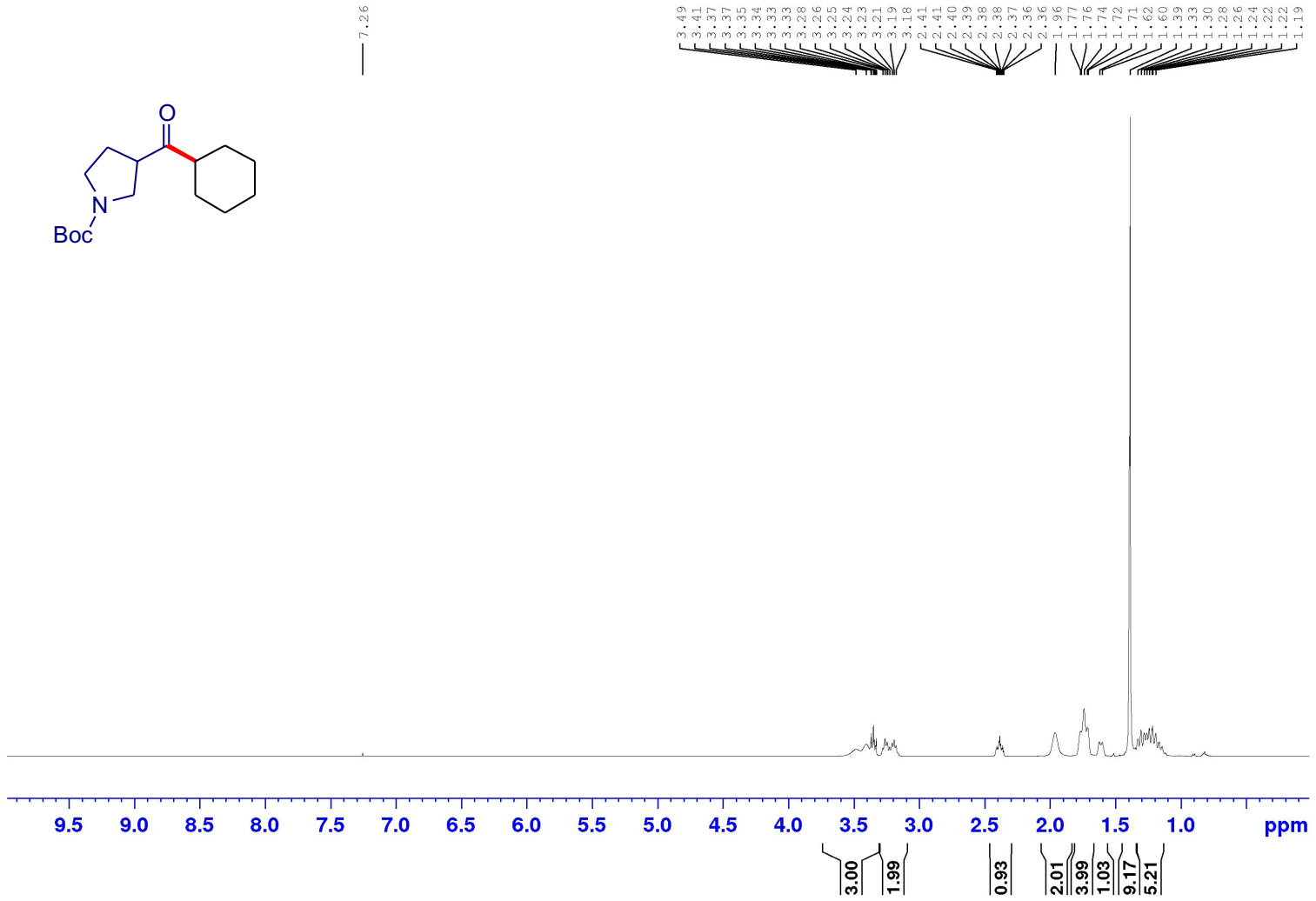
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of 1-cyclohexyl-4-(thiophen-2-yl)butane-1,4-dione (3l)



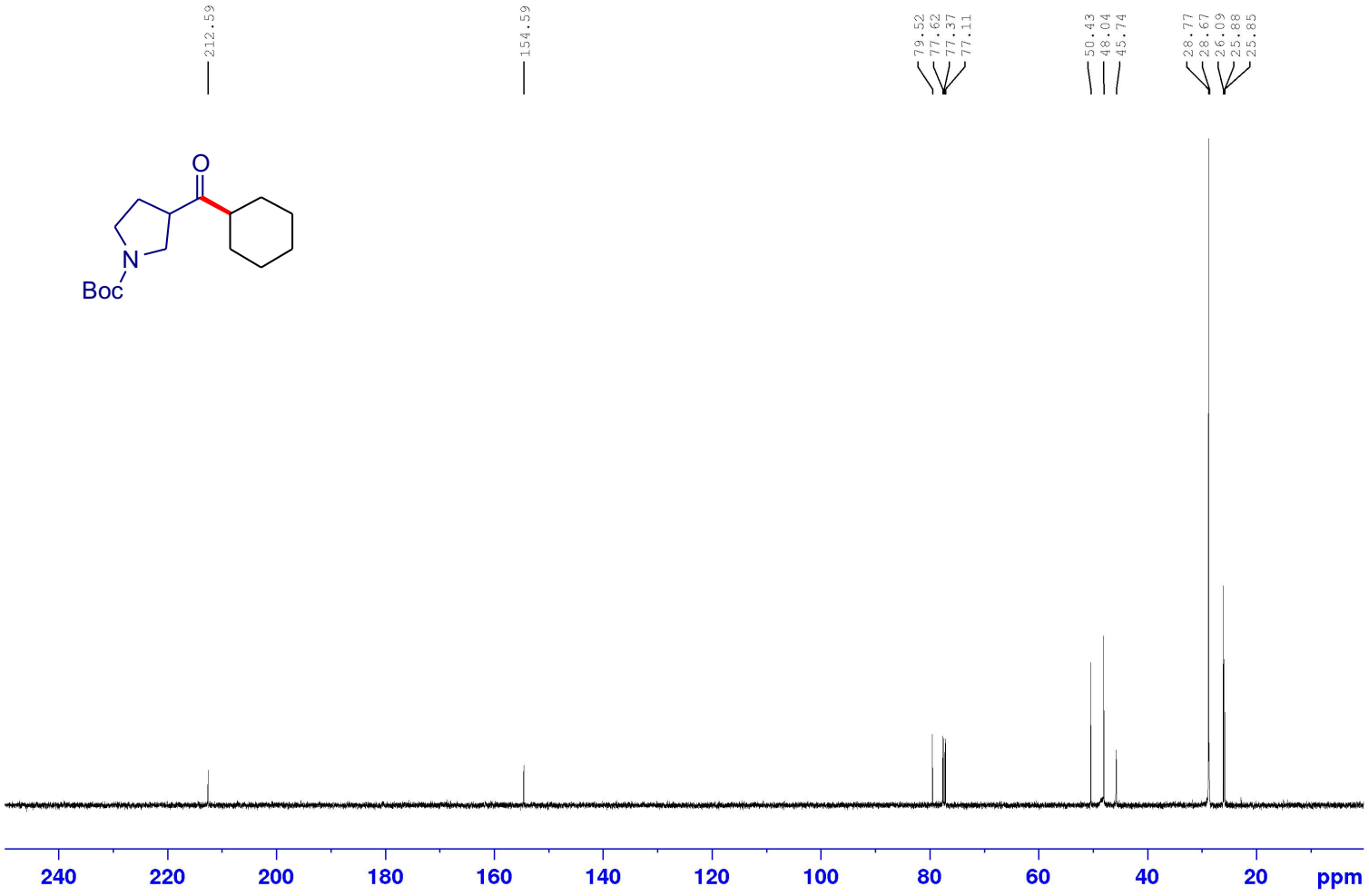
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 10-cyclohexyl-10-oxodecanenitrile (3m)



