

Supporting Information

Synergistic Visible Light Photoredox/Nickel Catalyzed Synthesis of Aliphatic Ketones via N-C Cleavage of Imides

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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. Anhydrous 2-MeTHF, anhydrous CPME, $\text{IrCl}_3 \cdot x\text{H}_2\text{O}$, and $\text{NiCl}_2 \cdot \text{dme}$ were purchased from commercial sources. Compounds **2a**,¹ **2b**,² **2c**,³ and **2g**,⁴ $[\text{Ni}(\text{dtbbpy})(\text{H}_2\text{O})_4]\text{Cl}_2$,⁵ and $[\text{Ni}(\text{bpy})(\text{H}_2\text{O})_4]\text{Cl}_2$ ⁵ were synthesized according to the literature procedure. All other reagents were purchased commercially and used as received. Photoredox reactions were irradiated with blue LED lamp (Kessil KSH150B Blue LED Grow Light). Melting points ($^{\circ}\text{C}$) are uncorrected. NMR spectra were recorded on a 400 or 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. 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:

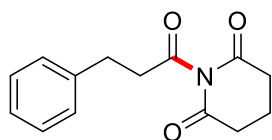
Most of the potassium alkyltrifluoroborates were purchased commercially. In cases where the desired potassium organotrifluoroborate was not available, the corresponding boronic acid derivative was converted to the trifluoroborate by the following procedure.

General procedure for conversion of boronic acids to trifluoroborates. To a solution of boronic acid derivative in MeOH (0.1 M) at 0°C saturated aq KHF_2 (4.5 M) was added dropwise over 30 min. After completion of the reaction, followed by ^{11}B NMR, the resulting suspension was concentrated under reduced pressure. The remaining H_2O was removed by lyophilizer. The remaining solid was suspended in hot acetone (3 x 100 mL) and filtered. The filtrate was

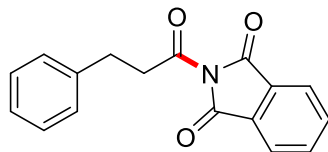
concentrated to a minimal volume (5 – 20 mL) and hexane or Et₂O (~200 mL) were added to yield a white precipitate. The precipitate was isolated by filtration, washing with hexanes (~30 mL) and CH₂Cl₂ (~30 mL), to afford the desired secondary alkyltrifluoroborate.

Synthesis of Ir[dF(CF₃)ppy]₂(bpy)PF₆ as the photocatalyst 1:

Photocatalyst **I** was synthesized according to the literature procedure.⁶



Synthesis of 1-(3-phenylpropanoyl)piperidine-2,6-dione (2d). To a solution of glutarimide (565.5 mg, 5.0 mmol) in anhydrous THF (50 mL) was slowly added a 2.5 M solution of *n*-BuLi in hexane (2.0 mL, 5.0 mmol) at -78 °C under Ar atmosphere. The reaction mixture was stirred for 30 min at -78 °C. Then hydrocinnamoyl chloride (927.4 mg, 5.5 mmol) was added to the reaction mixture at -78 °C. After 10 min the solution was allowed to reach 0 °C. Then it was treated with saturated aqueous NaHCO₃ (25 mL) and transferred to a separatory funnel. The organic layer was separated and the aqueous layer was extracted with EtOAc (3 × 20 mL). The combined organic extracts were dried over anhydrous MgSO₄ and concentrated. The residue was purified by column chromatography on silica gel, with EtOAc/hexanes mixtures as the eluent, to obtain product in pure form. The title compound was obtained as a white solid in 70% yield (2.2 mmol scale, 377.7 mg). mp 93-94 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.30-7.27 (m, 2H), 7.21-7.18 (m, 3H), 3.03 (s, 4H), 2.60 (t, *J* = 6.5 Hz, 4H), 1.98-1.92 (m, 2H); ¹³C NMR (125.8 MHz, CDCl₃): δ 177.7, 171.8, 139.8, 128.8, 128.5, 126.6, 42.5, 32.4, 29.7, 17.4. FT-IR (neat): 1805, 1744, 1708, 1302, 1169, 1093, 629 cm⁻¹; HRMS (CI⁺) *m/z* calcd. for C₁₄H₁₅NO₃ [M]⁺ 245.1052, found 245.1050.

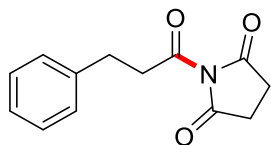


Synthesis of 2-(3-phenylpropanoyl)isoindoline-1,3-dione (2f). To a solution of phthalimide (735.6 mg, 5.0 mmol) in anhydrous THF (50 mL) was slowly added a 2.5 M solution of *n*-BuLi in hexane (2.0 mL, 2.0 mmol) at -78 °C under Ar atmosphere. The reaction mixture was stirred at -78 °C for 30 min. Then hydrocinnamoyl chloride (927.4 mg, 2.2 mmol) was added to the reaction mixture at -78 °C. After 10 min the solution was allowed to reach 0 °C. Then it was treated with saturated aqueous NaHCO₃ (25 mL) and transferred to a separatory funnel. The organic layer was separated and the aqueous layer was extracted with EtOAc (3 × 20 mL). The combined organic extracts were dried over anhydrous MgSO₄ and concentrated. The residue was purified by column chromatography on silica gel, with EtOAc/hexanes mixtures as the eluent, to obtain product in pure form. The title compound was obtained as a white solid in 61% yield (2.2 mmol scale, 374.8 mg). mp 112-113 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.91-7.89 (m, 2H), 7.79-7.78 (m, 2H), 7.23 (s, 4H), 7.16-7.15 (m, 1H), 3.31 (t, *J* = 7.5 Hz, 2H), 3.03 (t, *J* = 7.5 Hz, 2H); ¹³C NMR (125.8 MHz, CDCl₃): δ 171.4, 165.6, 140.4, 135.7, 131.2, 128.8, 128.7, 126.5, 124.6, 40.7, 30.2; FT-IR (neat): 1802, 1781, 1759, 1749, 1286, 1132, 715, 699 cm⁻¹; HRMS (ES⁺) *m/z* calcd. for C₁₇H₁₄NO₃ [M+H]⁺ 280.0974, found 280.0967.

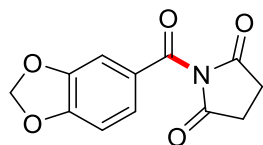
General Procedure for the Synthesis of *N*-acylpyrrolidine-2,5-diones. To a flame-dried 25 mL round bottom flask equipped with a Teflon-coated magnetic stir bar, anhydrous DCM (8 mL) and anhydrous DMF (2.0 mmol) were added under Ar atmosphere. Then oxalyl chloride (2.3 mmol, 291.9 mg) and anhydrous pyridine (2.0 mmol) were added to the mixture at 0°C. The reaction mixture was stirred until it appeared as a brown suspension. A solution of desired carboxylic acid

(2.0 mmol) in anhydrous DCM (8 mL) was added dropwise to the reaction mixture under Ar at 0°C. The resulting mixture was stirred at room temperature under inert atmosphere for 4 h.

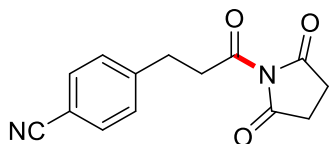
At the same time a solution of succinimide (1.9 mmol, 188.3 mg), TEA (3.8 mmol, 384.5 mg), and DMAP (0.5 mmol, 61.1 mg) in anhydrous DCM (10 mL) was prepared in another flame-dried 50 mL round bottom flask equipped with a Teflon-coated magnetic stir bar under Ar atmosphere. The mixture was then cooled to 0°C. The resulting mixture from the first reaction was collected carefully by a syringe under Ar and transferred dropwise to the second flask at 0°C. The reaction mixture was stirred overnight. Then it was treated with 1M HCl (15 mL) and transferred to a separatory funnel. The organic layer washed with H₂O (5 × 30 mL), brine solution (30 mL), and dried over anhydrous MgSO₄. 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 product in pure form.



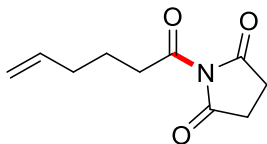
1-(3-Phenylpropanoyl)pyrrolidine-2,5-dione (2e).⁶ The title compound was obtained as a white solid in 60% yield (2.0 mmol scale, 277.5 mg). ¹H NMR (500 MHz, CDCl₃): δ 7.27-7.23 (m, 2H), 7.20-7.16 (m, 3H), 3.18 (t, *J* = 7.5 Hz, 2H), 2.98 (t, *J* = 7.5 Hz, 2H), 2.68 (s, 4H); ¹³C NMR (125.8 MHz, CDCl₃): δ 177.8, 171.8, 139.8, 128.8, 128.6, 126.7, 42.5, 32.4, 29.7, 17.4.



1-(Benzo[d][1,3]dioxole-5-carbonyl)pyrrolidine-2,5-dione. The title compound was obtained as a yellow solid in 61% yield (2.0 mmol scale, 301.5 mg). mp 148-149 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.42 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.31-7.30 (m, 1H), 6.86 (d, *J* = 8.0 Hz, 1H), 6.08 (s, 2H), 2.90 (s, 4H); ¹³C NMR (125.8 MHz, CDCl₃): δ 175.0, 166.6, 154.1, 148.8, 128.2, 125.9, 110.1, 108.7, 102.7, 29.3; FT-IR (neat): 1718, 1689, 1256, 617 cm⁻¹; HRMS (ES⁺) *m/z* calcd. for C₁₂H₁₀NO₅ [M+H]⁺ 248.0559, found 248.0556.

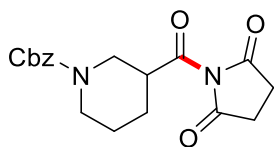


4-(3-(2,5-Dioxopyrrolidin-1-yl)-3-oxopropyl)benzonitrile. The title compound was obtained as a white solid in 47% yield (2.0 mmol scale, 240.8 mg). mp 110-112 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.56 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 3.22 (t, *J* = 7.5 Hz, 2H), 3.06 (t, *J* = 7.5 Hz, 2H), 2.77 (s, 4H); ¹³C NMR (125.8 MHz, CDCl₃): δ 174.1, 171.2, 145.6, 132.4, 129.5, 118.9, 110.5, 39.8, 29.7, 28.5; FT-IR (neat): 1805, 1748, 1711, 1301, 1161, 627 cm⁻¹; HRMS (CI⁺) *m/z* calcd. for C₁₄H₁₂N₂O₃ [M]⁺ 256.0848, found 256.0839.

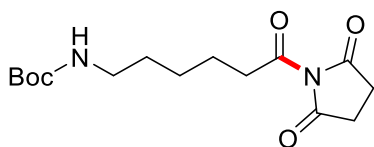


1-(Hex-5-enoyl)pyrrolidine-2,5-dione. The title compound was obtained as a liquid in 43% yield (2.0 mmol scale, 167.8 mg). ¹H NMR (500 MHz, CDCl₃): δ 5.78-5.71 (m, 1H), 5.03-4.97 (m, 2H), 2.86 (t, *J* = 7.5 Hz, 2H), 2.77 (s, 4H), 2.13-2.08 (m, 2H), 1.78-1.76 (m, 2H); ¹³C NMR (125.8 MHz, CDCl₃): δ 174.3, 172.8, 137.5, 115.7, 38.2, 32.7, 28.6, 22.9; FT-IR (neat): 1805, 1744, 1708,

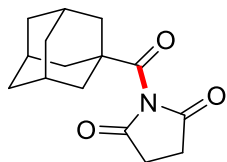
1302, 1169, 1093, 629 cm^{-1} ; HRMS (ES+) m/z calcd. for $\text{C}_{10}\text{H}_{14}\text{NO}_3$ $[\text{M}+\text{H}]^+$ 196.0974, found 196.0975.



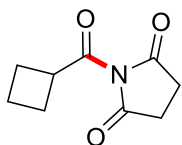
Benzyl 3-(2,5-dioxopyrrolidine-1-carbonyl)piperidine-1-carboxylate. The title compound was obtained as a liquid in 49% yield (2.0 mmol scale, 337.4 mg). ^1H NMR (500 MHz, CD_3CN): δ 7.41-7.35 (m, 5H), 5.15 (s, 2H), 4.04 (d, $J = 12.5$ Hz, 1H), 3.79-3.76 (m, 1H), 3.41-3.31 (m, 2H), 3.18 (t, $J = 10$ Hz, 1H), 2.72 (s, 4H), 2.03-2.01 (m, 1H), 1.79-1.77 (m, 2H), 1.53-1.51 (m, 1H); ^{13}C NMR (125.8 MHz, CD_3CN): δ 176.0, 175.4, 156.2, 138.6, 129.6, 129.0, 128.7, 68.0, 46.3, 45.3, 30.6, 29.7, 27.4, 24.6; FT-IR (neat): 1804, 1744, 1693, 1428, 1234, 1150, 697 cm^{-1} ; HRMS (ES+) m/z calcd. for $\text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_5\text{Na}$ $[\text{M}+\text{Na}]^+$ 367.1270, found 367.1263.



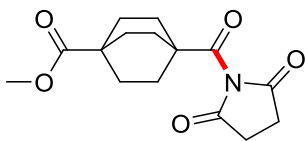
tert-Butyl (6-(2,5-dioxopyrrolidin-1-yl)-6-oxohexyl)carbamate. The title compound was obtained as a white solid in 46% yield (0.5 mmol scale, 287.3 mg). mp 80-81 $^\circ\text{C}$; ^1H NMR (500 MHz, CDCl_3): δ 4.45 (s, 1H), 3.12-3.08 (m, 2H), 2.85 (t, $J = 7.5$ Hz, 2H), 2.75 (s, 4H), 1.73-1.67 (m, 2H), 1.51-1.37 (m, 13H); ^{13}C NMR (125.8 MHz, CDCl_3): δ 174.3, 172.9, 156.2, 79.3, 40.8, 39.0, 30.0, 28.8, 28.7, 26.3, 23.7; FT-IR (neat): 3366, 1745, 1689, 1526, 1174, 627 cm^{-1} ; HRMS (ES+) m/z calcd. for $\text{C}_{15}\text{H}_{24}\text{N}_2\text{O}_5\text{Na}$ $[\text{M}+\text{Na}]^+$ 335.1583, found 335.1587.



1-(Adamantane-1-carbonyl)pyrrolidine-2,5-dione. The title compound was obtained as a white solid in 55% yield (2.0 mmol scale, 287.4 mg). mp 107-109 °C; ^1H NMR (500 MHz, CDCl_3): δ 2.76 (s, 4H), 2.01 (s, 3H), 1.89-1.88 (m, 6H), 1.67 (dd, $J = 23.5, 12.5$ Hz, 6H); ^{13}C NMR (125.8 MHz, CDCl_3): δ 182.2, 175.5, 46.2, 37.8, 36.3, 29.5, 27.8; FT-IR (neat): 1785, 1706, 1163, 651 cm^{-1} ; HRMS (ES+) m/z calcd. for $\text{C}_{15}\text{H}_{19}\text{NO}_3\text{Na}$ $[\text{M}+\text{Na}]^+$ 284.1263, found 284.1276.



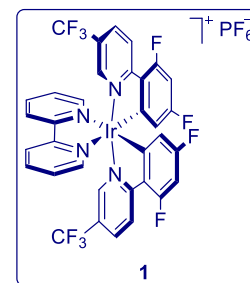
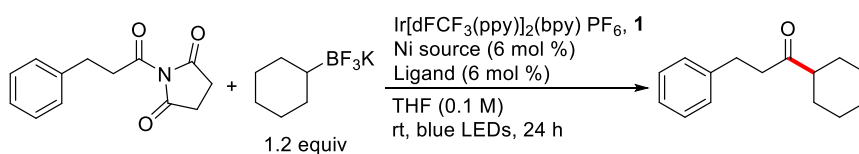
1-(Cyclobutanecarbonyl)pyrrolidine-2,5-dione. The title compound was obtained as a white solid in 51% yield (0.5 mmol scale, 184.8 mg). mp 88-89 °C; ^1H NMR (500 MHz, CDCl_3): δ 3.88-3.81 (m, 1H), 2.76 (s, 4H), 2.37-2.29 (m, 2H), 2.26-2.19 (m, 2H), 2.03-1.85 (m, 2H); ^{13}C NMR (125.8 MHz, CDCl_3): δ 174.7, 174.5, 41.8, 28.8, 24.8, 17.9; FT-IR (neat): 1804, 1736, 1700, 1175, 638 cm^{-1} ; HRMS (ES+) m/z calcd. for $\text{C}_9\text{H}_{12}\text{NO}_3$ $[\text{M}+\text{H}]^+$ 182.0817, found 182.0835.



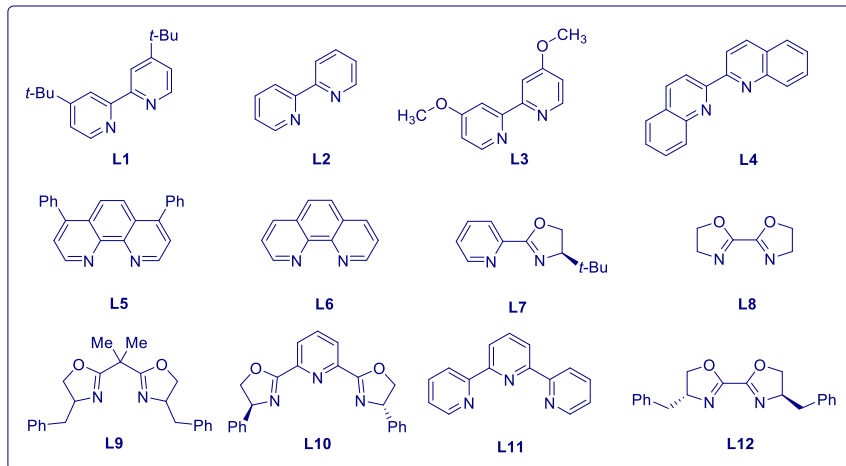
Methyl 4-(2,5-dioxopyrrolidine-1-carbonyl)bicyclo[2.2.2]octane-1-carboxylate. The title compound was obtained as a white solid in 68% yield (2.0 mmol scale, 398.9 mg). mp 104-106 °C; ^1H NMR (500 MHz, CDCl_3): δ 3.64 (s, 3H), 2.80 (s, 4H), 1.90-1.87 (m, 6H), 1.84-1.81 (m, 6H); ^{13}C NMR (125.8 MHz, CDCl_3): δ 182.0, 177.6, 175.1, 52.2, 52.1, 44.7, 39.0, 29.5, 27.7, 27.2; FT-

IR (neat): 1732, 1715, 1702, 1329, 1254, 1071, 649 cm^{-1} ; HRMS (ES+) m/z calcd. for $\text{C}_{15}\text{H}_{20}\text{NO}_5$ $[\text{M}+\text{H}]^+$ 294.1341, found 294.1368.