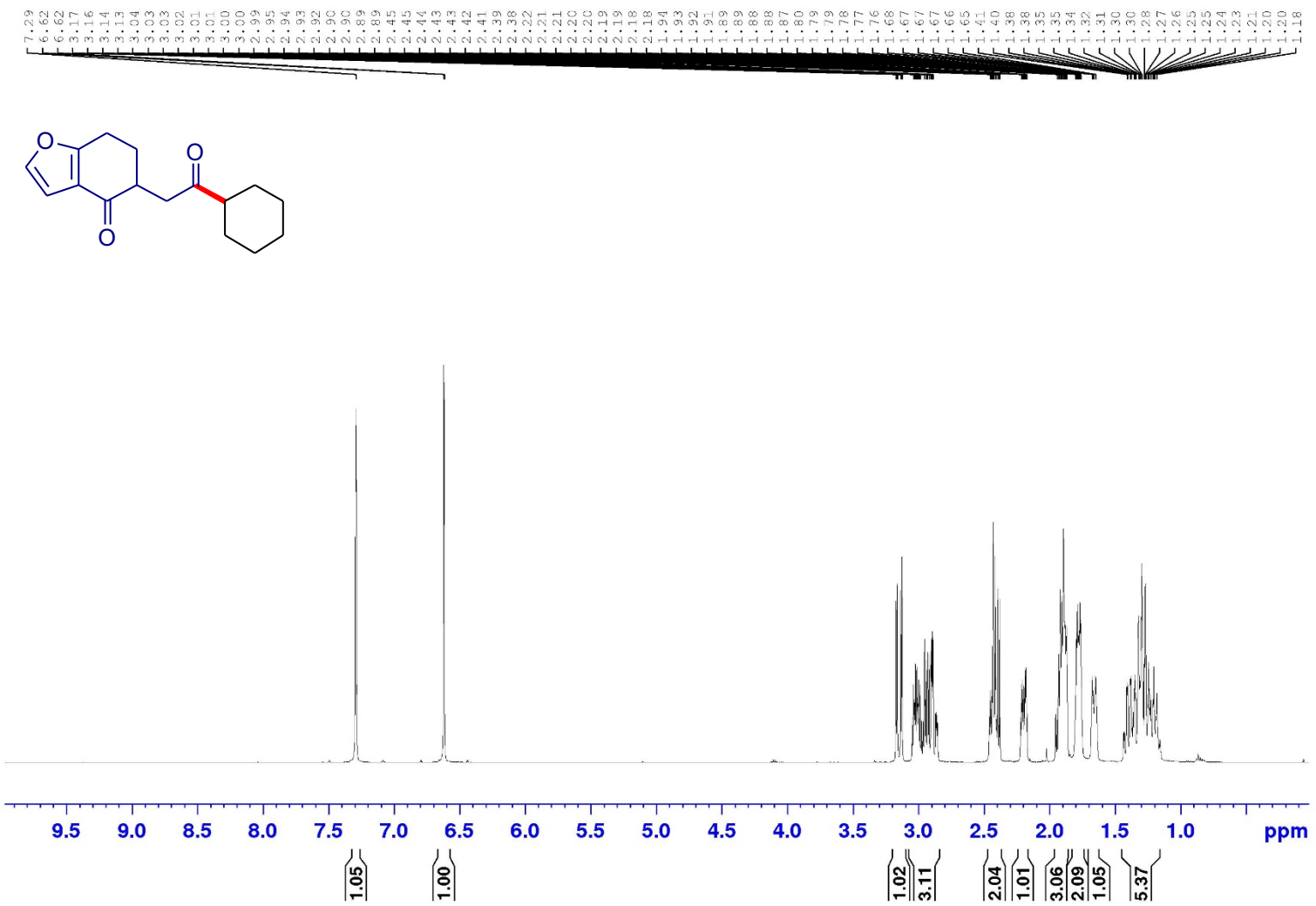
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of 10-cyclohexyl-10-oxodecanenitrile (3m)



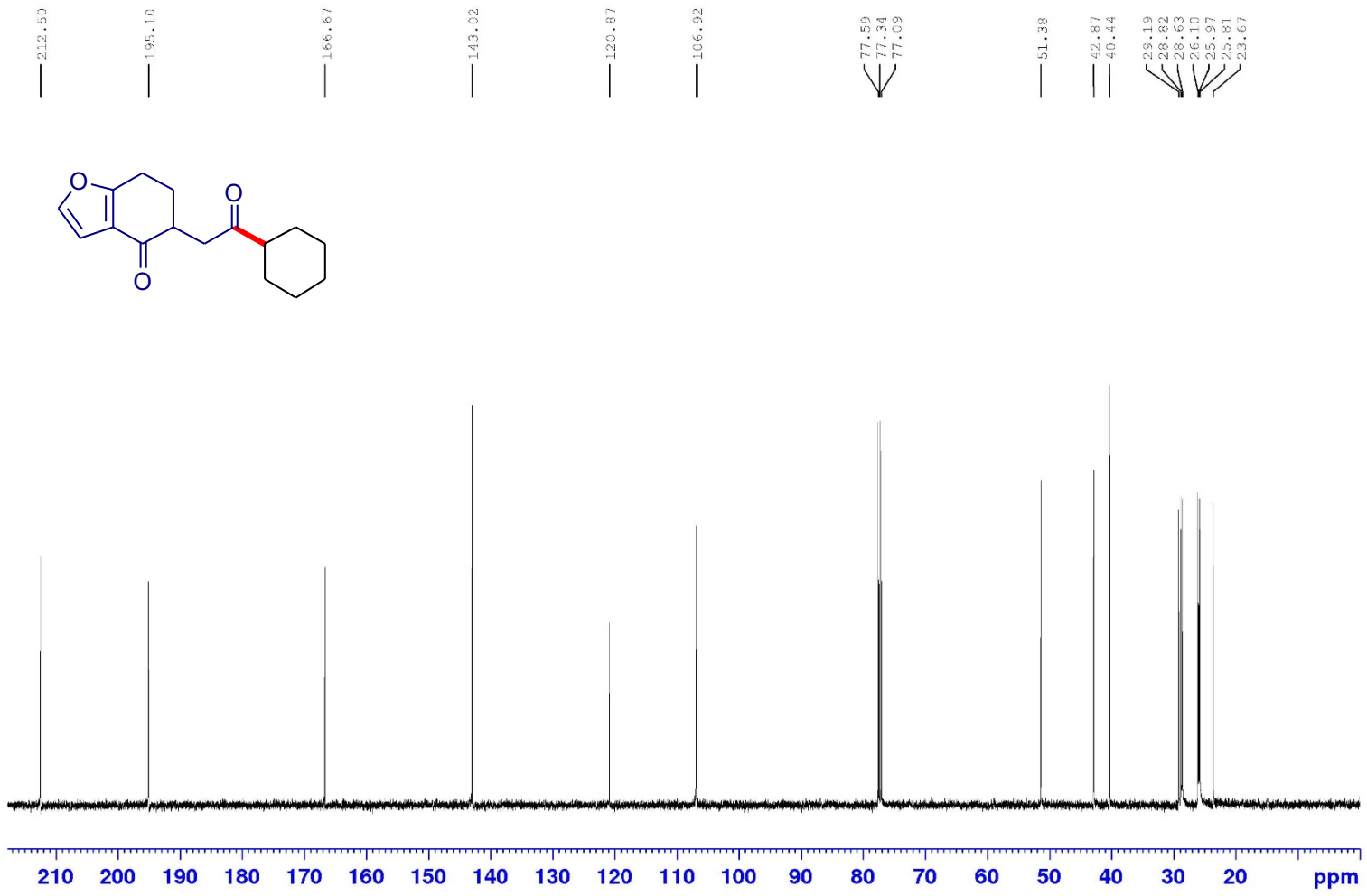
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of *tert*-butyl 3-(cyclohexanecarbonyl)pyrrolidine-1-carboxylate (3n)



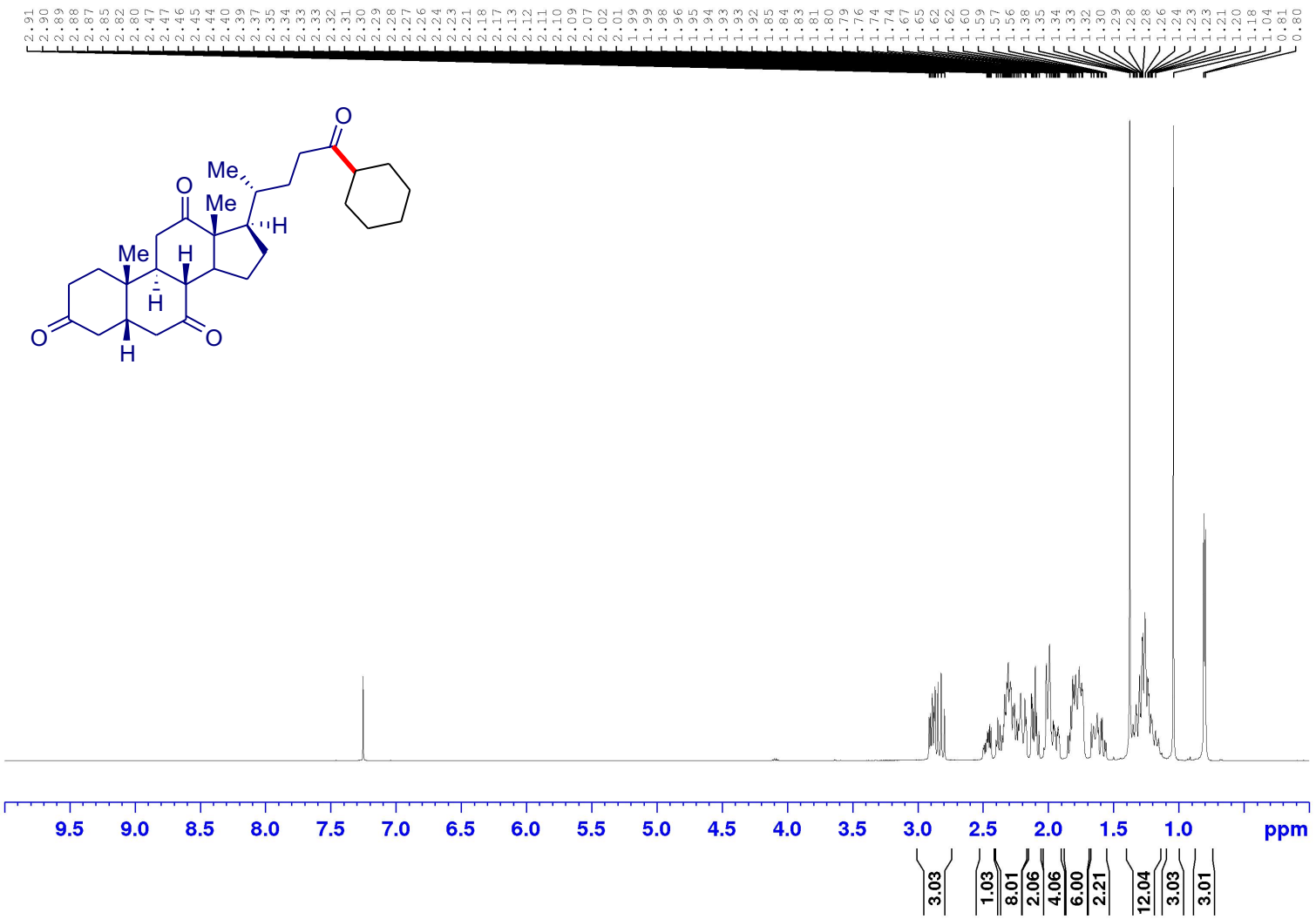
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of *tert*-butyl 3-(cyclohexanecarbonyl)pyrrolidine-1-carboxylate (3n)



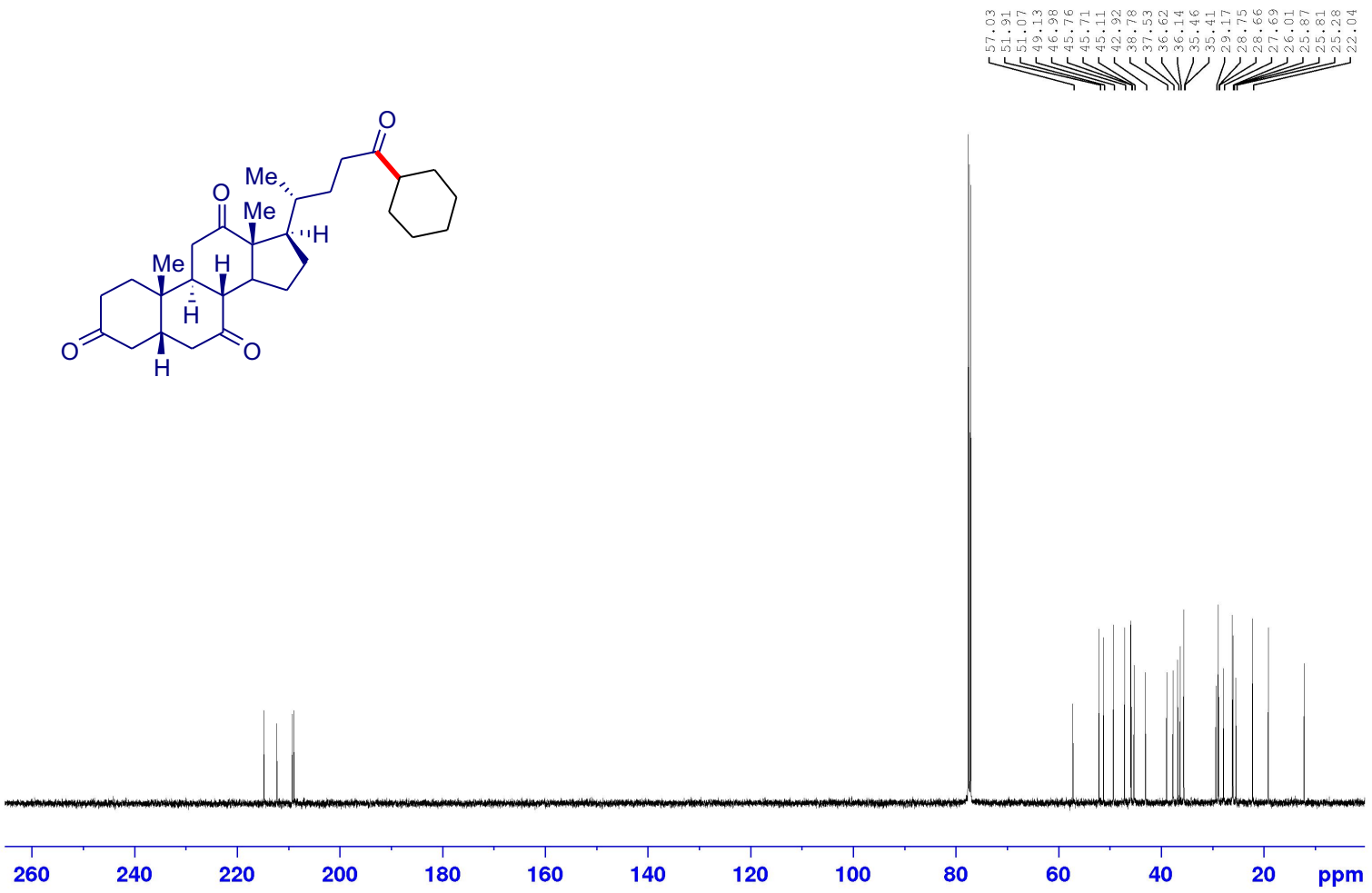
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 5-(2-cyclohexyl-2-oxoethyl)-6,7-dihydrobenzofuran-4(5H)-one (3o)