High-Throughput Experiments in design and optimization of the photoredox cross-coupling of 2e with potassium cyclohexyltrifluoroborate as model coupling partners. 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, 1-(3-phenylpropanoyl)pyrrolidine-2,5-dione, **2e**, 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.



First Screen Variables:



<u>solvents</u>	<u>ligands</u>	<u>bases</u>	<u>Ni sources</u>
THF	L1	no base	Ni(COD) ₂
	L2		NiCl ₂ .dme
	L3		
	L4		
	L5		
	L6		
	L7		
	L8		
	L9		
	L10		
	L11		
	L12		

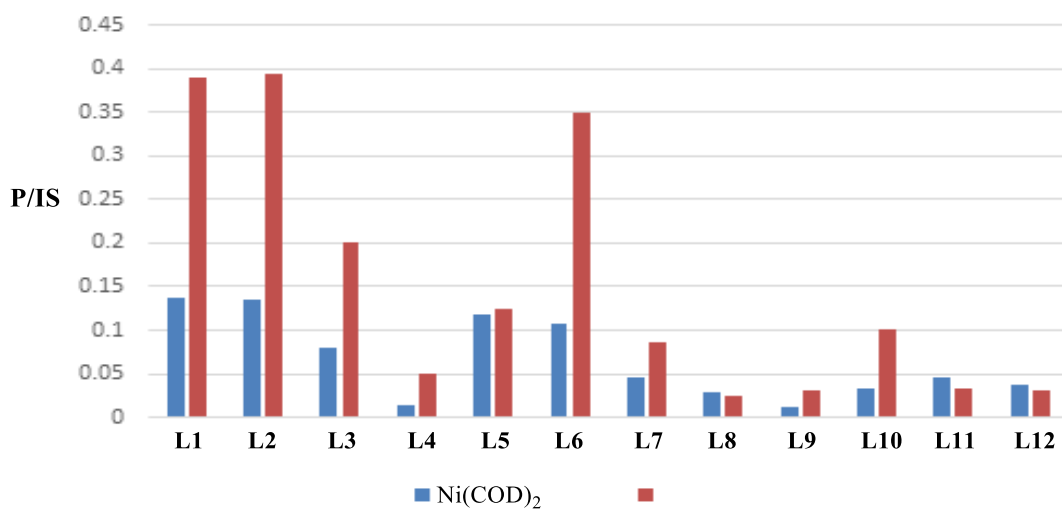


Figure S1. P/IS of the cross-coupling of 2e with potassium cyclohexyltrifluoroborate using the first screen variables.

According to this screen the best results were obtained for the reaction in the presence of $\text{NiCl}_2 \cdot \text{dme}/\text{L1}$ and also $\text{NiCl}_2 \cdot \text{dme}/\text{L2}$.

Second Screen Variables:

solvents	ligands	bases	Ni sources
THF	L1	no base	NiCl_2
	L2		$[(\text{TMEDA})\text{Ni}(o\text{-tolyl})\text{Cl}]$
			$\text{NiCl}_2 \cdot \text{dme}$
			$\text{NiBr}_2 \cdot \text{dme}$
			$\text{Ni}(\text{NO}_3)_2$
			Nickel(II) hexafluoroacetylacetonate
			Nickel(II) acetylacetonate
			$\text{Ni}(\text{CF}_3\text{SO}_3)_2$
			$\text{Ni}(\text{NH}_3)_6\text{Cl}_2$
			$\text{Ni}(\text{TMHD})_2$
			Nickel(II) dimethylglyoxime
			dppeNiCl_2

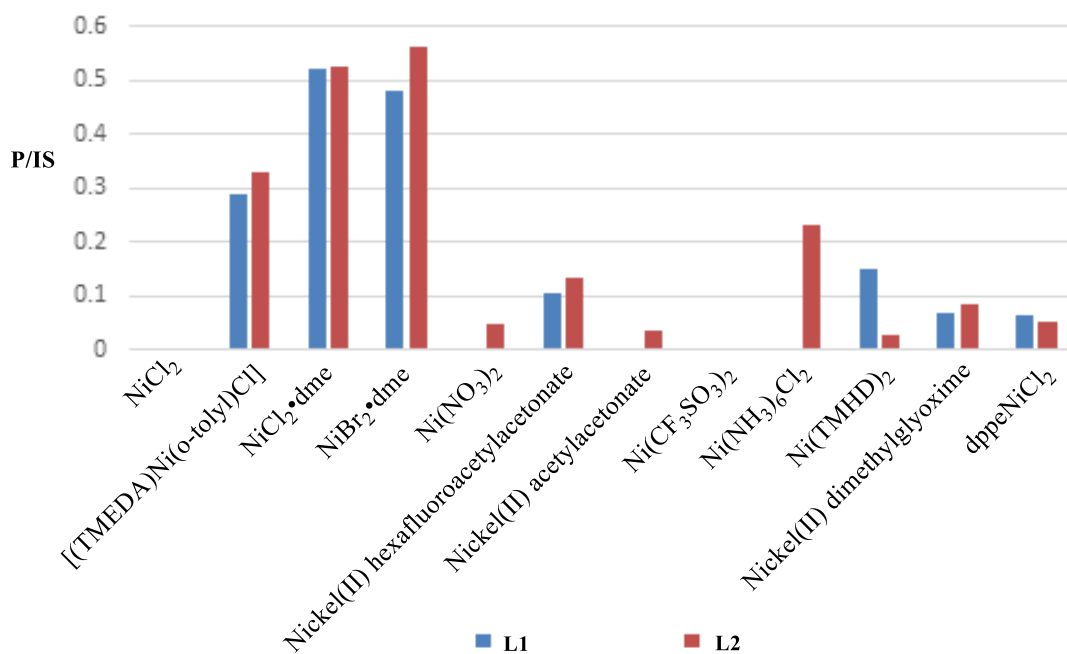


Figure S2. P/IS of the cross-coupling of 2e with potassium cyclohexyltrifluoroborate using the second screen variables.

According to the second screen, the highest product to internal standard ratio was obtained for the reaction in the presence of $\text{NiBr}_2 \cdot \text{dme}/\text{L2}$.

To investigate the effect of the preformed nickel catalysts, **2e** was reacted with potassium cyclohexyltrifluoroborate in the presence of 6 mol % of $[\text{Ni}(\text{dtbbpy})(\text{H}_2\text{O})_4]\text{Cl}_2$ and also $[\text{Ni}(\text{bpy})(\text{H}_2\text{O})_4]\text{Cl}_2$. The P/IS of these reactions were compared with the P/IS of the corresponding reaction in the presence of 6 mol % of $\text{NiBr}_2\cdot\text{dme}/\text{L2}$ (Figure S3).

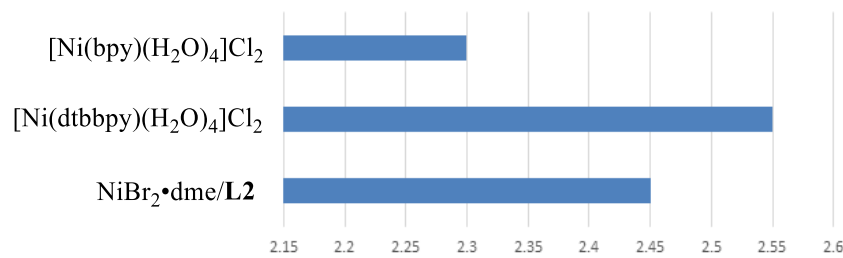


Figure S3. P/IS of the cross-coupling of **2e with potassium cyclohexyltrifluoroborate using $[\text{Ni}(\text{bpy})(\text{H}_2\text{O})_4]\text{Cl}_2$, $[\text{Ni}(\text{dtbbpy})(\text{H}_2\text{O})_4]\text{Cl}_2$, and $\text{NiBr}_2\cdot\text{dme}/\text{L2}$.**

To improve the yield of the reaction further, screens using a variety of inorganic bases as well as solvents were carried out (Figure S4).

Third Screen Variables:

<u>Solvents</u>	<u>bases</u>	<u>Ni sources</u>
THF	no base lutidine NaH_2PO_4 Cs_2CO_3 CsHCO_3 KF K_2CO_3 Na_2CO_3 NaHCO_3 Li_2CO_3 K_2HPO_4 KHCO_3	$[\text{Ni}(\text{dtbbpy})(\text{H}_2\text{O})_4]\text{Cl}_2$

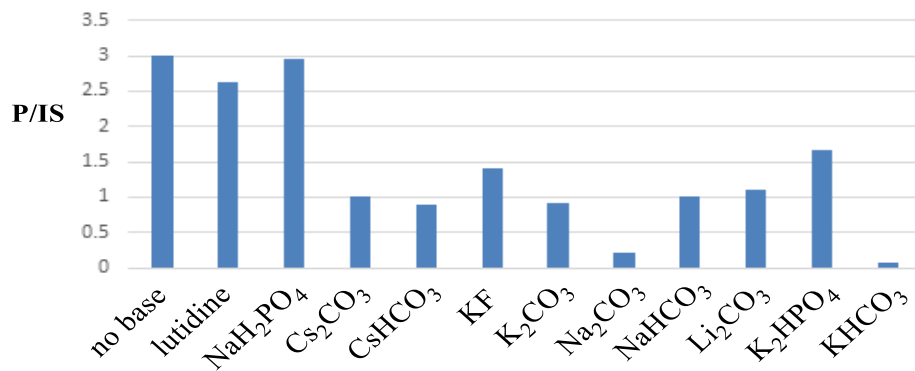


Figure S4. P/IS of the cross-coupling of 2e with potassium cyclohexyltrifluoroborate using different bases.

Fourth Screen Variables:

<u>Solvents</u>	<u>bases</u>	<u>Ni sources</u>
THF	no base	[Ni(dtbbpy)(H ₂ O) ₄]Cl ₂
2-MeTHF		
dioxane		
DME		
DMF		
DMA		
EtOAc		
acetone		

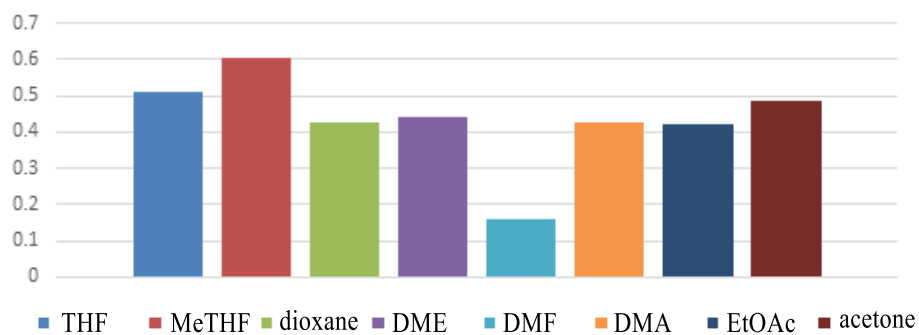


Figure S5. P/IS of the cross-coupling of 2e with potassium cyclohexyltrifluoroborate using different solvents.

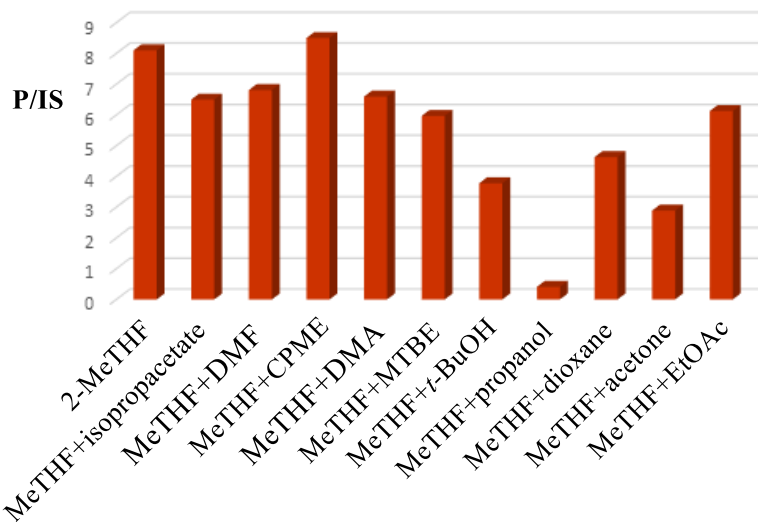
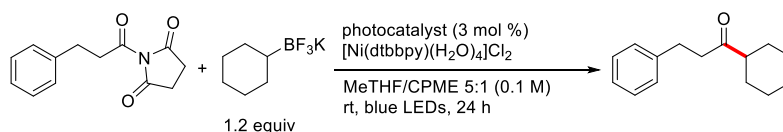


Figure S6. P/IS of the cross-coupling of 2e with potassium cyclohexyltrifluoroborate in 2-MeTHF using different co-solvents with 5:1 ratio.

Accordingly, the best solvent for this reaction is 2-MeTHF/CPME (5:1) that has the highest P/IS.

Control experiments for the cross-coupling of *N*-acylpyrrolidine-2,5-dione with potassium alkyltrifluoroborates:

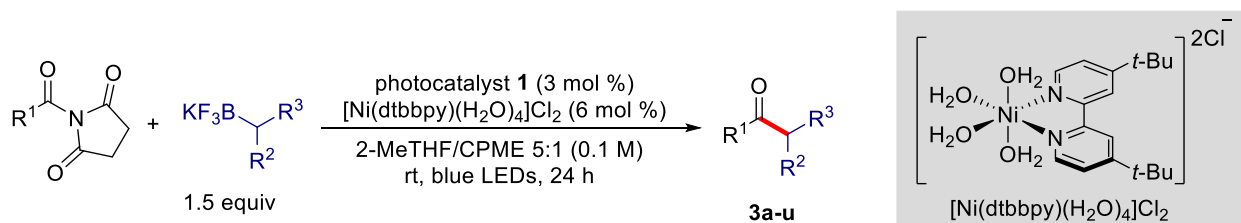
Table S1. Control Experiments.



entry	Conditions ^a	additive	Yield ^c
1	6 mol % [Ni(dtbbpy)(H ₂ O) ₄]Cl ₂	-	70%
2	no Ir photocatalyst	-	<10%
3	no [Ni(dtbbpy)(H ₂ O) ₄]Cl ₂	-	0%
4	[Ru(bpy) ₃](PF ₆) ₃	-	59%
5	4CzIPN ^b	-	21%
5	5 mol % [Ni(dtbbpy)(H ₂ O) ₄]Cl ₂	-	66%
6	4 mol % [Ni(dtbbpy)(H ₂ O) ₄]Cl ₂	-	61%
7	1.1 equiv of R-BF ₃ K	-	68%
8	1.3 equiv of R-BF ₃ K	-	73%
9	1.5 equiv of R-BF ₃ K	-	78%

^a Reactions were carried out using 3 mol % of photocatalyst at 0.1 M. ^b 2,4,5,6-tetrakis(carbazol-9-yl)-1,3-dicyanobenzene. ^c Isolated yields.

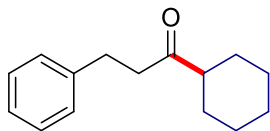
General procedure for the photoredox cross-coupling reaction of *N*-acylpyrrolidine-2,5-dione with potassium alkyltrifluoroborates:



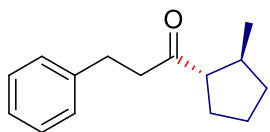
To a two dram (8 mL) borosilicate glass vial equipped with a Teflon-coated magnetic stir bar was added [Ni(dtbbpy)(H₂O)₄]Cl₂ (14.1 mg, 0.3 mmol), the corresponding imide (0.5 mmol), Ir[dFCF₃ppy]₂(bpy)PF₆ **1** (15.1 mg, 0.015 mmol), and potassium alkyltrifluoroborate (0.75 mmol,

1.5equiv). The vial was sealed and subsequently purged and evacuated with Ar four times. A mixture of anhydrous and degassed 2-MeTHF and CPME (5:1, 5 mL) was then added by syringe under Ar. The resulting reaction mixture was stirred for 24 h in the presence of blue LED lamp (Kessil KSH150B Blue LED Grow Light) while a fan was blown across the reaction setup to maintain an ambient temperature of 24 °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.

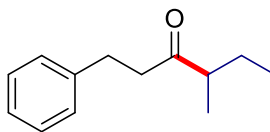
Gram scale reaction: To a ~125 mL long, thin-walled vacuum flask equipped with a Teflon-coated magnetic stir bar was added [Ni(dtbbpy)(H₂O)₄]Cl₂ (0.135 mmol, 63.5 mg), potassium cyclohexyltrifluoroborate (1.28 g, 6.75 mmol, 1.5 equiv), Ir[dFCF₃ppy]₂(bpy)PF₆ **1** (0.066 mmol, 66.6 mg), and 1-(3-phenylpropanoyl)pyrrolidine-2,5-dione (4.5 mmol, 1.04 g). The vial was sealed and subsequently purged and evacuated with Ar four times. A mixture of anhydrous and degassed 2-MeTHF and CPME (5:1, 45 mL) was then 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 °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 product in pure form.



1-Cyclohexyl-3-phenylpropan-1-one (3a).⁸ The title compound was obtained as a liquid in 78% yield (0.5 mmol scale, 84.3 mg) and in 72% yield on gram scale (4.5 mmol scale, 700.9 mg). ¹H NMR (500 MHz, CDCl₃): δ 7.26-7.23 (m, 2H), 7.17-7.15 (m, 3H), 2.86 (t, *J* = 7.5 Hz, 2H), 2.73 (t, *J* = 7.5 Hz, 2H), 2.31-2.26 (m, 1H), 1.80-1.73 (m, 4H), 1.65-1.62 (m, 1H), 1.34-1.27 (m, 5H); ¹³C NMR (125.8 MHz, CDCl₃): δ 213.2, 141.6, 128.6, 128.5, 126.2, 51.2, 42.4, 30.0, 28.6, 26.1, 25.9

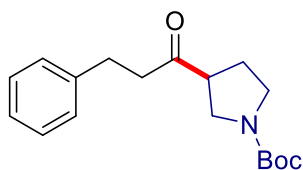


1-(2-Methylcyclopentyl)-3-phenylpropan-1-one (3b).⁹ The title compound was obtained as a liquid in 66% yield (0.5 mmol scale, 71.4 mg). ¹H NMR (500 MHz, CDCl₃): δ 7.27-7.23 (m, 2H), 7.18-7.15 (m, 3H), 2.89 (t, *J* = 7.5 Hz, 2H), 2.76-2.72 (m, 2H), 2.40-2.35 (m, 1H), 2.15-2.10 (m, 1H), 1.86-1.81 (m, 2H), 1.70-1.58 (m, 3H), 1.21-1.13 (m, 1H), 0.97 (d, *J* = 6.5 Hz, 3H); ¹³C NMR (125.8 MHz, CDCl₃): δ 212.7, 141.6, 128.7, 128.6, 126.2, 59.9, 44.1, 37.9, 35.1, 30.0, 29.9, 24.9, 20.3.