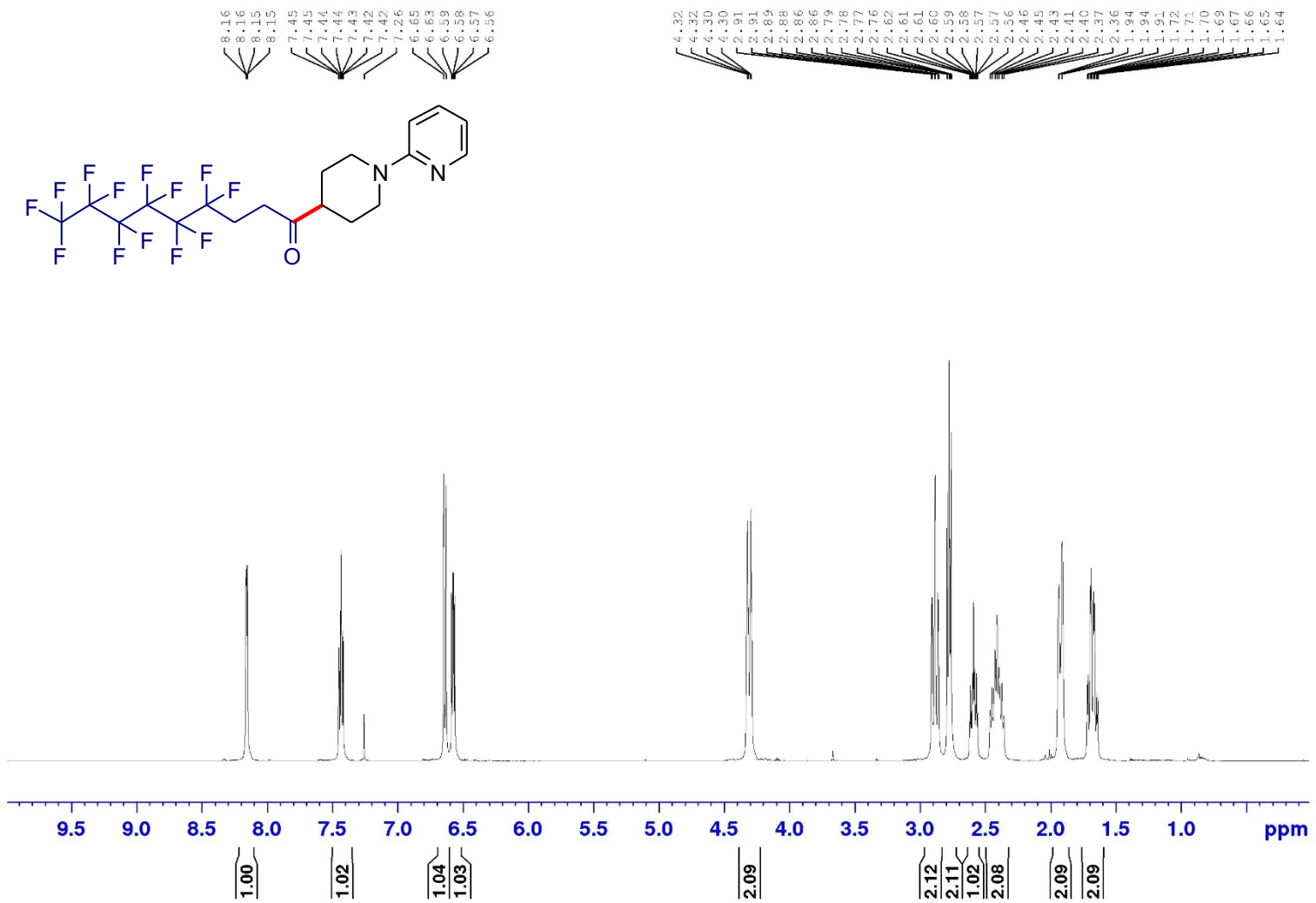
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of 5-(2-cyclohexyl-2-oxoethyl)-6,7-dihydrobenzofuran-4(5H)-one (3o)



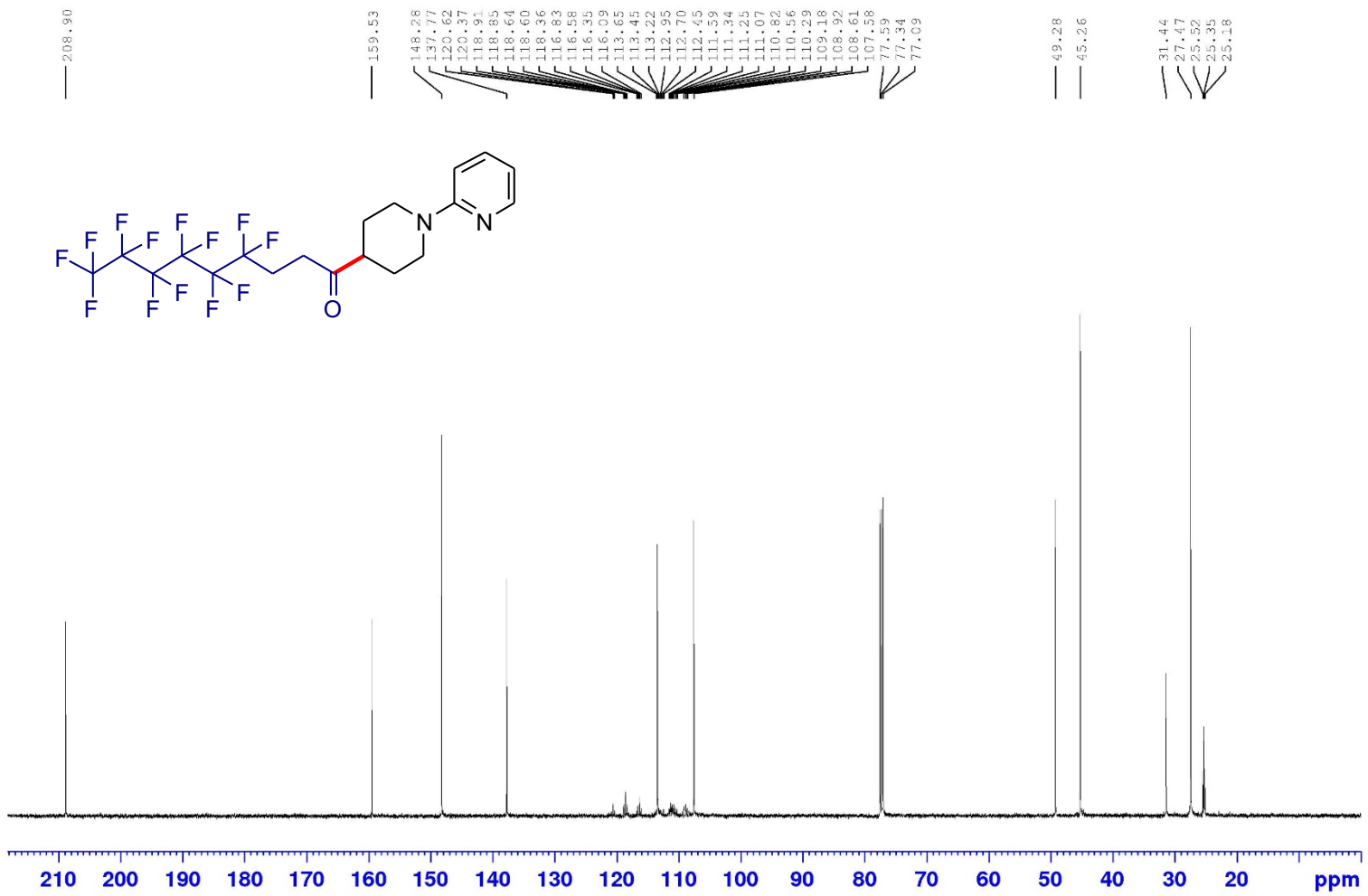
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of (5*S*,8*R*,9*S*,10*S*,13*R*,14*S*,17*R*)-17-((*R*)-5-cyclohexyl-5-oxopentan-2-yl)-10,13-dimethyldodecahydro-3H-cyclopenta[a]phenanthrene-3,7,12(2H,4H)-trione (3p)



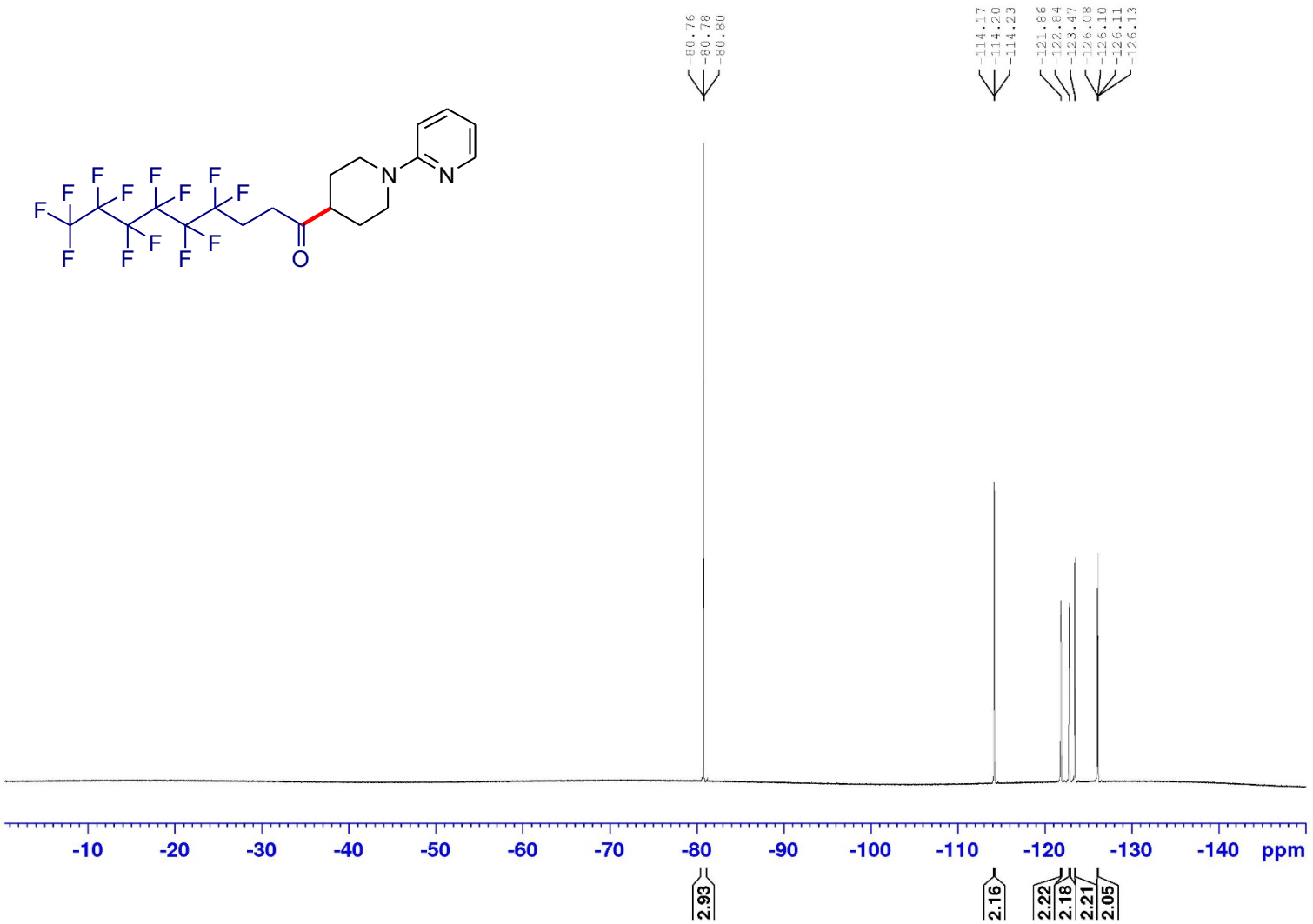
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of (5*S*,8*R*,9*S*,10*S*,13*R*,14*S*,17*R*)-17-((*R*)-5-cyclohexyl-5-oxopentan-2-yl)-10,13-dimethyldodecahydro-3H-cyclopenta[a]phenanthrene-3,7,12(2H,4H)-trione (3p)