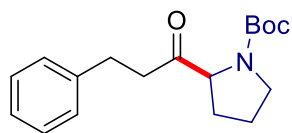


4-Methyl-1-phenylhexan-3-one (3c).¹⁰ The title compound was obtained as a liquid in 70% yield (0.5 mmol scale, 66.6 mg). ¹H NMR (500 MHz, CDCl₃): δ 7.27-7.23 (m, 2H), 7.18-7.15 (m, 3H),

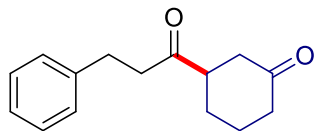
2.88 (t, $J = 7.5$ Hz, 2H), 2.79-2.68 (m, 2H), 2.44-2.38 (m, 1H), 1.69-1.60 (m, 1H), 1.39-1.31 (m, 1H), 1.02 (d, $J = 7.0$ Hz, 3H), 0.83 (t, $J = 7.5$ Hz, 3H); ^{13}C NMR (125.8 MHz, CDCl_3): δ 213.9, 141.5, 128.6, 128.5, 126.2, 48.2, 43.0, 29.9, 26.0, 16.0, 11.8.



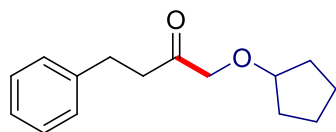
***tert*-Butyl 3-(3-Phenylpropanoyl)pyrrolidine-1-carboxylate (3d).**⁹ The title compound was obtained as a liquid in 70% yield (0.5 mmol scale, 106.2 mg). ^1H NMR (500 MHz, CDCl_3): δ 7.26-7.23 (m, 2H), 7.17-7.13 (m, 3H), 3.49-3.41 (m, 3H), 3.31-3.26 (m, 1H), 3.07-3.01 (m, 1H), 2.91-2.88 (m, 2H), 2.82-2.71 (m, 2H), 2.01-1.97 (m, 2H), 1.44 (s, 9H); ^{13}C NMR (125.8 MHz, CDCl_3): δ 208.5, 154.5, 141.1, 128.7, 128.5, 126.4, 79.5, 47.3, 45.5, 43.4, 29.9, 28.8, 28.7, 28.0.



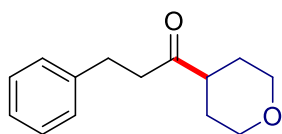
***tert*-Butyl-2-(3-phenylpropanoyl)pyrrolidine-1-carboxylate (3e).** The title compound was obtained as a liquid in 61% yield (0.5 mmol scale, 92.5 mg). ^1H NMR (500 MHz, CD_3CN): δ 7.32-7.29 (m, 2H), 7.26-7.25 (m, 2H), 7.22-7.19 (m, 1H), 4.32 (dd, $J = 8.5, 5.0$ Hz, 1H), 3.51-3.40 (m, 2H), 2.95-2.79 (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 $\text{C}_{18}\text{H}_{25}\text{NO}_3\text{Na}$ $[\text{M}+\text{Na}]^+$ 326.1732, found 326.1711.



3-(3-Phenylpropanoyl)cyclohexan-1-one (3f). The title compound was obtained as a liquid in 58% yield (0.5 mmol scale, 66.7 mg). ^1H NMR (500 MHz, CDCl_3): δ 7.24-7.21 (m, 2H), 7.16-7.11 (m, 3H), 2.87-2.67 (m, 5H), 2.43 (dd, $J = 14.5, 11$ Hz, 1H), 2.33-2.20 (m, 3H), 2.01-1.94 (m, 2H), 1.68-1.55 (m, 2H); ^{13}C NMR (125.8 MHz, CDCl_3): δ 210.0, 209.9, 141.0, 128.7, 128.5, 126.4, 50.5, 42.8, 42.6, 41.1, 29.8, 27.3, 25.0; FT-IR (neat): 1705, 1452, 1224, 749, 699 cm^{-1} ; HRMS (ES+) m/z calcd. for $\text{C}_{15}\text{H}_{19}\text{O}_2$ $[\text{M}+\text{H}]^+$ 231.1385, found 231.1386.

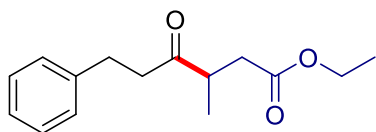


1-(Cyclopentyloxy)-4-phenylbutan-2-one (3g). The title compound was obtained as a liquid in 83% yield (0.5 mmol scale, 96.4 mg). ^1H NMR (500 MHz, CDCl_3): δ 7.27-7.23 (m, 2H), 7.18-7.15 (m, 3H), 3.92 (s, 2H), 3.88-3.84 (m, 1H), 2.89 (t, $J = 7.5$ Hz, 2H), 2.78 (t, $J = 7.5$ Hz, 2H), 1.70-1.63 (m, 6H), 1.53-1.48 (m, 2H); ^{13}C NMR (125.8 MHz, CDCl_3): δ 209.1, 141.2, 128.6, 128.5, 126.3, 82.5, 74.6, 40.8, 32.2, 29.5, 23.6; FT-IR (neat): 1710, 1453, 1107, 1077, 748, 697 cm^{-1} ; HRMS (ES+) m/z calcd. for $\text{C}_{15}\text{H}_{20}\text{O}_2\text{Na}$ $[\text{M}+\text{Na}]^+$ 255.1361, found 255.1360.

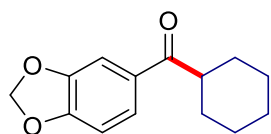


3-Phenyl-1-(tetrahydro-2H-pyran-4-yl)propan-1-one (3h).⁹ The title compound was obtained as a liquid in 80% yield (0.5 mmol scale, 87.3 mg). ^1H NMR (500 MHz, CDCl_3): δ 7.27-7.24 (m,

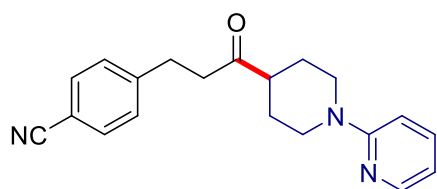
2H), 7.18-7.15 (m, 3H), 3.97-3.93 (m, 2H), 3.37 (dt, $J = 11.5, 3.0$ Hz, 2H), 2.88 (t, $J = 7.5$ Hz, 2H), 2.75 (t, $J = 7.5$ Hz, 2H), 2.49-2.46 (m, 1H), 1.71-1.62 (m, 4H); ^{13}C NMR (125.8 MHz, CDCl_3): δ 211.1, 141.3, 128.7, 128.5, 126.3, 67.4, 47.9, 42.1, 29.8, 28.2.



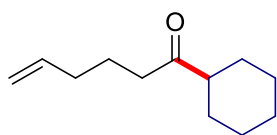
Ethyl 3-methyl-4-oxo-6-phenylhexanoate (3i).¹¹ The title compound was obtained as a liquid in 69% yield (0.5 mmol scale, 85.6 mg). ^1H NMR (500 MHz, CDCl_3): δ 7.28-7.25 (m, 2H), 7.19-7.16 (m, 3H), 4.09 (q, $J = 7.0$ Hz, 2H), 3.00-2.96 (m, 1H), 2.92-2.82 (m, 4H), 2.78-2.73 (m, 1H), 2.30-2.26 (m, 1H), 1.22 (t, $J = 7.0$ Hz, 3H), 1.07 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (125.8 MHz, CDCl_3): δ 212.0, 172.4, 141.4, 128.6, 128.5, 126.2, 60.7, 43.0, 42.3, 37.2, 29.8, 16.7, 14.4.



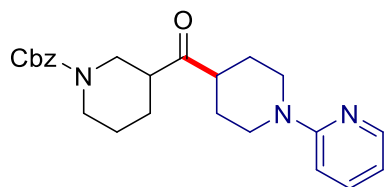
Benzo[d][1,3]dioxol-5-yl(cyclohexyl)methanone (3j).⁹ The title compound was obtained as a white solid in 82% yield (0.5 mmol scale, 95.2 mg). mp 76-77 °C; ^1H NMR (500 MHz, CDCl_3): δ 7.52 (dd, $J = 8.0, 1.5$ Hz, 1H), 7.39 (d, $J = 1.5$ Hz, 1H), 6.81 (d, $J = 8.5$ Hz, 1H), 5.98 (s, 2H), 3.16-3.10 (m, 1H), 1.83-1.78 (m, 4H), 1.71-1.68 (m, 1H), 1.50-1.18 (m, 5H); ^{13}C NMR (125.8 MHz, CDCl_3): δ 202.1, 151.7, 148.4, 131.3, 124.5, 108.4, 108.1, 102.0, 45.7, 29.8, 26.2, 26.1.



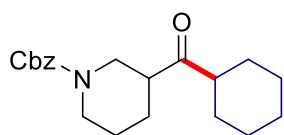
4-(3-Oxo-3-(1-(pyridin-2-yl)piperidin-4-yl)propyl)benzonitrile (3k). The title compound was obtained as a yellow solid in 84% yield (0.5 mmol scale, 134.1 mg). mp 94-96 °C; ¹H NMR (500 MHz, CDCl₃): δ 8.16 (dd, *J* = 5.0, 1.0 Hz, 1H), 7.56 (dd, *J* = 6.5, 1.5 Hz, 2H), 7.47-7.43 (m, 1H), 7.29-7.28 (m, 2H), 6.64 (d, *J* = 8.5 Hz, 1H), 6.60-6.58 (m, 1H), 4.28 (td, *J* = 13.5, 3.0 Hz, 2H), 2.96 (t, *J* = 7.5 Hz, 2H), 2.90-2.85 (m, 2H), 2.82 (t, *J* = 7.0 Hz, 2H), 2.55-2.49 (m, 1H), 1.89-1.86 (m, 2H), 1.68-1.59 (m, 2H); ¹³C NMR (125.8 MHz, CDCl₃): δ 210.5, 159.3, 148.1, 147.0, 137.6, 132.4, 129.3, 119.0, 113.2, 110.2, 107.4, 49.2, 45.1, 41.3, 29.6, 27.2; FT-IR (neat): 2225, 1705, 1593, 1480, 1436, 976, 772 cm⁻¹; HRMS (ES⁺) *m/z* calcd. for C₂₀H₂₂N₃O [M+H]⁺ 320.1763, found 320.1750.



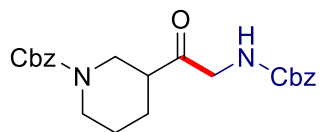
1-Cyclohexylhex-5-en-1-one (3l). The title compound was obtained as a liquid in 82% yield (0.5 mmol scale, 73.9 mg). ¹H NMR (500 MHz, CDCl₃): δ 5.77-5.69 (m, 1H), 4.99-4.92 (m, 2H), 2.40 (t, *J* = 7.0 Hz, 2H), 2.31-2.27 (m, 1H), 2.04-1.99 (m, 2H), 1.80-1.72 (m, 4H), 1.66-1.60 (m, 3H), 1.31-1.15 (m, 5H); ¹³C NMR (125.8 MHz, CDCl₃): δ 214.3, 138.4, 115.3, 51.1, 39.9, 33.4, 28.7, 26.1, 25.9, 22.9; FT-IR (neat): 2928, 2854, 1705, 1449, 996, 909 cm⁻¹; HRMS (ES⁺) *m/z* calcd. for C₁₂H₂₁O [M+H]⁺ 181.1592, found 181.1590.



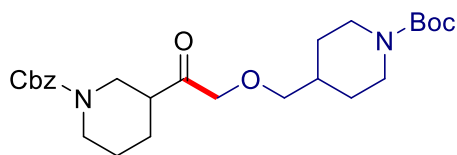
Benzyl 3-(1-(pyridin-2-yl)piperidine-4-carbonyl)piperidine-1-carboxylate (3m). The title compound was obtained as a liquid in 78% yield (0.5 mmol scale, 158.9 mg). ^1H NMR (500 MHz, CDCl_3): δ 8.15-8.14 (m, 1H), 7.42-7.39(m, 1H), 7.33-7.25 (m, 5H), 6.60 (d, J = 8.5 Hz, 1H), 6.55-6.53 (m, 1H), 5.12 (dd, J = 15.5, 12.5 Hz, 2H), 4.24 (t, J = 12.5 Hz, 2H), 4.13 (d, J = 12.0 Hz, 1H), 4.02 (d, J = 13.0 Hz, 1H), 2.97-2.81 (m, 4H), 2.70-2.64 (m, 2H), 1.91-1.82 (m, 3H), 1.73-1.46 (m, 5H); ^{13}C NMR (125.8 MHz, CDCl_3): δ 212.5, 159.5, 155.4, 148.2, 137.5, 137.4, 137.1, 128.7, 128.2, 128.0, 113.1, 107.4, 107.3, 67.4, 47.9, 46.9, 46.2, 45.1, 44.6, 27.3, 24.6; FT-IR (neat): 1734, 1693, 1593, 1480, 1433, 1233, 1144, 766, 697 cm^{-1} ; HRMS (ES+) m/z calcd. for $\text{C}_{24}\text{H}_{30}\text{N}_3\text{O}_3$ $[\text{M}+\text{H}]^+$ 408.2287, found 408.2282.



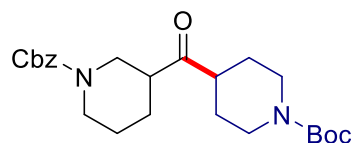
Benzyl 3-(cyclohexanecarbonyl)piperidine-1-carboxylate (3n). The title compound was obtained as a liquid in 80% yield (0.5 mmol scale, 131.8 mg). ^1H NMR (500 MHz, CDCl_3): δ 7.32-7.26 (m, 5H), 5.12 (dd, J = 18.5, 12.5 Hz, 2H), 4.15-4.03 (m, 2H), 2.91-2.77 (m, 2H), 2.68-2.64 (m, 1H), 2.46-2.42 (m, 1H), 1.90-1.87 (m, 1H), 1.79-1.63 (m, 6H), 1.55-1.45 (m, 2H), 1.34-1.17 (m, 5H); ^{13}C NMR (125.8 MHz, CDCl_3): δ 213.9, 155.4, 137.1, 128.6, 128.1, 128.0, 67.3, 49.9, 47.0, 46.3, 44.6, 28.6, 27.3, 26.0, 25.8, 24.7; FT-IR (neat): 1693, 1427, 1256, 1232, 1148, 732, 696 cm^{-1} ; HRMS (ES+) m/z calcd. for $\text{C}_{20}\text{H}_{28}\text{NO}_3$ $[\text{M}+\text{H}]^+$ 330.2069, found 330.2071.



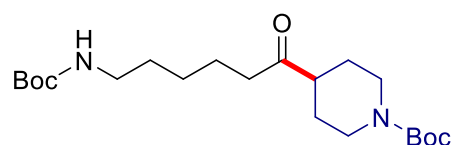
Benzyl 3-(((benzyloxy)carbonyl)glycyl)piperidine-1-carboxylate (3o). The title compound was obtained as a liquid in 70% yield (0.5 mmol scale, 143.6 mg). ^1H NMR (500 MHz, CDCl_3): δ 7.32-7.25 (m, 10H), 5.42 (s, 1H), 5.11-5.08 (m, 4H), 4.13-4.01 (m, 3H), 3.95-3.92 (m, 1H), 3.06 (dd, $J = 13.0, 10.5$ Hz, 1H), 2.90-2.85 (m, 1H), 2.54-2.50 (m, 1H), 1.93-1.90 (m, 1H), 1.71-1.67 (m, 1H), 1.62-1.54 (m, 1H), 1.49-1.41 (m, 1H); ^{13}C NMR (125.8 MHz, CDCl_3): δ 205.8, 156.6, 155.3, 137.0, 136.7, 128.7, 128.3, 128.2, 128.1, 67.5, 67.2, 49.6, 46.3, 45.6, 44.5, 27.1, 24.3; FT-IR (neat): 3340, 1730, 1694, 1430, 1234, 1149, 1043, 696 cm^{-1} ; HRMS (ES+) m/z calcd. for $\text{C}_{23}\text{H}_{27}\text{N}_2\text{O}_5$ $[\text{M}+\text{H}]^+$ 411.1920, found 411.1929.



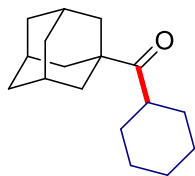
Benzyl 3-(2-(((1-(tert-butoxycarbonyl)piperidin-4-yl)methoxy)acetyl)piperidine-1-carboxylate (3p). The title compound was obtained as a liquid in 80% yield (0.5 mmol scale, 189.8 mg). ^1H NMR (500 MHz, CDCl_3): δ 7.33-7.27 (m, 5H), 5.11 (dd, $J = 18.0, 12.5$ Hz, 2H), 4.08-4.03 (m, 5H), 3.93-3.91 (m, 1H), 2.29 (d, $J = 6.0$ Hz, 2H), 3.14-3.09 (m, 1H), 2.97-2.92 (m, 1H), 2.76-2.67 (m, 3H), 1.93-1.90 (m, 1H), 1.75-1.59 (m, 5H), 1.49-1.42 (m, 10H), 1.19-1.11 (m, 2H); ^{13}C NMR (125.8 MHz, CDCl_3): δ 209.0, 155.3, 155.0, 137.1, 128.6, 128.2, 128.0, 79.4, 76.7, 75.6, 67.4, 45.5, 45.0, 44.6, 43.8, 36.7, 29.1, 28.9, 28.8, 28.7, 26.7, 24.4; FT-IR (neat): 1736, 1688, 1467, 1423, 1233, 1166, 1137, 698 cm^{-1} ; HRMS (ES+) m/z calcd. for $\text{C}_{26}\text{H}_{39}\text{N}_2\text{O}_6$ $[\text{M}+\text{H}]^+$ 475.2808, found 475.2820.



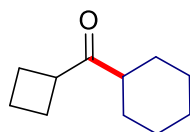
Benzyl 3-(1-(*tert*-butoxycarbonyl)piperidine-4-carbonyl)piperidine-1-carboxylate (3q). The title compound was obtained as a liquid in 75% yield (0.5 mmol scale, 161.4 mg). ^1H NMR (500 MHz, CDCl_3): δ 7.32-7.26 (m, 5H), 5.12 (dd, $J = 18.0, 12.5$ Hz, 2H), 4.11-4.00 (m, 4H), 2.97-2.92 (m, 1H), 2.87-2.81 (m, 1H), 2.75 (t, $J = 12.5$ Hz, 2H), 2.69-2.56 (m, 2H), 1.89-1.87 (m, 1H), 1.73-1.70 (m, 3H), 1.59-1.41 (m, 13H); ^{13}C NMR (125.8 MHz, CDCl_3): δ 212.2, 155.3, 154.7, 137.0, 128.6, 128.1, 128.0, 79.6, 67.3, 47.5, 46.8, 46.1, 44.6, 43.4, 28.7, 28.6, 27.6, 24.5; FT-IR (neat): 1737, 1688, 1422, 1365, 1232, 1150, 976, 763 cm^{-1} ; HRMS (ES+) m/z calcd. for $\text{C}_{24}\text{H}_{35}\text{N}_2\text{O}_5$ $[\text{M}+\text{H}]^+$ 431.2546, found 431.2555.



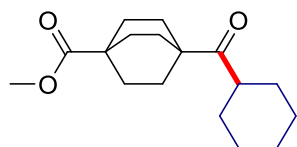
***tert*-Butyl 4-(6-((*tert*-butoxycarbonyl)amino)hexanoyl)piperidine-1-carboxylate (3r).** The title compound was obtained as a liquid in 78% yield (0.5 mmol scale, 155.4 mg). ^1H NMR (500 MHz, CDCl_3): δ 6.46 (s, 1H), 4.04 (d, $J = 13.5$ Hz, 2H), 3.07 (dd, $J = 13.0, 7.0$ Hz, 2H), 2.79-2.74 (m, 2H), 2.42-2.39 (m, 3H), 1.76-1.73 (m, 2H), 1.58-1.38 (m, 24H), 1.31-1.25 (m, 2H); ^{13}C NMR (125.8 MHz, CDCl_3): δ 211.9, 156.3, 155.0, 79.8, 79.3, 48.9, 43.6, 40.6, 30.2, 28.8, 28.7, 27.9, 26.7, 23.6; FT-IR (neat): 1689, 1517, 1236, 1163, 1133, 769 cm^{-1} ; HRMS (ES+) m/z calcd. for $\text{C}_{21}\text{H}_{39}\text{N}_2\text{O}_5$ $[\text{M}+\text{H}]^+$ 399.2859, found 399.2833.



(Adamantan-1-yl)(cyclohexyl)methanone (3s).⁹ The title compound was obtained as a white solid in 75% yield (0.5 mmol scale, 92.4 mg). mp 80-81 °C; ¹H NMR (500 MHz, CDCl₃): δ 2.84-2.78 (m, 1H), 2.00 (s, 3H), 1.77-1.64 (m, 15H), 1.54-1.52 (m, 2H), 1.36-1.18 (m, 5H); ¹³C NMR (125.8 MHz, CDCl₃): δ 218.4, 47.0, 44.1, 37.9, 36.8, 29.9, 28.1, 26.0.



Cyclobutyl(cyclohexyl)methanone (3t).¹² The title compound was obtained as a liquid in 79% yield (0.5 mmol scale, 65.6 mg). ¹H NMR (500 MHz, CDCl₃): δ 3.39-3.32 (m, 1H), 2.33-2.27 (m, 1H), 2.23-2.15 (m, 2H), 2.09-2.02 (m, 2H), 1.96-1.89 (m, 1H), 1.81-1.71 (m, 5H), 1.63-1.60 (m, 1H), 1.32-1.14 (m, 5H); ¹³C NMR (125.8 MHz, CDCl₃): δ 214.9, 49.0, 43.9, 28.7, 26.1, 25.9, 24.7, 18.0.

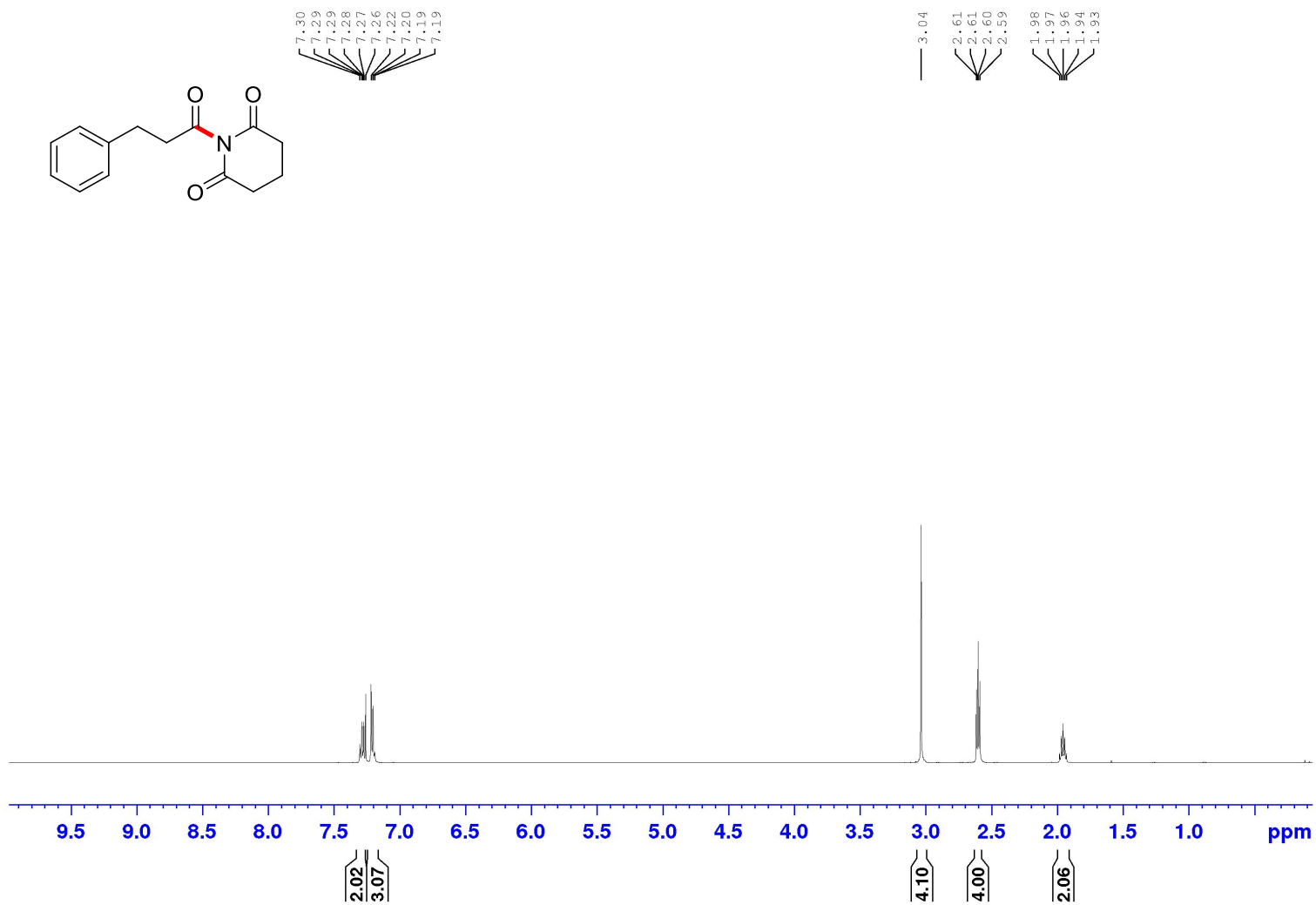


Methyl 4-(cyclohexanecarbonyl)bicyclo[2.2.2]octane-1-carboxylate (3u). The title compound was obtained as a white solid in 85% yield (0.5 mmol scale, 118.3 mg). mp 104-105 °C; ¹H NMR (500 MHz, CDCl₃): δ 3.59 (s, 3H), 2.75-2.69 (m, 1H), 1.77-1.73 (m, 6H), 1.70-1.67 (m, 8H), 1.61-1.60 (m, 1H), 1.53-1.50 (m, 2H), 1.31-1.14 (m, 5H); ¹³C NMR (125.8 MHz, CDCl₃): δ 217.9, 178.0, 51.9, 51.8, 45.4, 45.1, 39.1, 29.7, 27.9, 26.7, 25.8; FT-IR (neat): 1724, 1686, 1449, 1253,

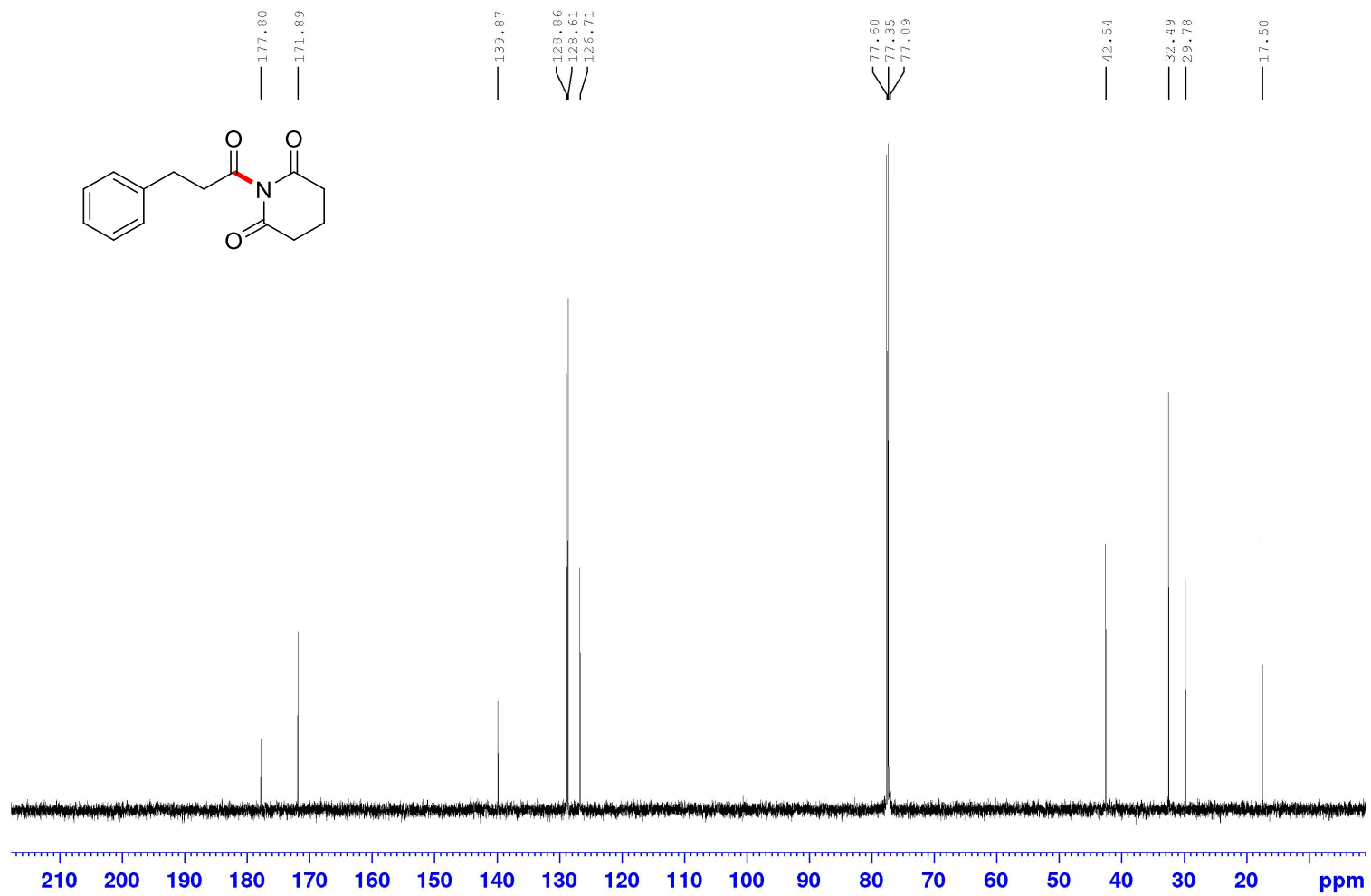
1236, 1079, 1010, 854 cm^{-1} ; HRMS (ES+) m/z calcd. for $\text{C}_{17}\text{H}_{27}\text{O}_3$ $[\text{M}+\text{H}]^+$ 279.1960, found 279.1966.

References:

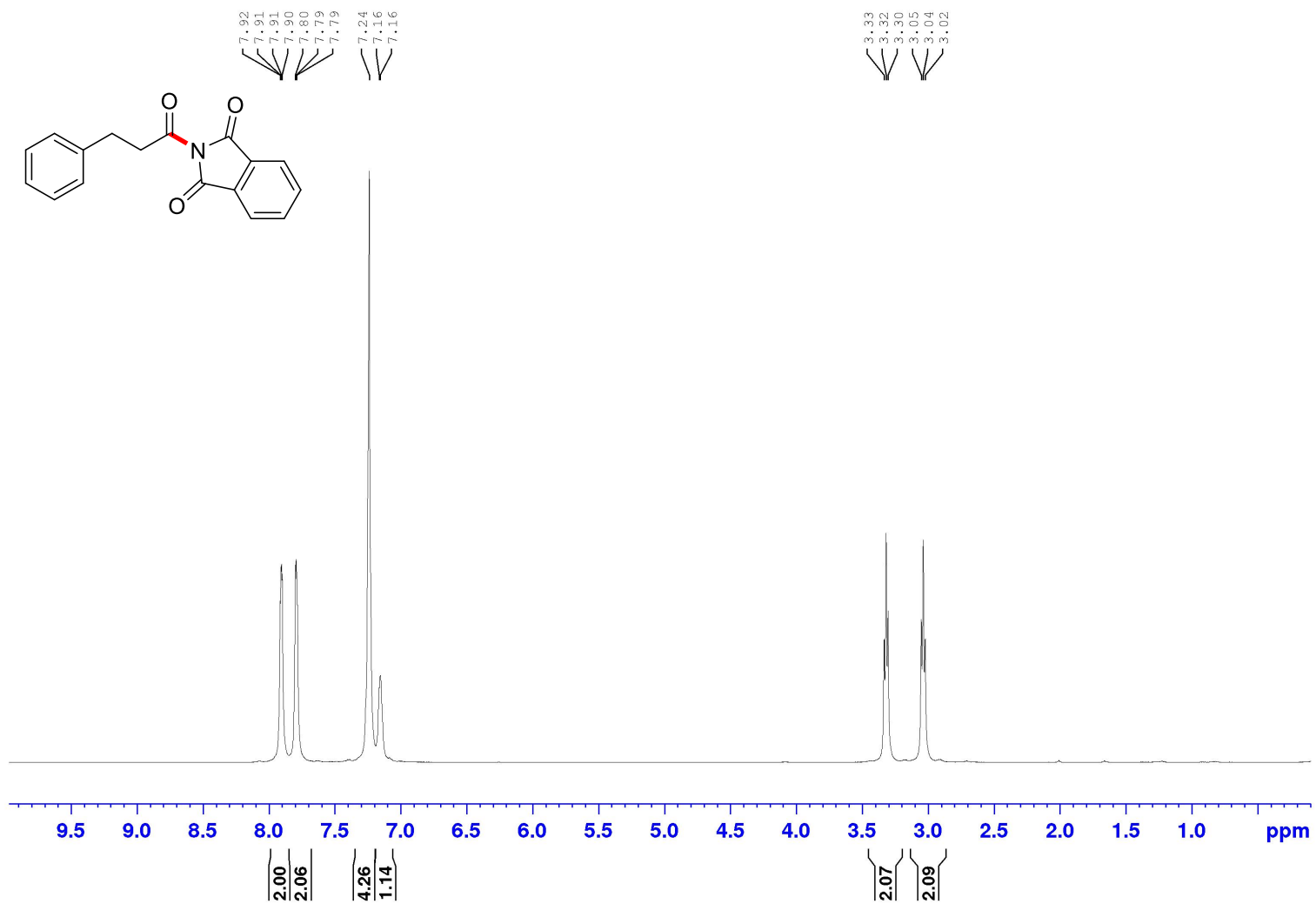
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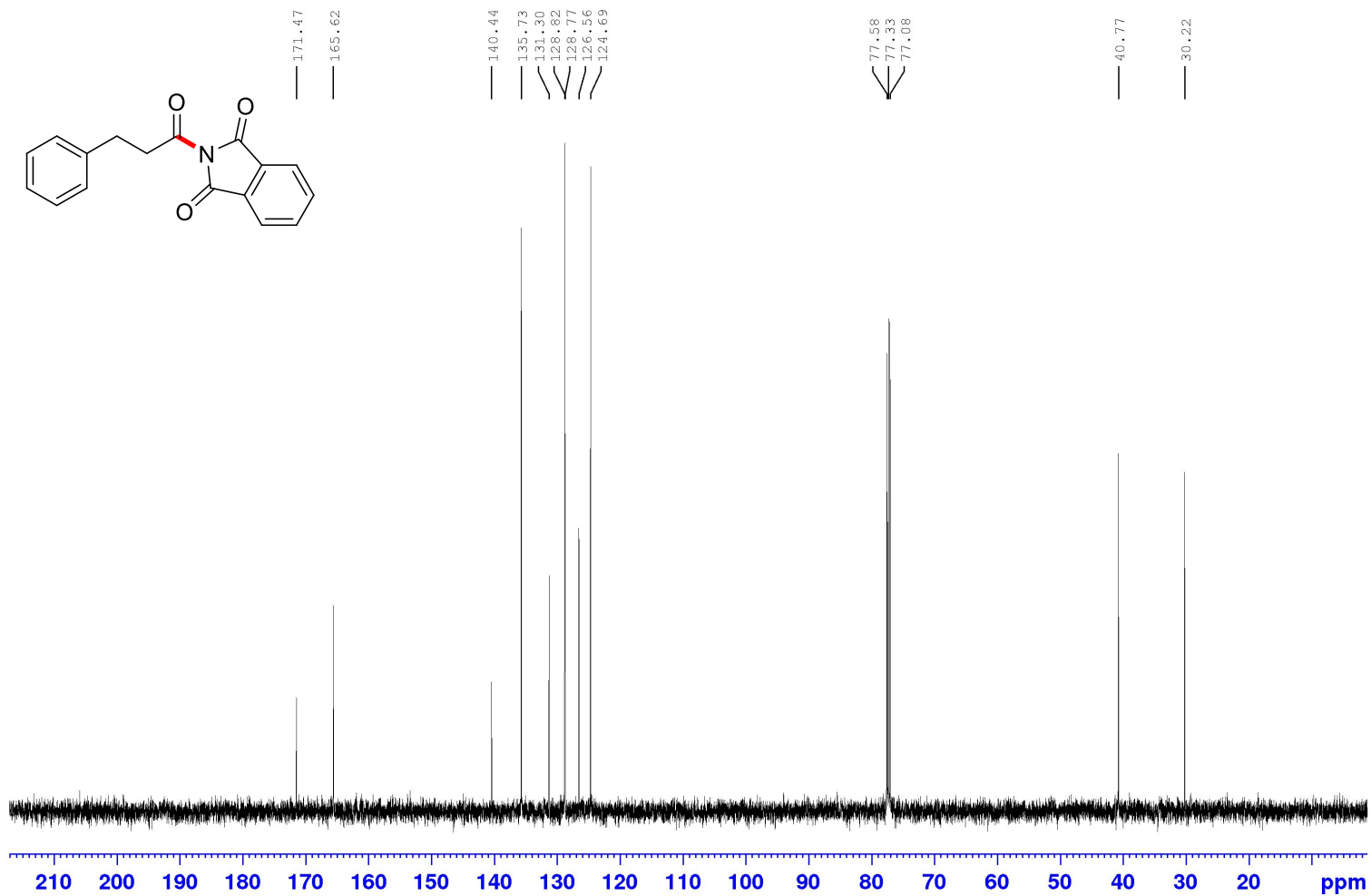
¹H NMR (500 MHz, CDCl₃) Spectrum of 1-(3-phenylpropanoyl)piperidine-2,6-dione (2d)