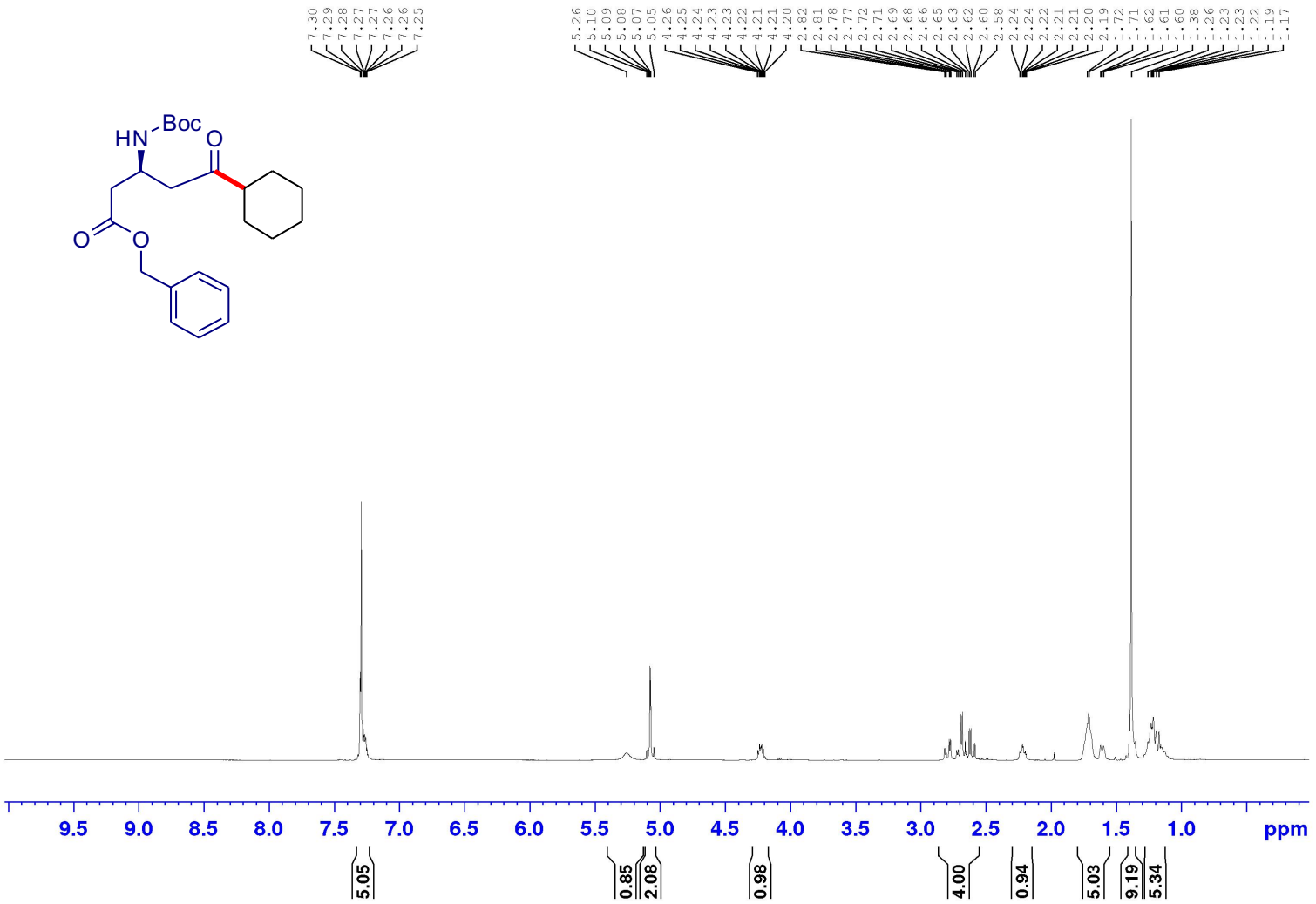
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 4,4,5,5,6,6,7,7,8,8,9,9,9-tridecafluoro-1-(1-(pyridin-2-yl)piperidin-4-yl)nonan-1-one (3q)



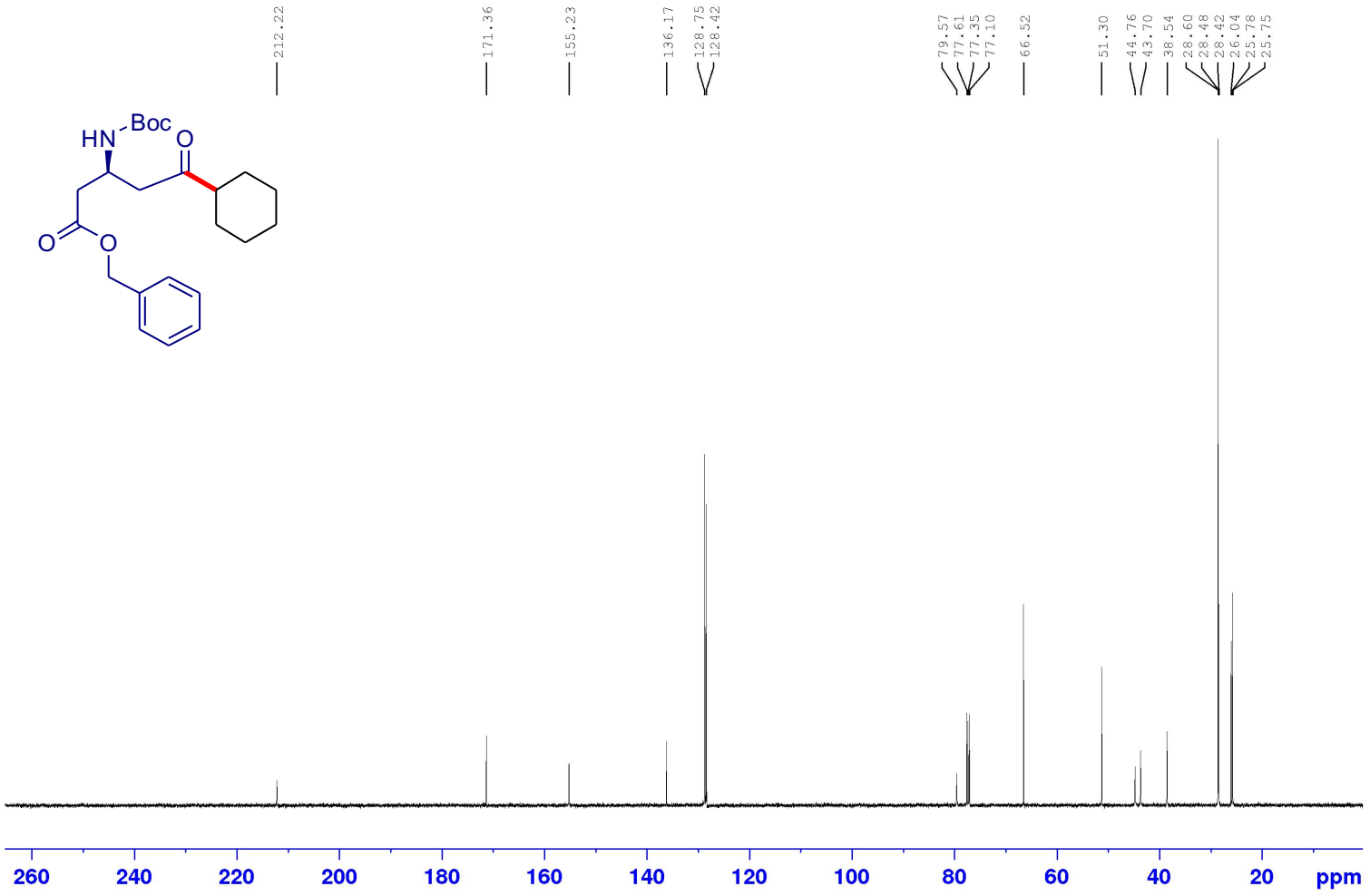
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of 4,4,5,5,6,6,7,7,8,8,9,9,9-tridecafluoro-1-(1-(pyridin-2-yl)piperidin-4-yl)nonan-1-one (3q)