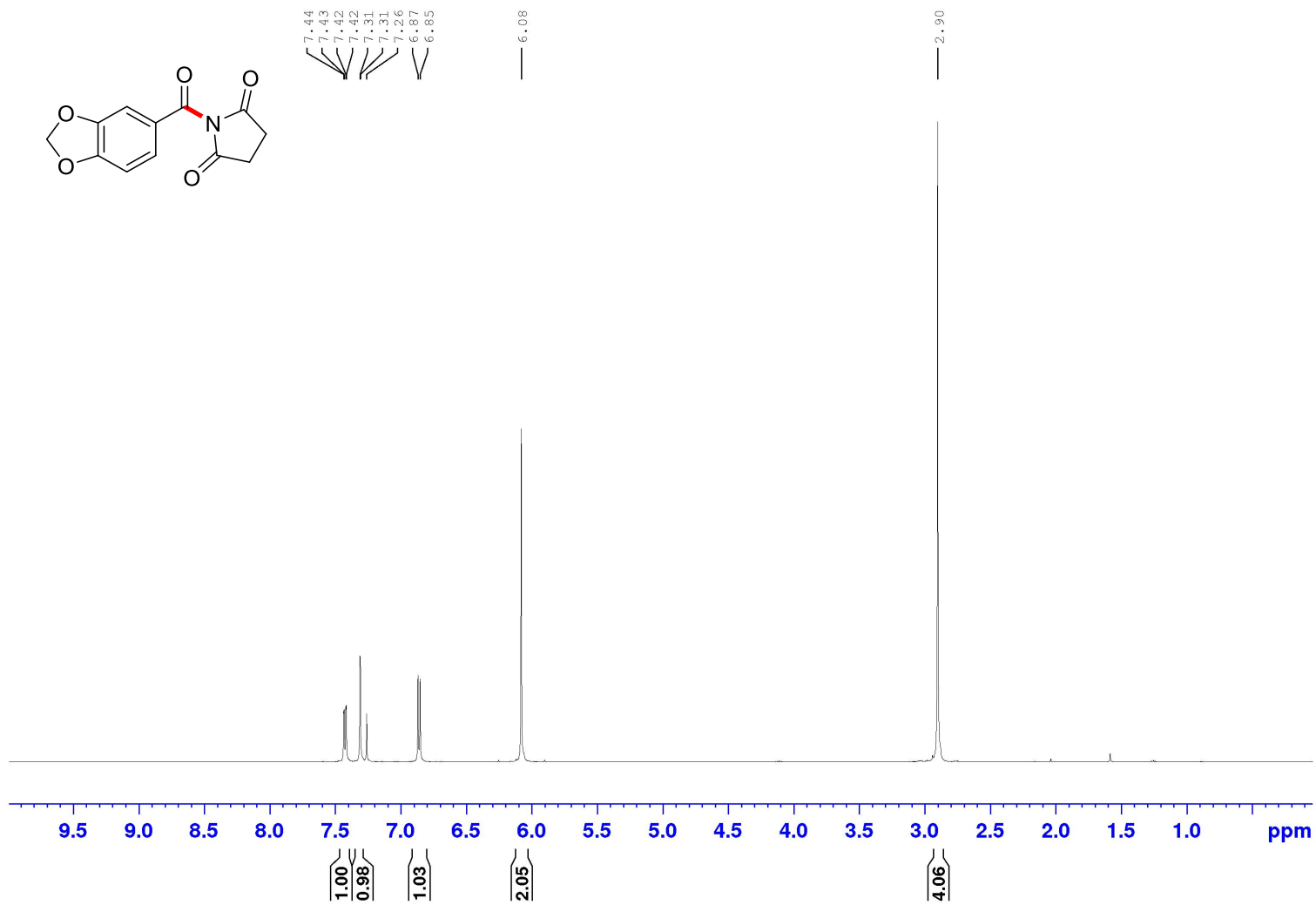
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of 1-(3-phenylpropanoyl)piperidine-2,6-dione (2d)



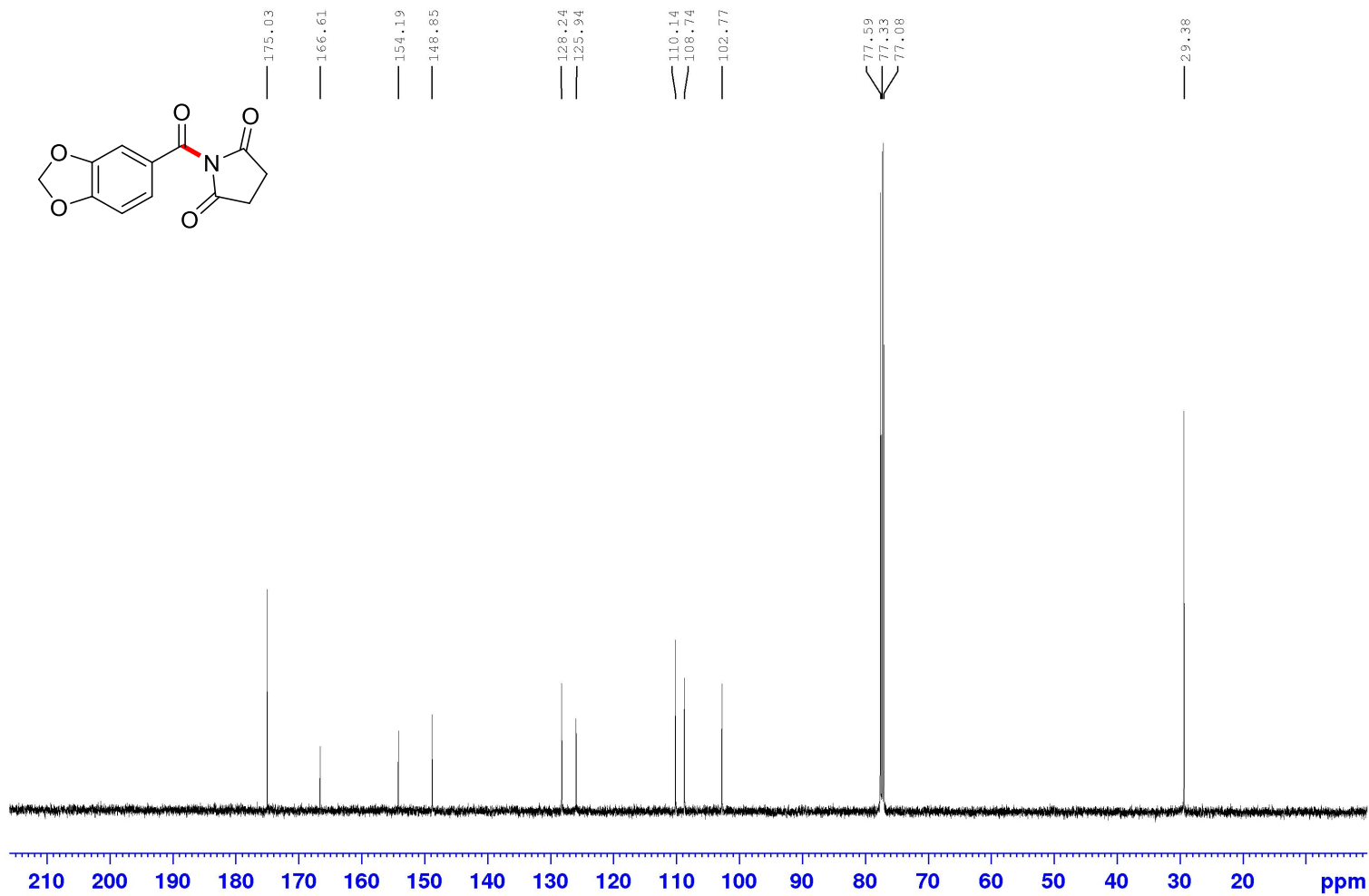
¹H NMR (500 MHz, CDCl₃) Spectrum of 2-(3-phenylpropanoyl)isoindoline-1,3-dione (2f)



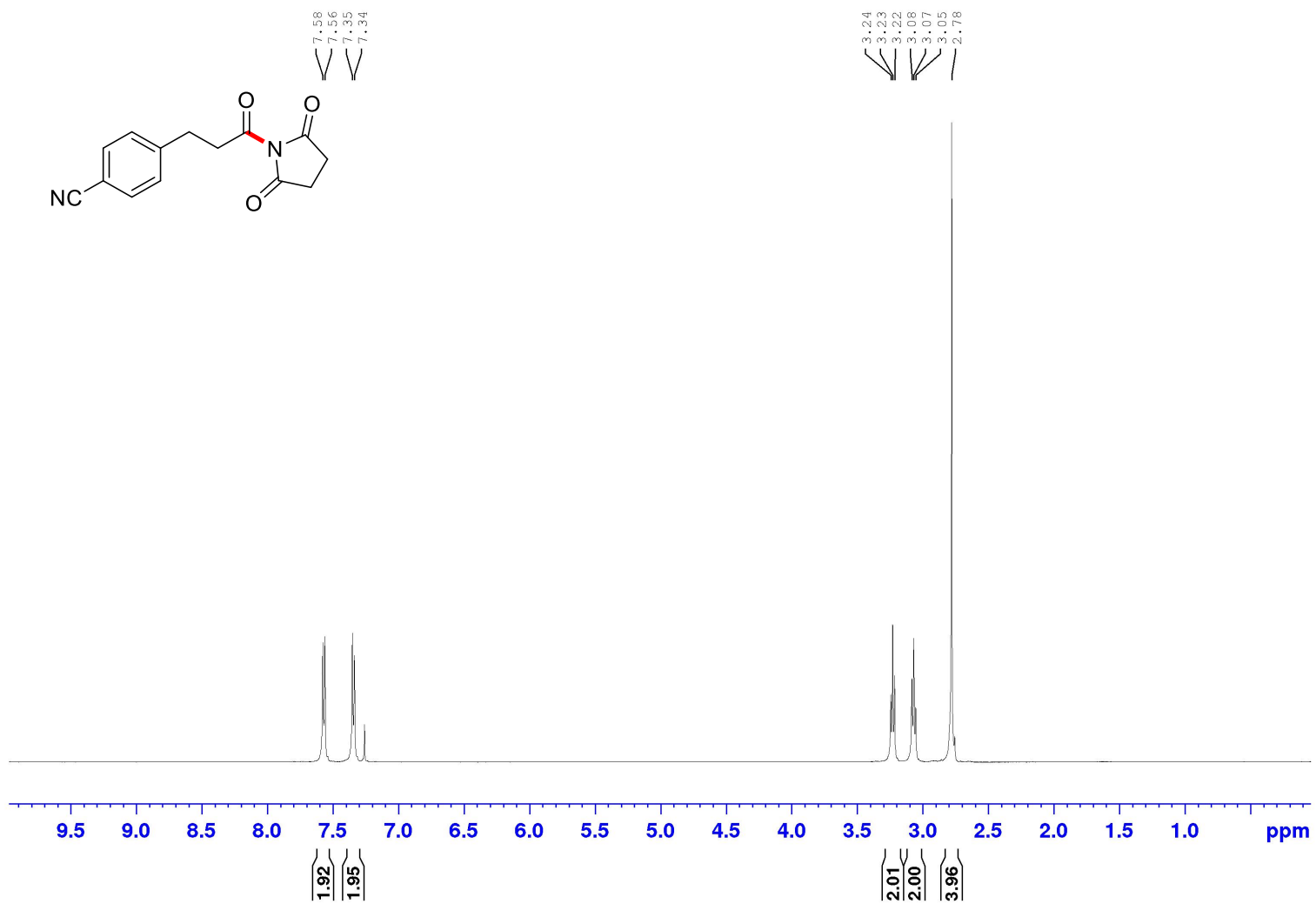
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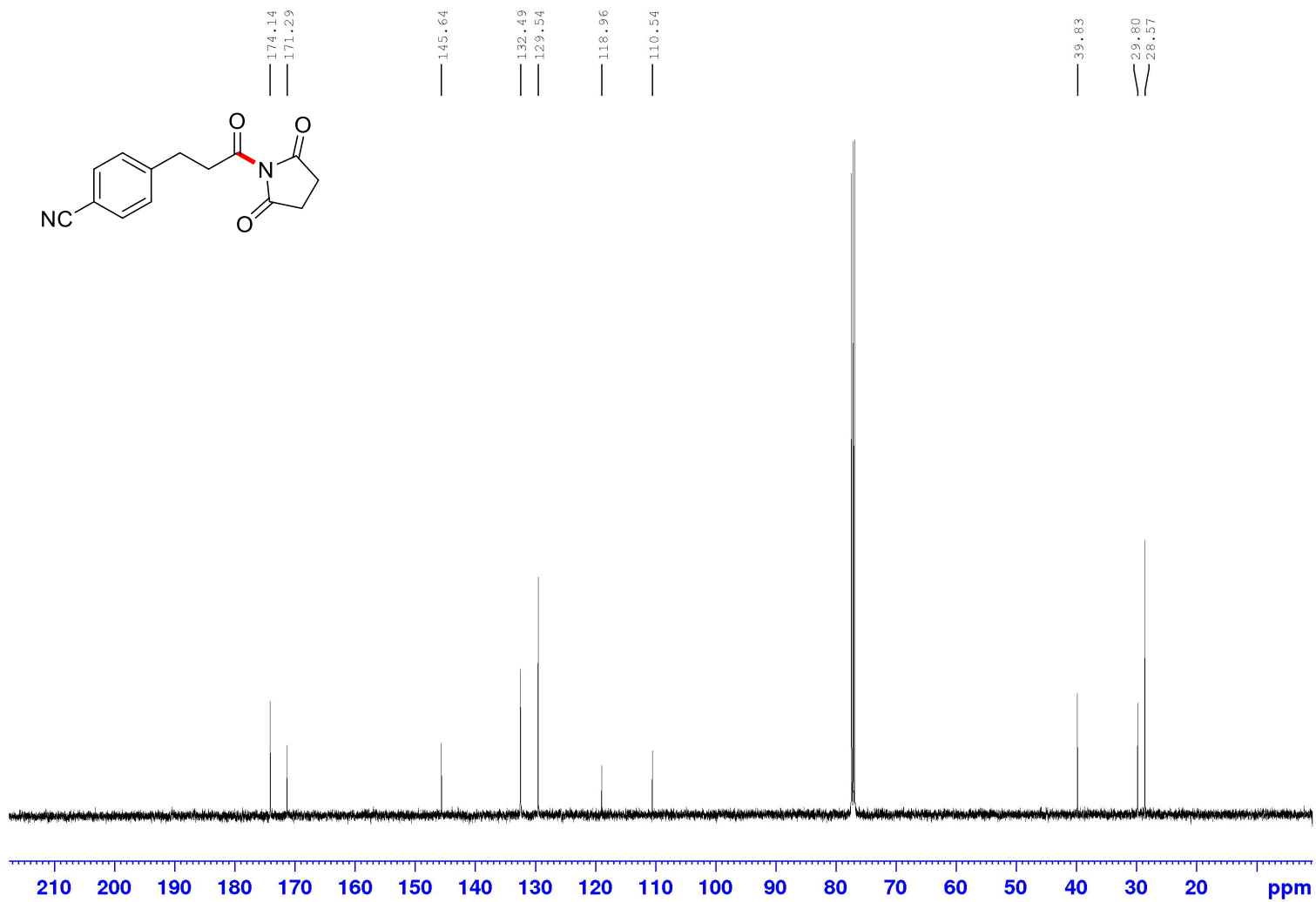
¹H NMR (500 MHz, CDCl₃) Spectrum of 1-(benzo[d][1,3]dioxole-5-carbonyl)pyrrolidine-2,5-dione



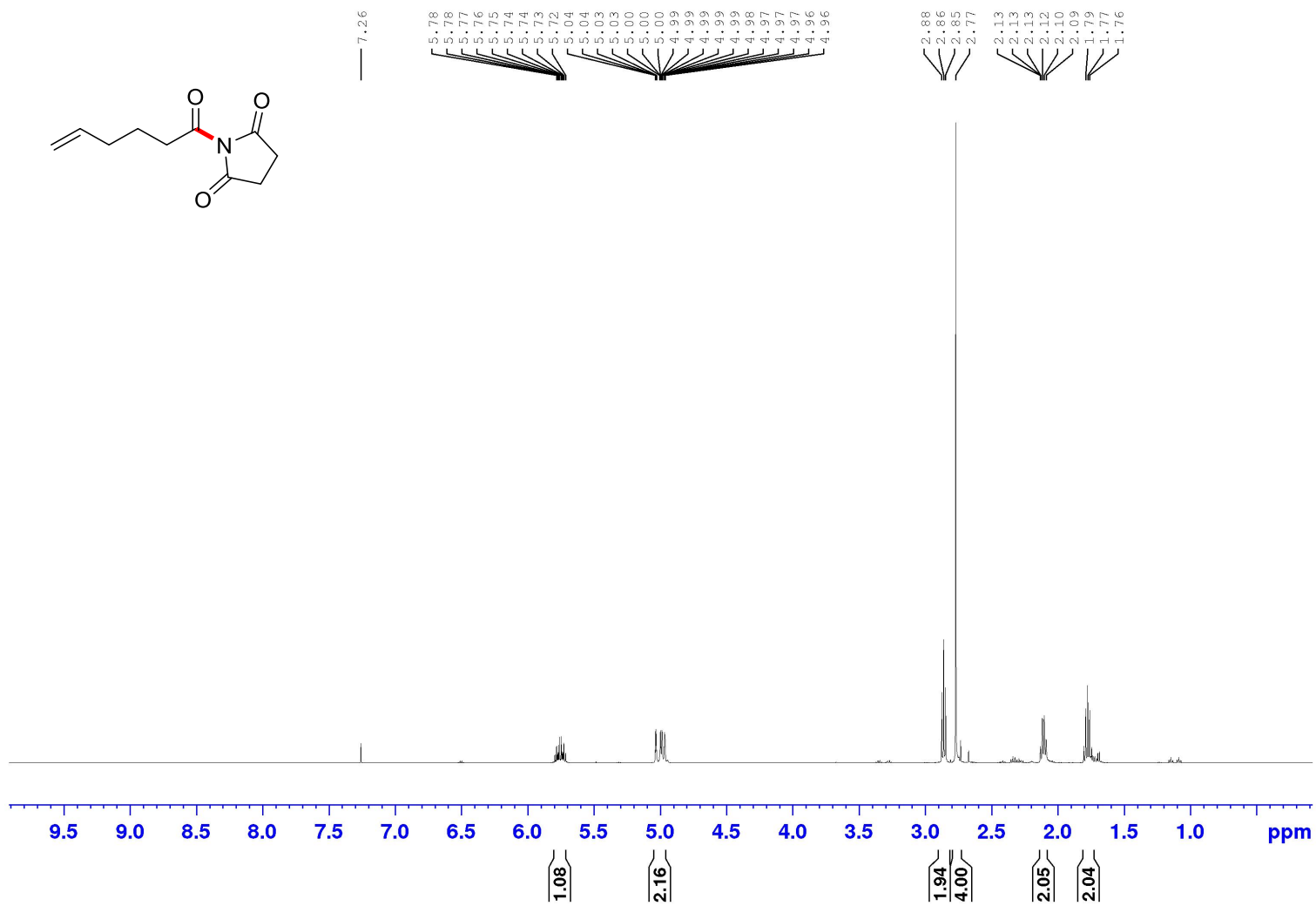
^{13}C NMR (125.8 MHz, CDCl_3) Spectrum of 1-(benzo[d][1,3]dioxole-5-carbonyl)pyrrolidine-2,5-dione



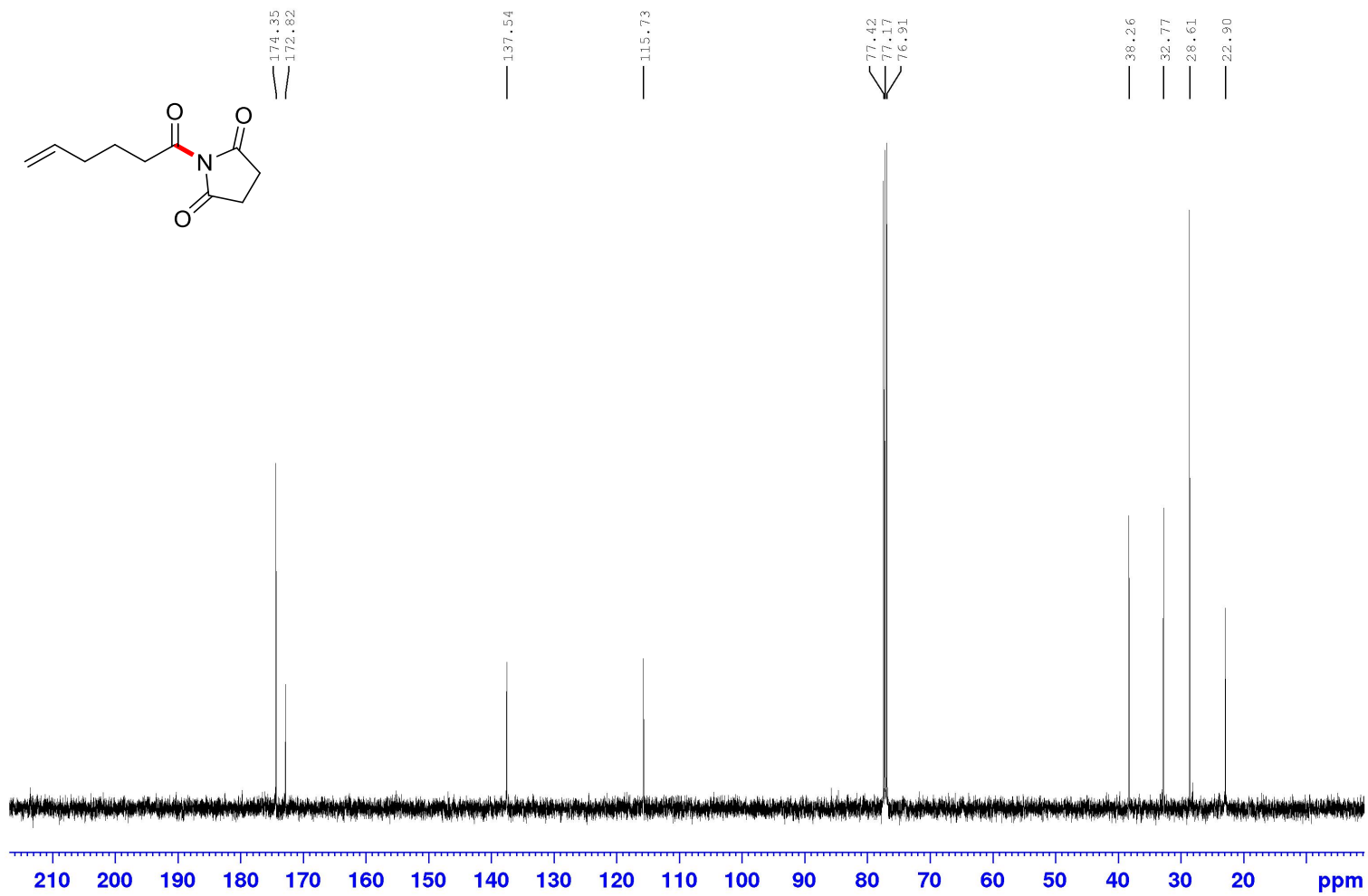
¹H NMR (500 MHz, CDCl₃) Spectrum of 4-(3-(2,5-dioxopyrrolidin-1-yl)-3-oxopropyl)benzonitrile



¹³C NMR (125.8 MHz, CDCl₃) Spectrum of 4-(3-(2,5-dioxopyrrolidin-1-yl)-3-oxopropyl)benzonitrile

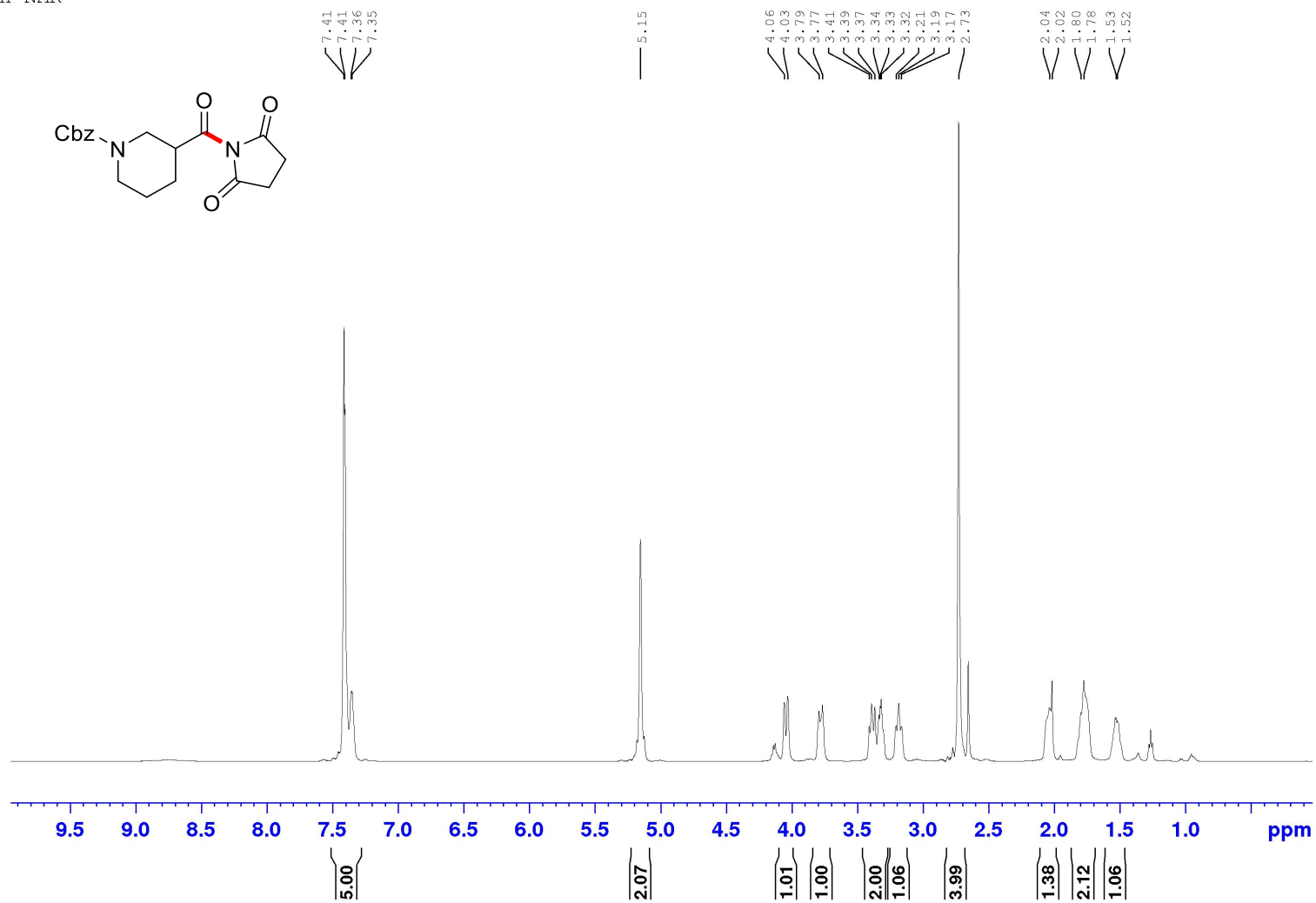


¹H NMR (500 MHz, CDCl₃) Spectrum of 1-(hex-5-enoyl)pyrrolidine-2,5-dione



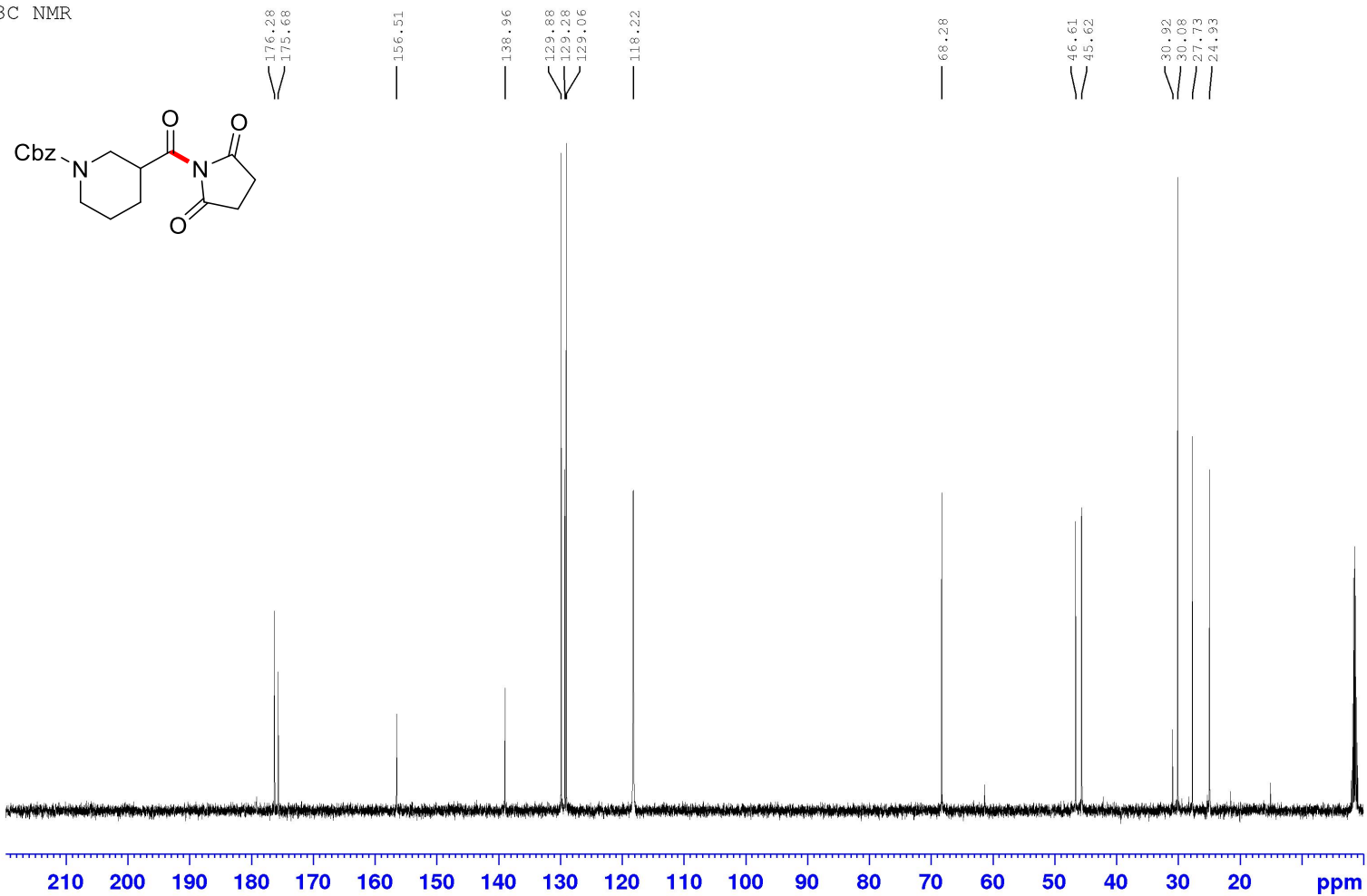
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¹H NMR

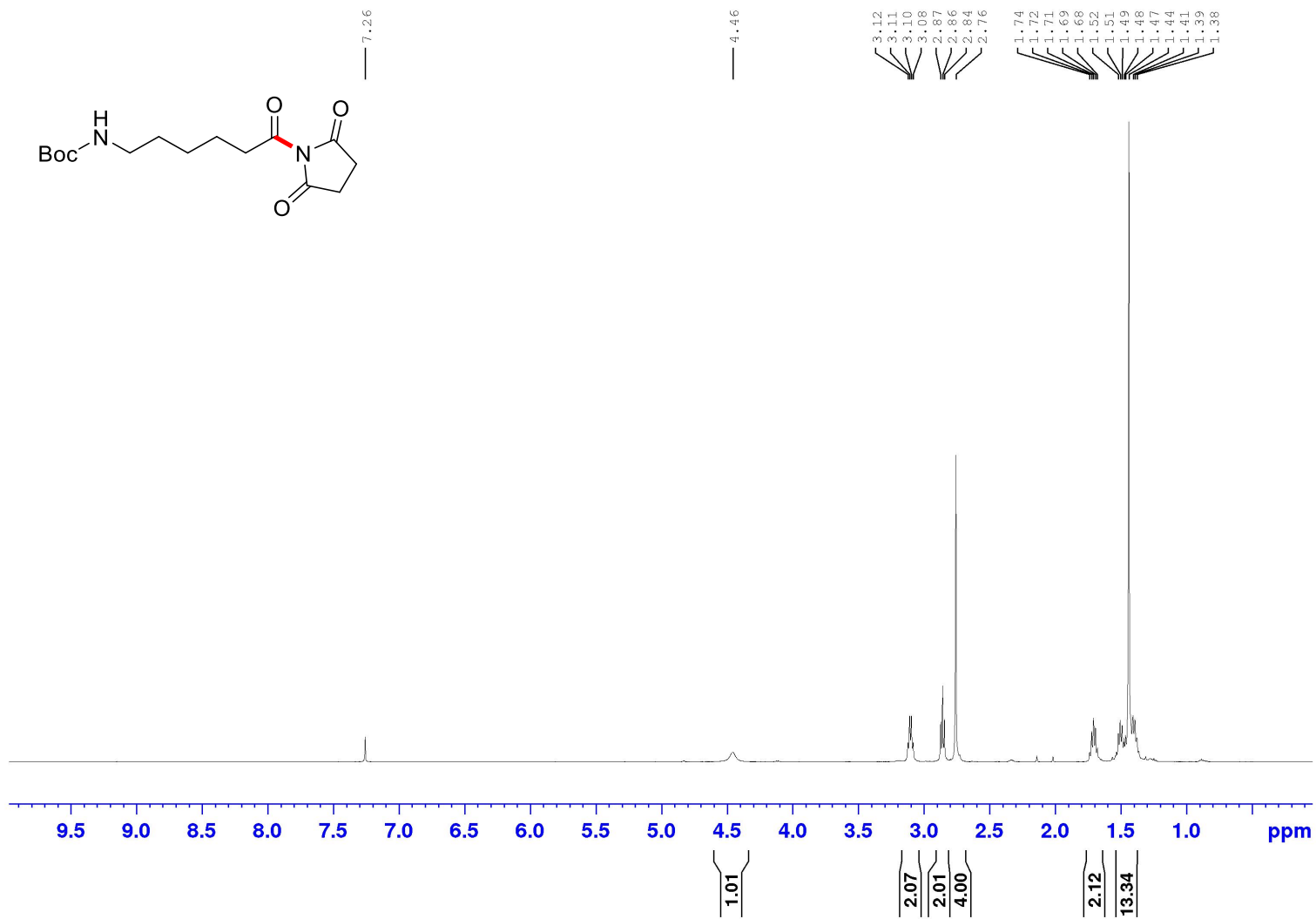


¹H NMR (500 MHz, CDCl₃) Spectrum of benzyl 3-(2,5-dioxopyrrolidine-1-carbonyl)piperidine-1-carboxylate