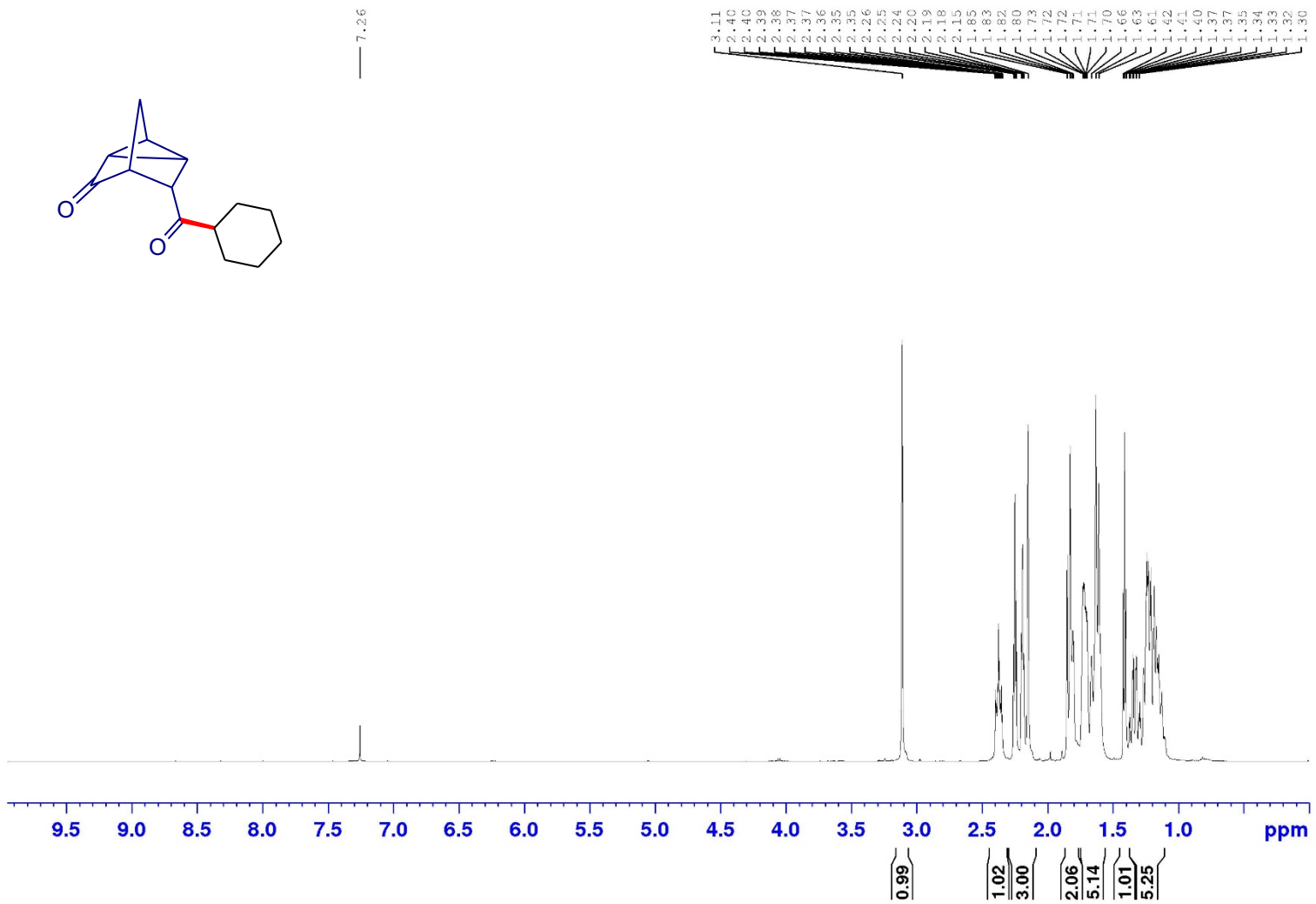
^{19}F NMR (470.7 MHz, $CDCl_3$) Spectrum of 4,4,5,5,6,6,7,7,8,8,9,9,9-tridecafluoro-1-(1-(pyridin-2-yl)piperidin-4-yl)nonan-1-one (3q)



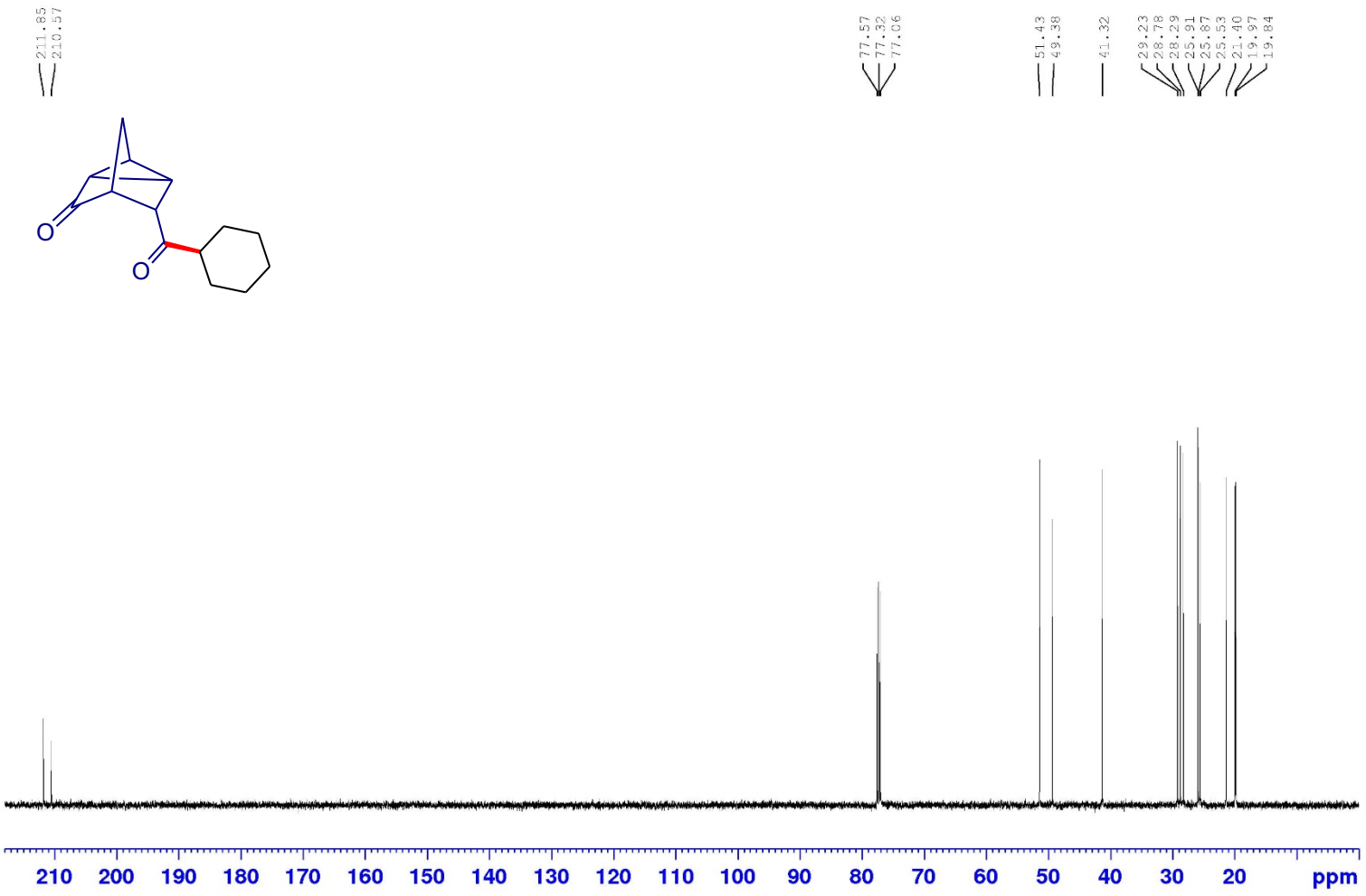
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of benzyl (S)-3-((tert-butoxycarbonyl)amino)-5-cyclohexyl-5-oxopentanoate (3r)