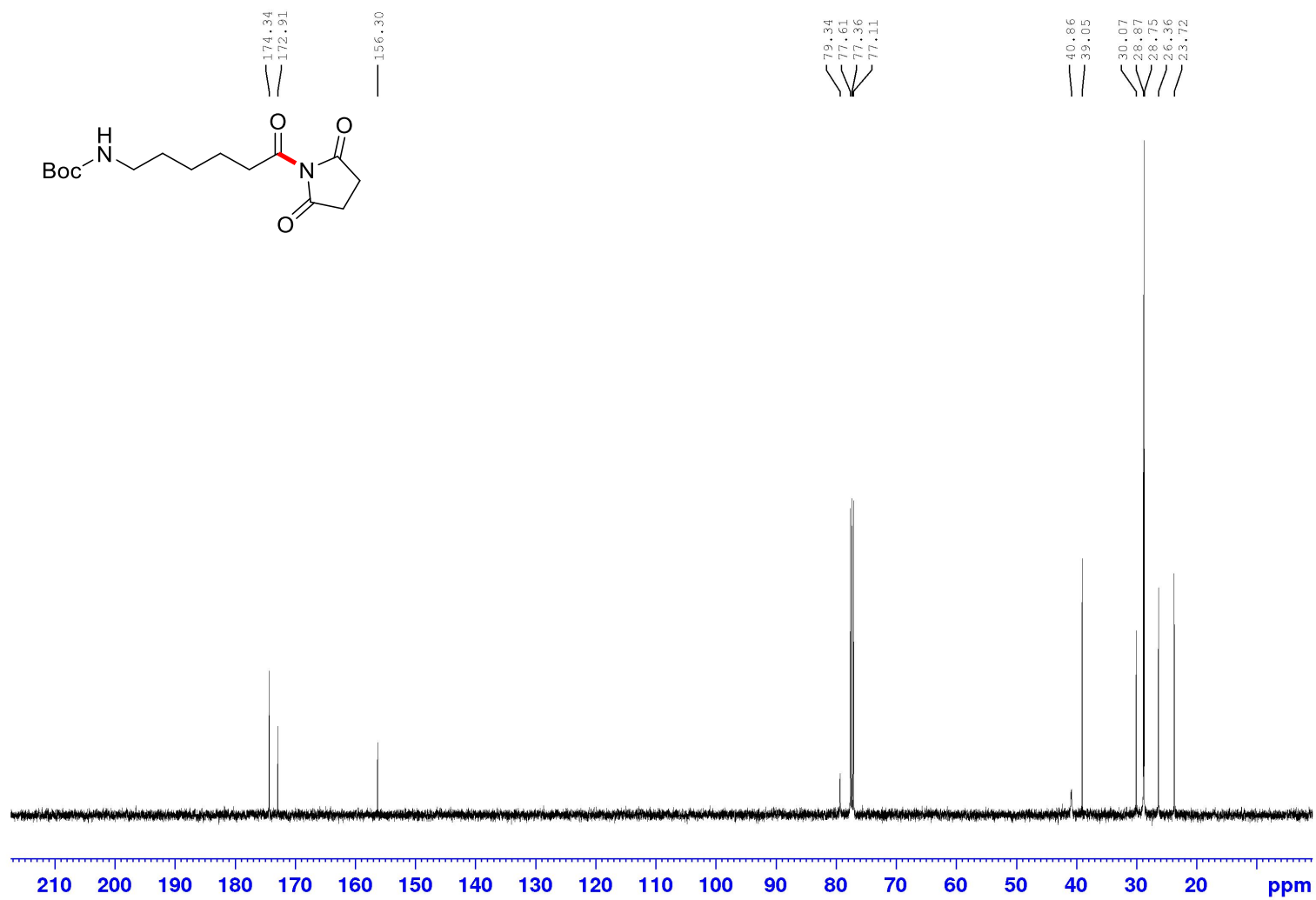
¹³C NMR



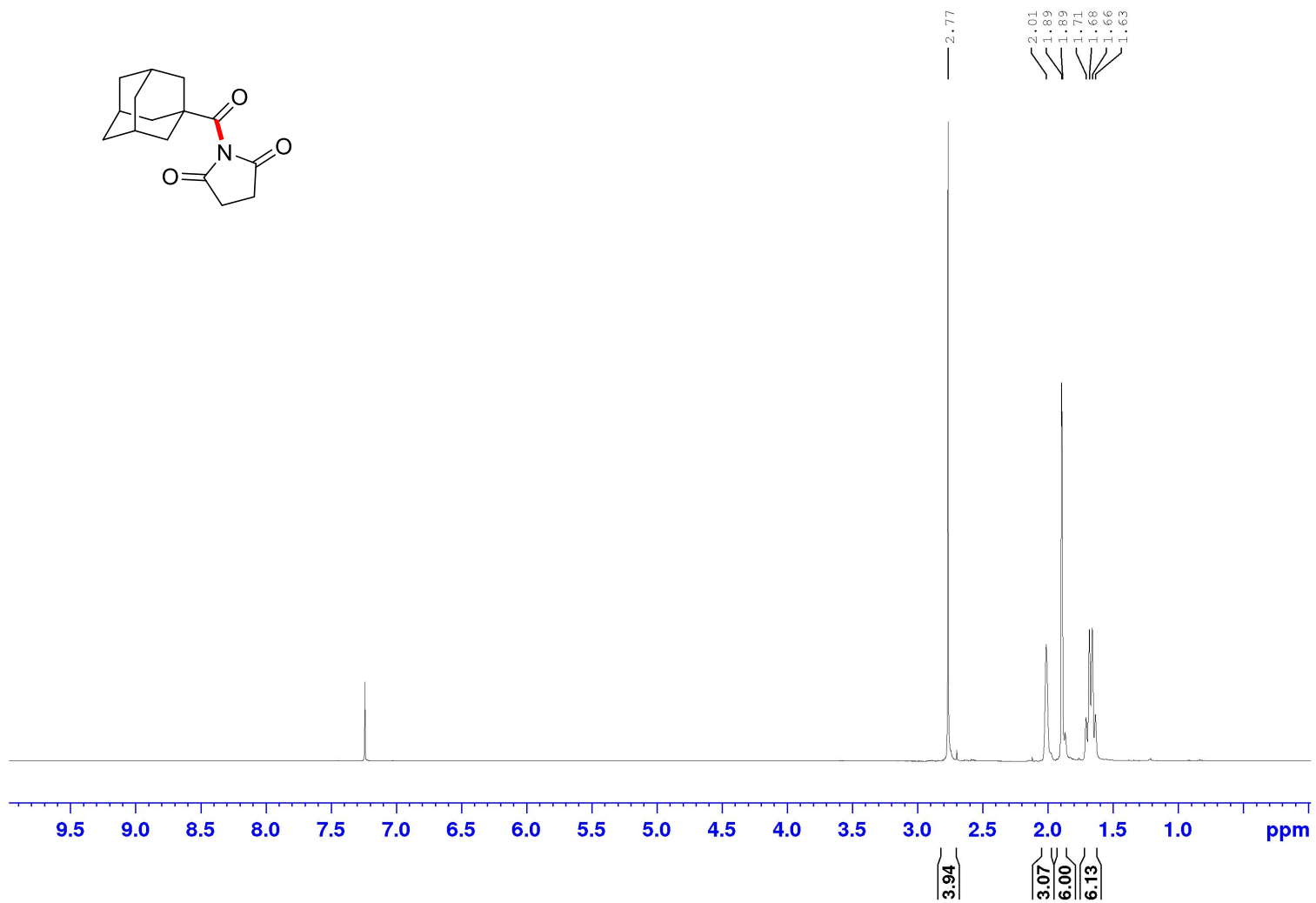
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of benzyl 3-(2,5-dioxopyrrolidine-1-carbonyl)piperidine-1-carboxylate



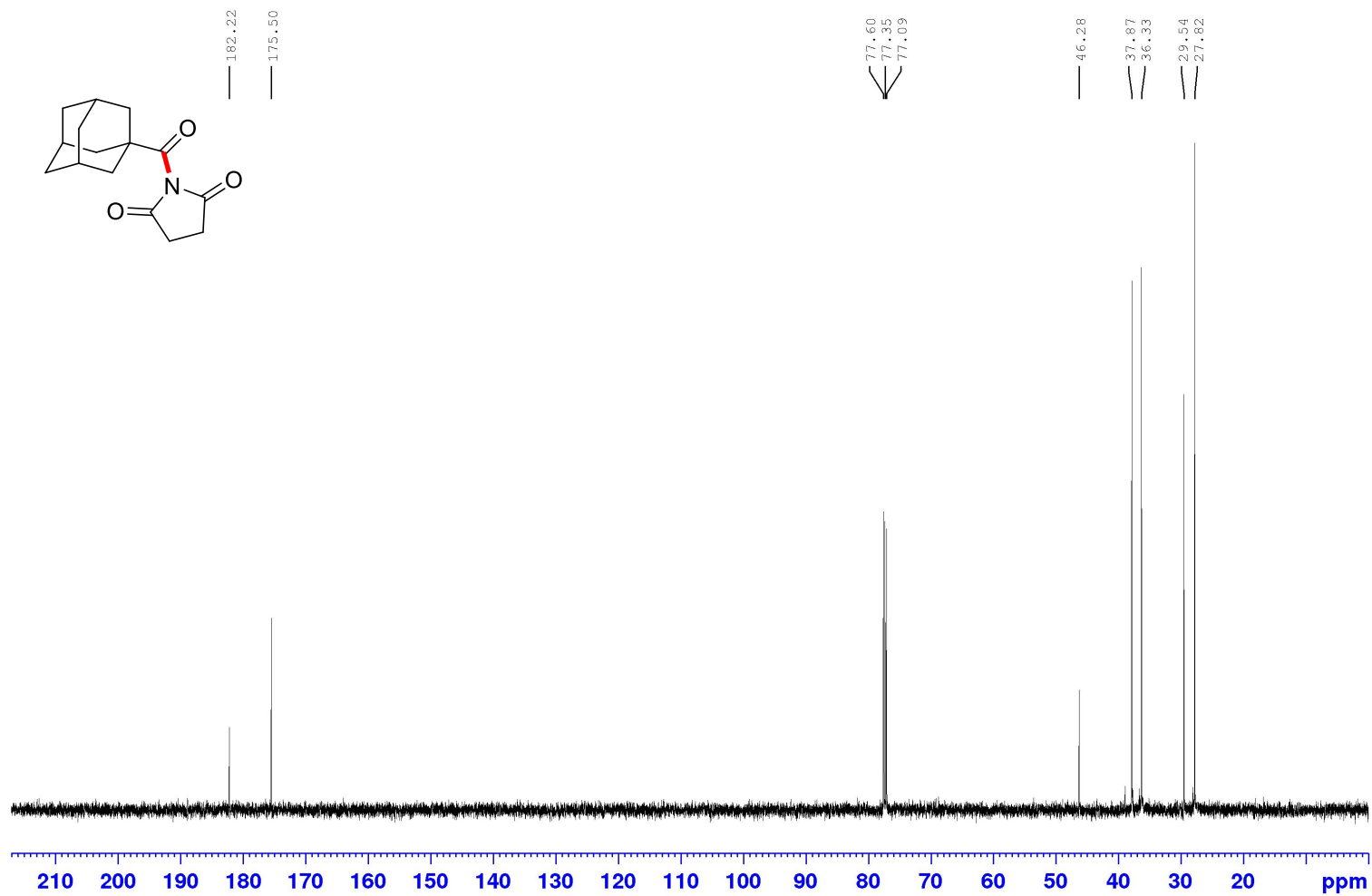
¹H NMR (500 MHz, CDCl₃) Spectrum of *tert*-butyl (6-(2,5-dioxopyrrolidin-1-yl)-6-oxohexyl)carbamate



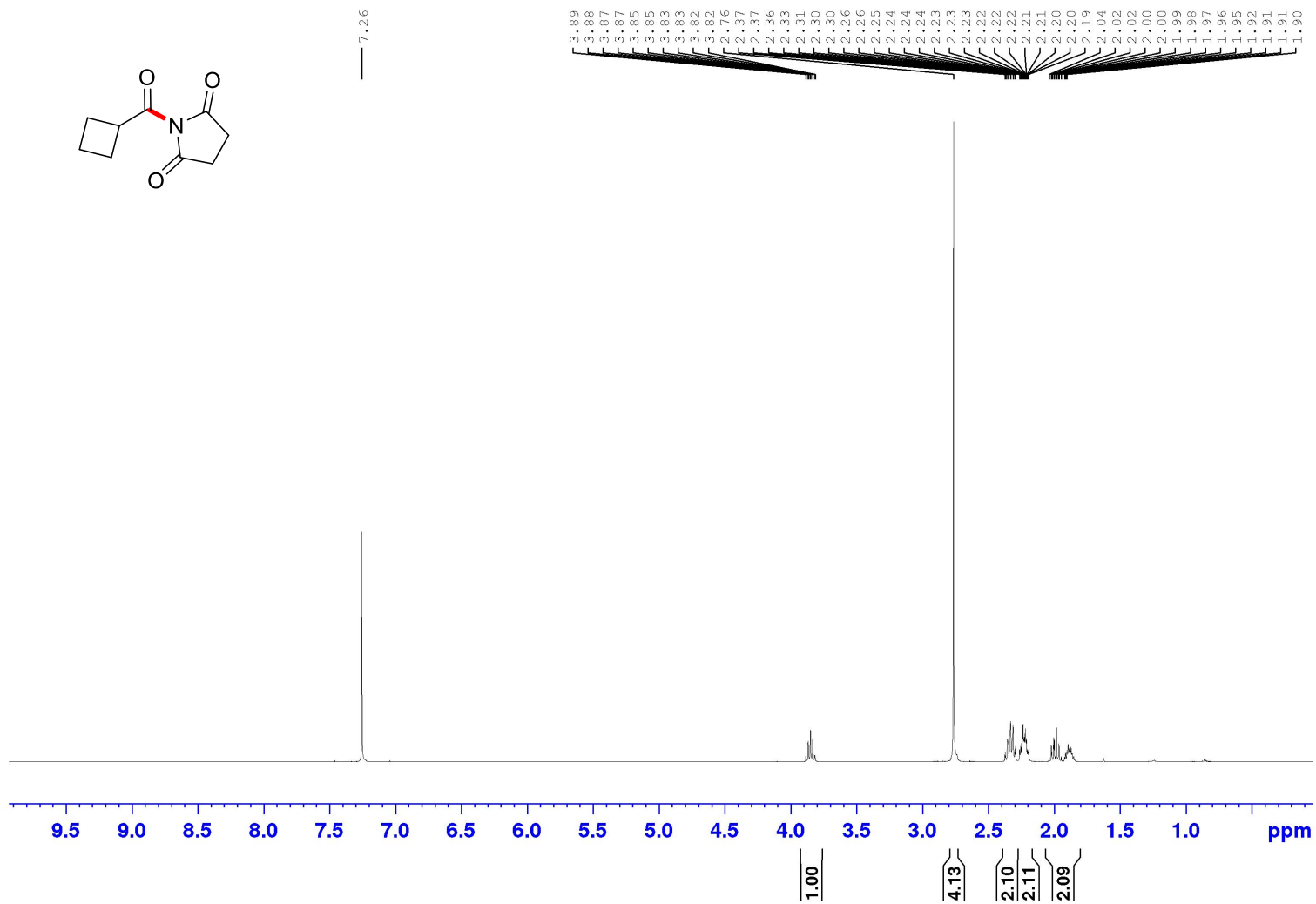
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of *tert*-butyl (6-(2,5-dioxopyrrolidin-1-yl)-6-oxohexyl)carbamate



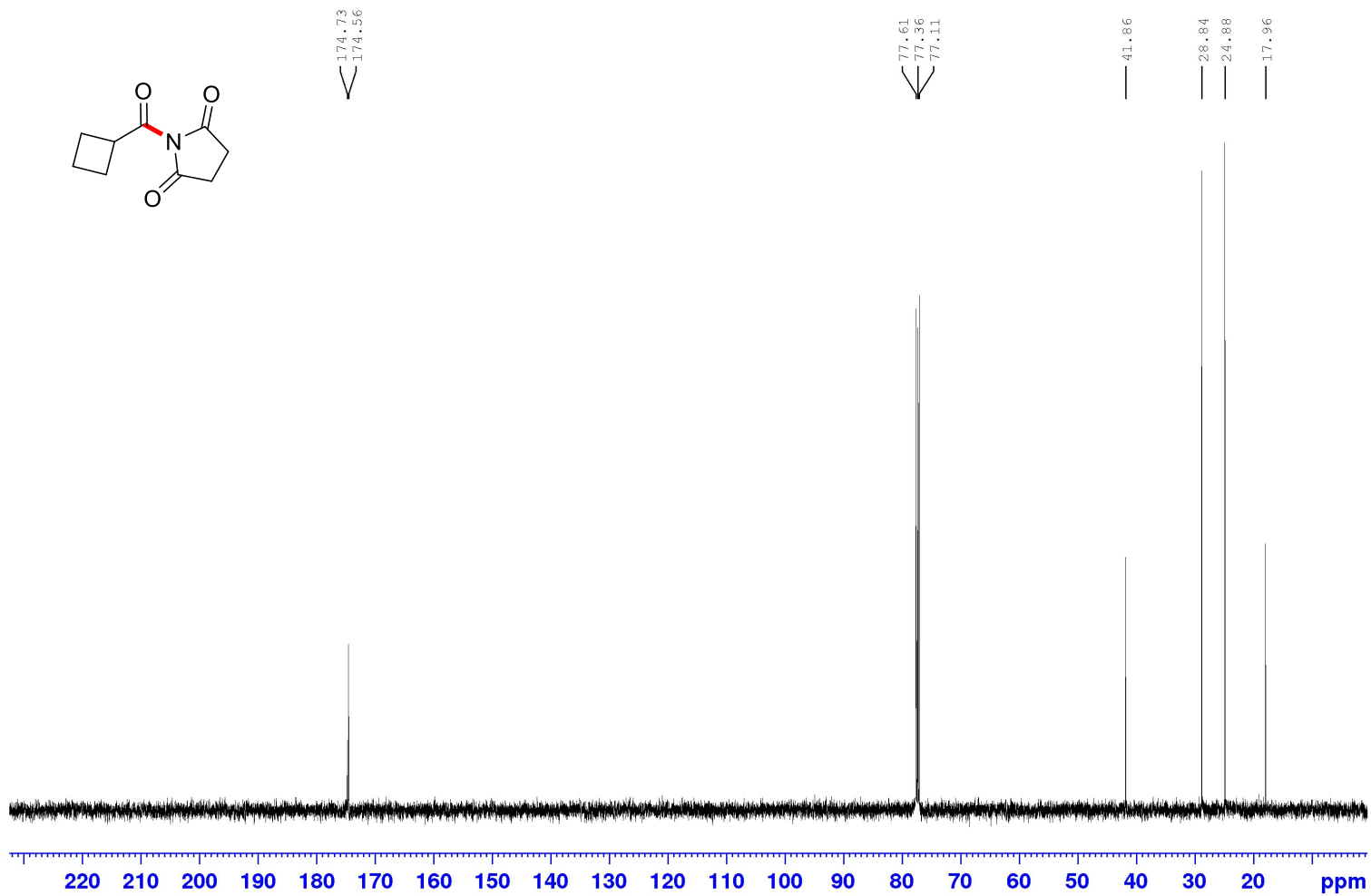
^1H NMR (500 MHz, CDCl_3) Spectrum of 1-(adamantane-1-carbonyl)pyrrolidine-2,5-dione