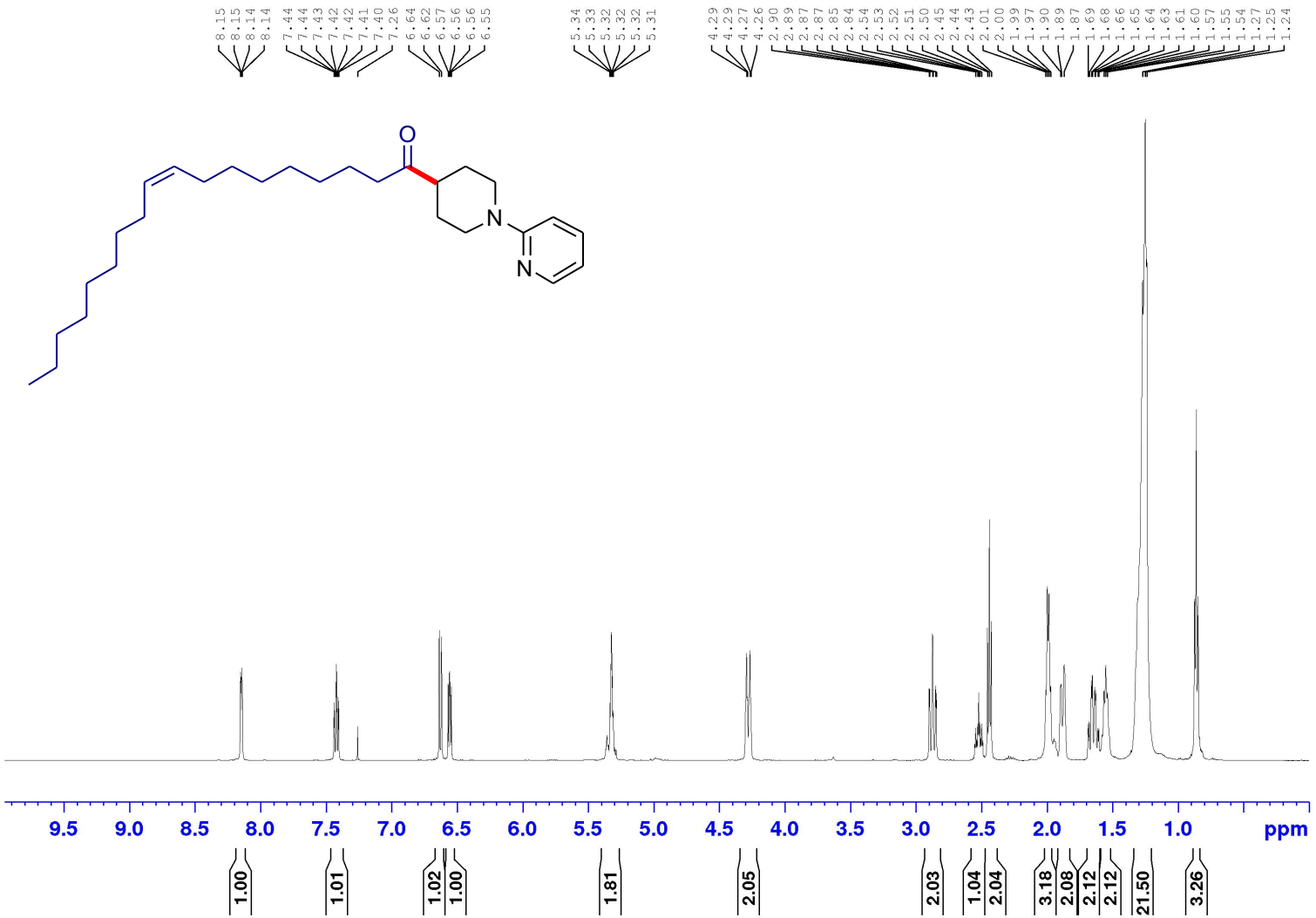
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of benzyl (*S*)-3-((*tert*-butoxycarbonyl)amino)-5-cyclohexyl-5-oxopentanoate (3r)



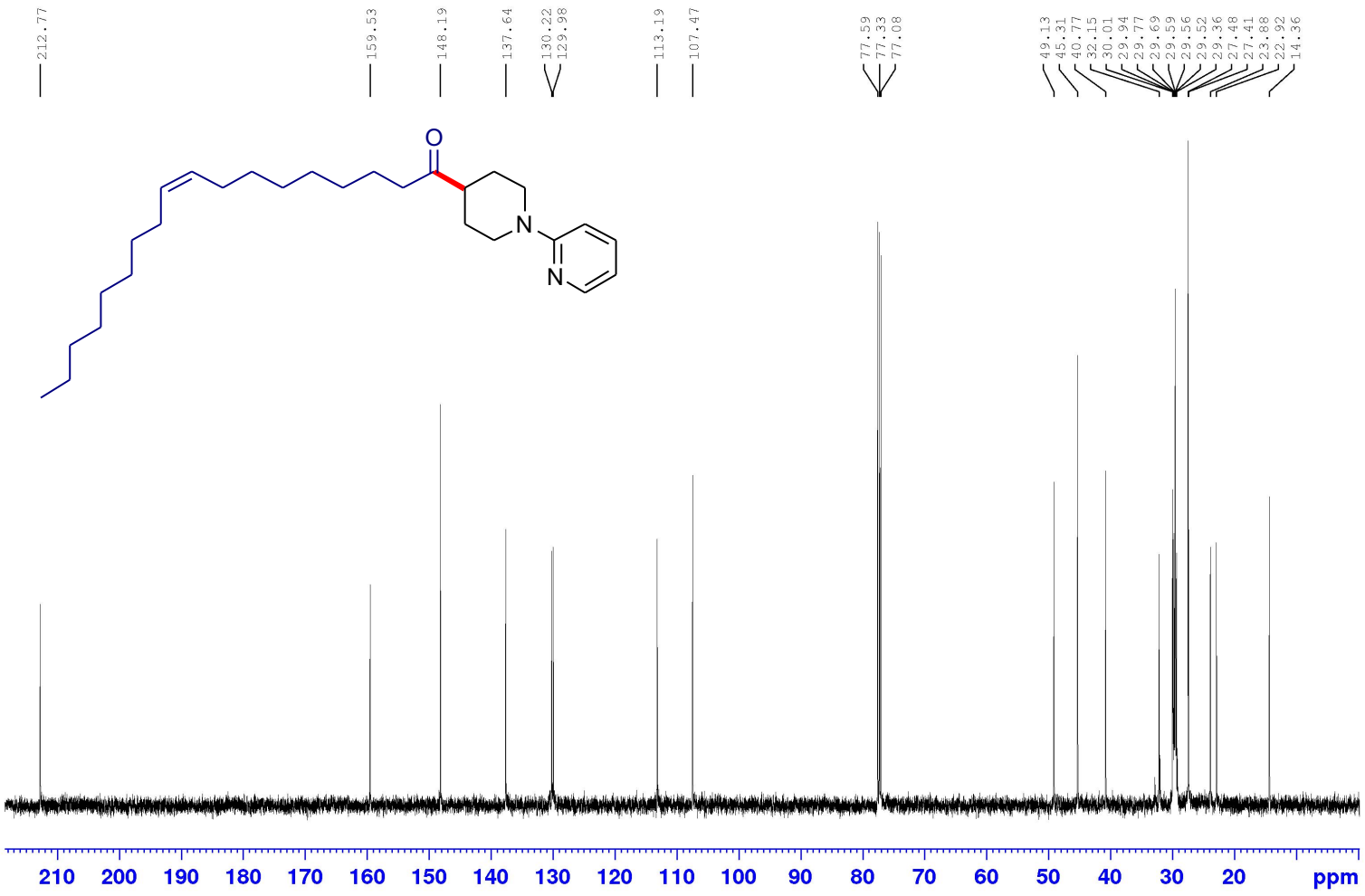
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 5-(cyclohexanecarbonyl)tricyclo[2.2.1.0^{2,6}]heptan-3-one (3s)



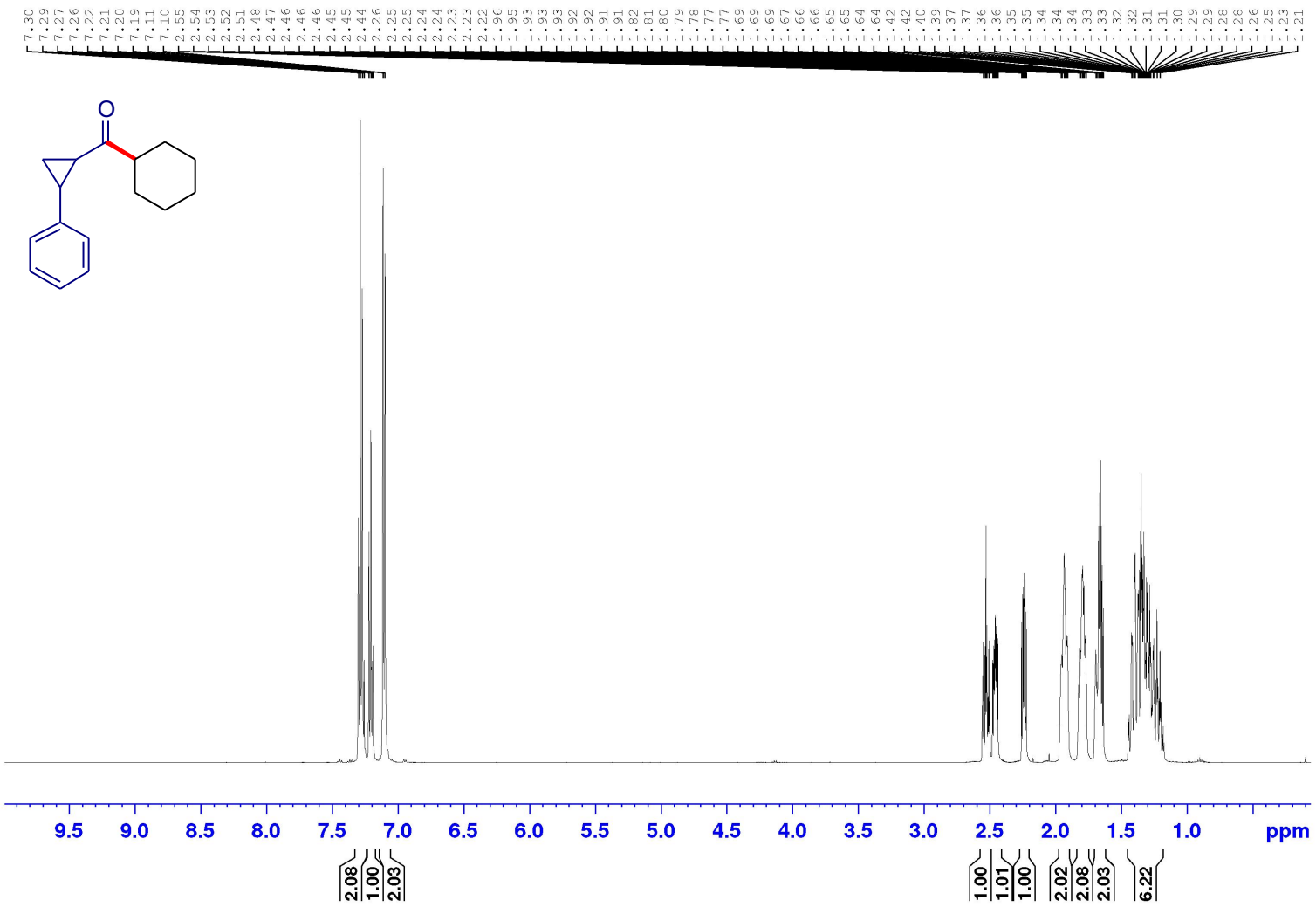
^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of 5-(cyclohexanecarbonyl)tricyclo[2.2.1.0^{2,6}]heptan-3-one (3s)



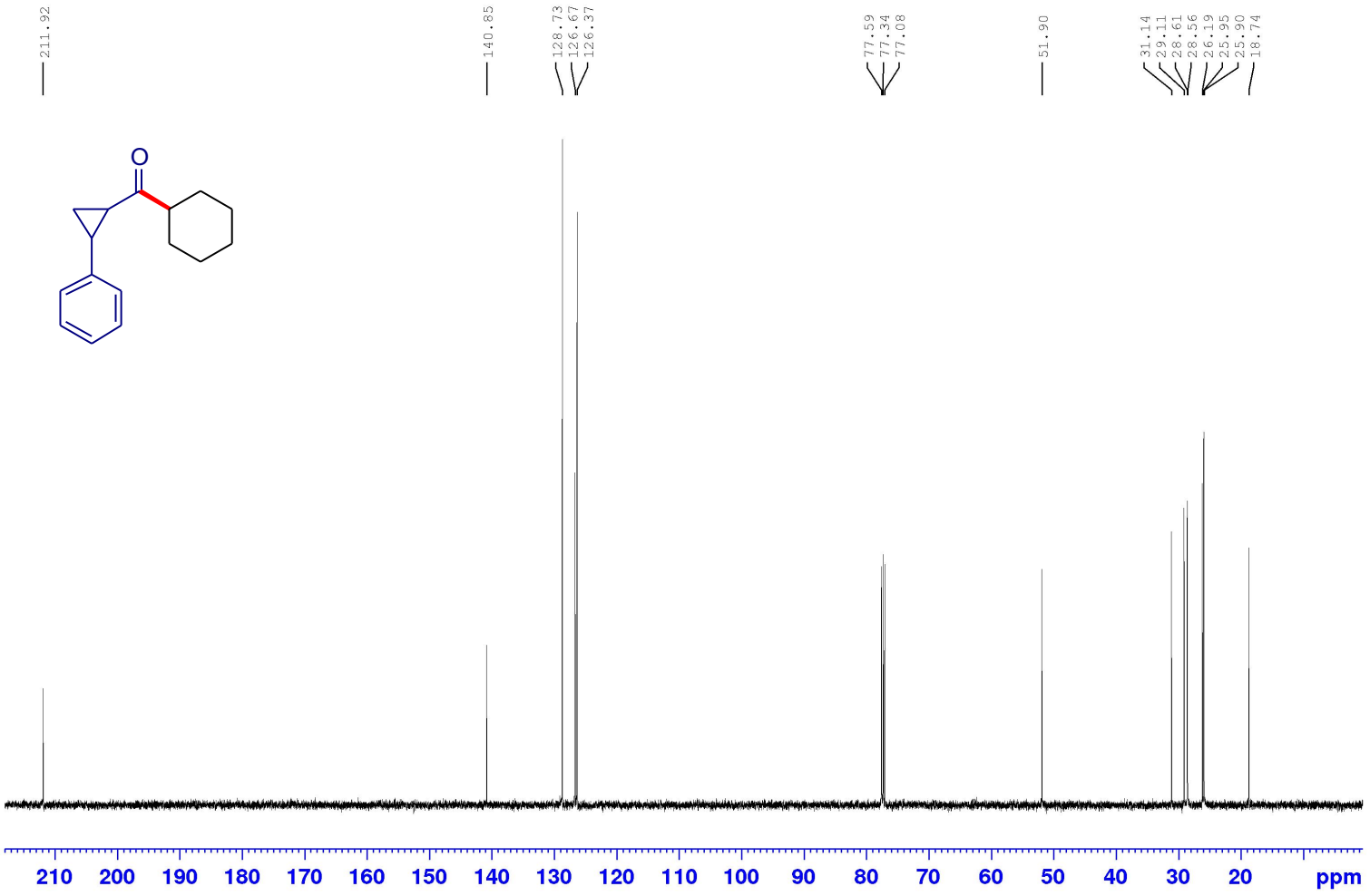
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of (*Z*)-1-(1-(pyridin-2-yl)piperidin-4-yl)octadec-9-en-1-one (3t)



^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of (*Z*)-1-(1-(pyridin-2-yl)piperidin-4-yl)octadec-9-en-1-one (3t)



^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of cyclohexyl(2-phenylcyclopropyl)methanone (3u)



^{13}C NMR (125.8 MHz, $CDCl_3$) Spectrum of cyclohexyl(2-phenylcyclopropyl)methanone (3u)