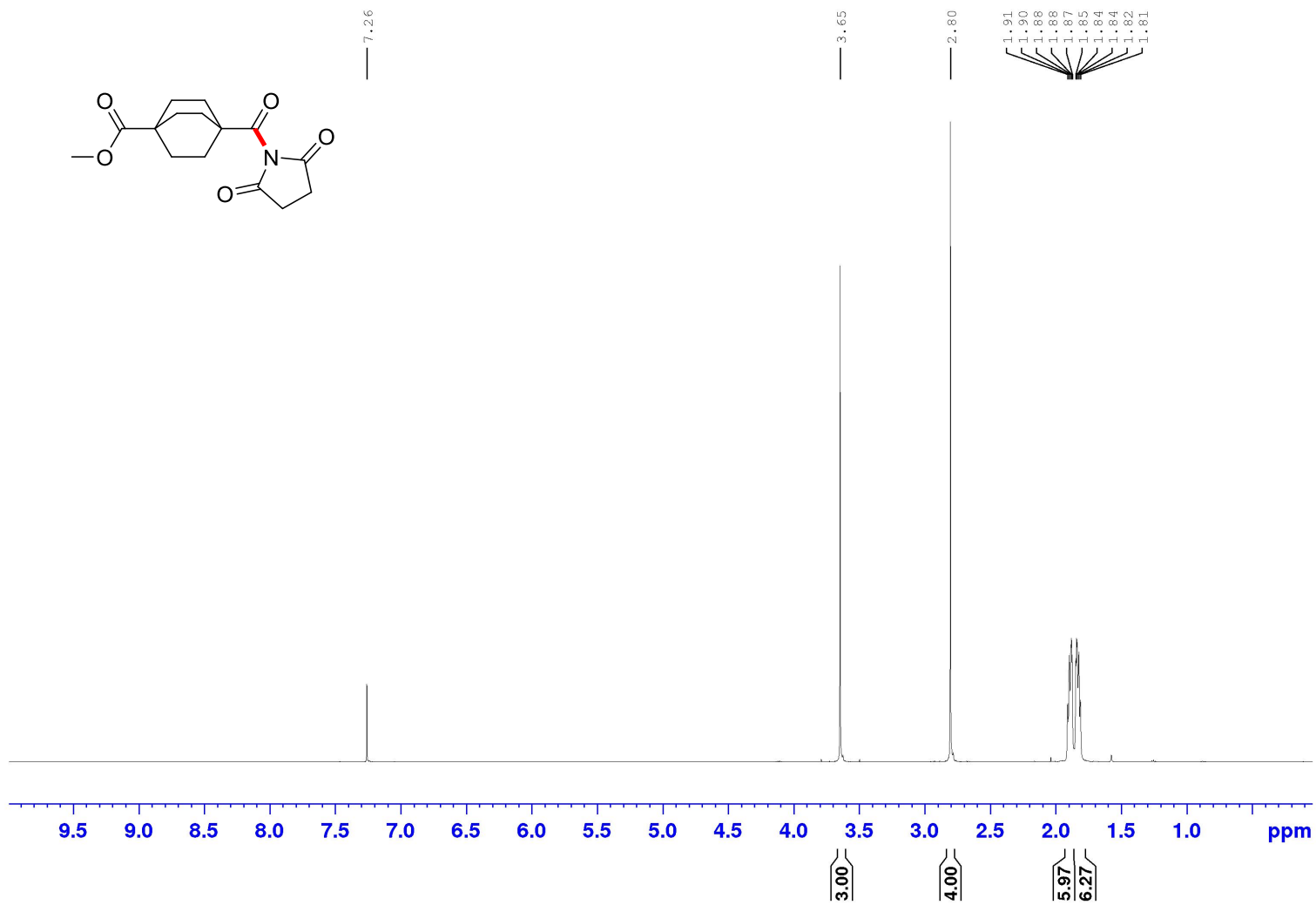
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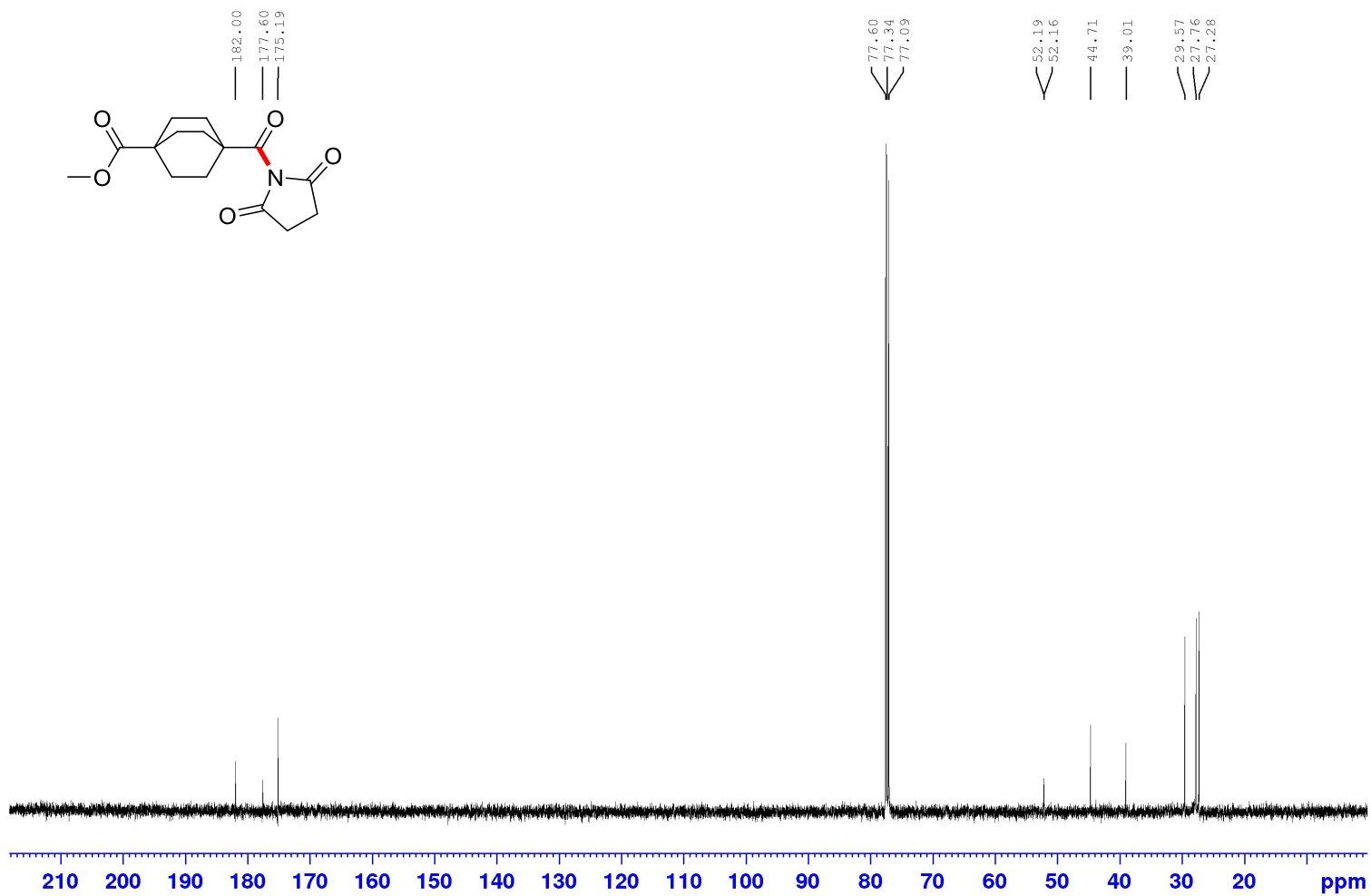
^1H NMR (500 MHz, CDCl_3) Spectrum of 1-(cyclobutanecarbonyl)pyrrolidine-2,5-dione



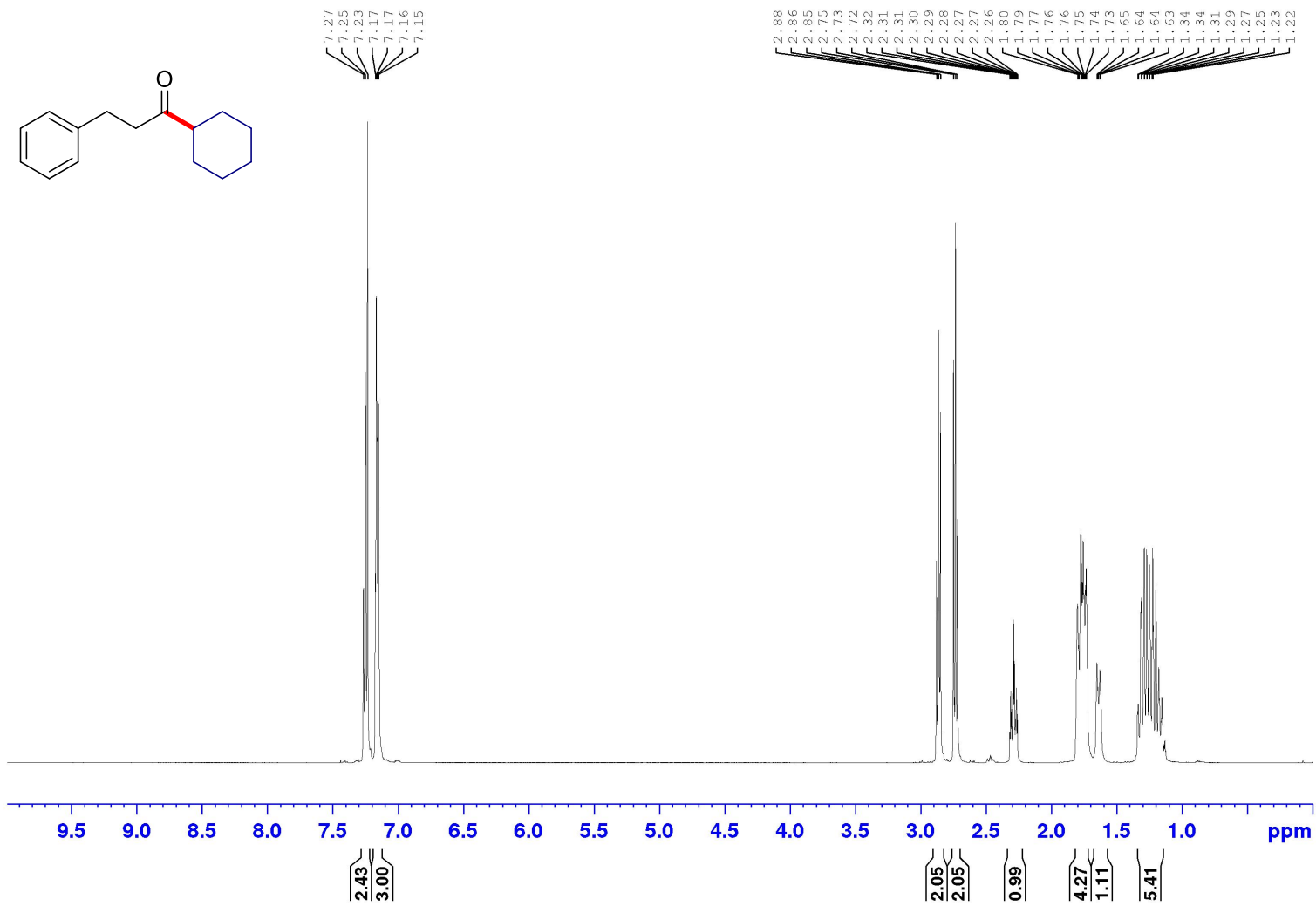
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of 1-(cyclobutanecarbonyl)pyrrolidine-2,5-dione



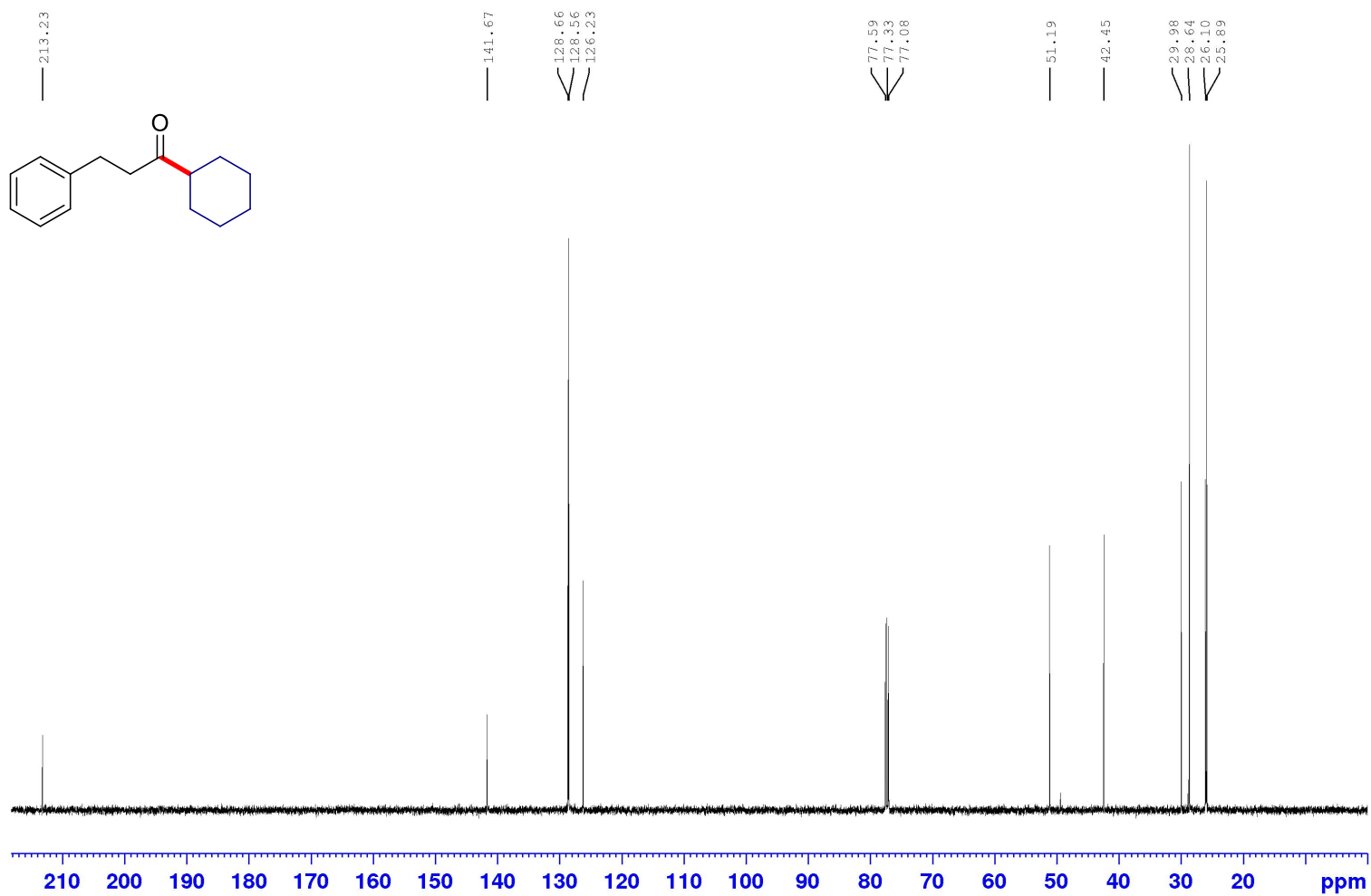
¹H NMR (500 MHz, CDCl₃) Spectrum of methyl 4-(2,5-dioxopyrrolidine-1-carbonyl)bicyclo[2.2.2]octane-1-carboxylate



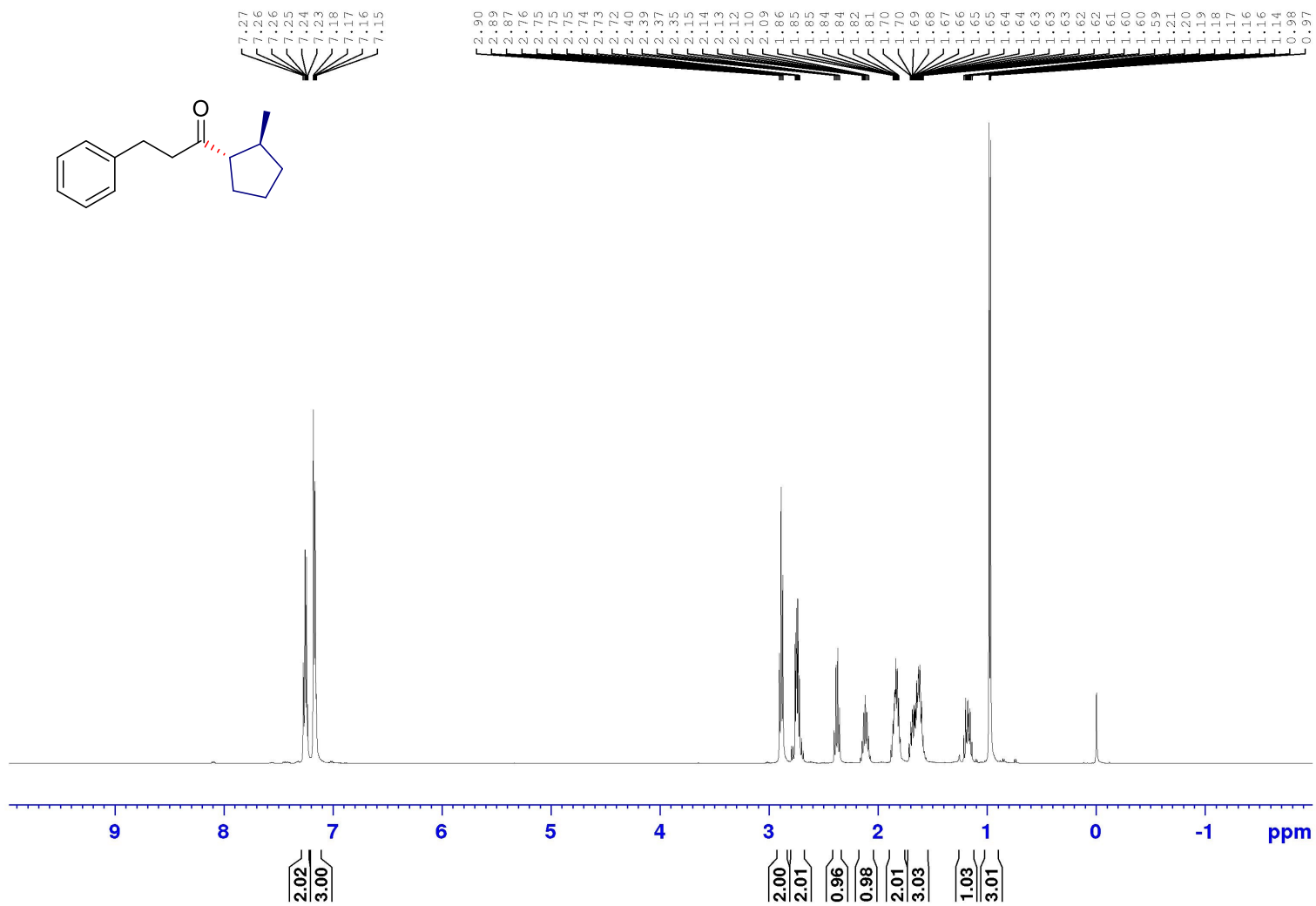
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of methyl 4-(2,5-dioxopyrrolidine-1-carbonyl)bicyclo[2.2.2]octane-1-carboxylate



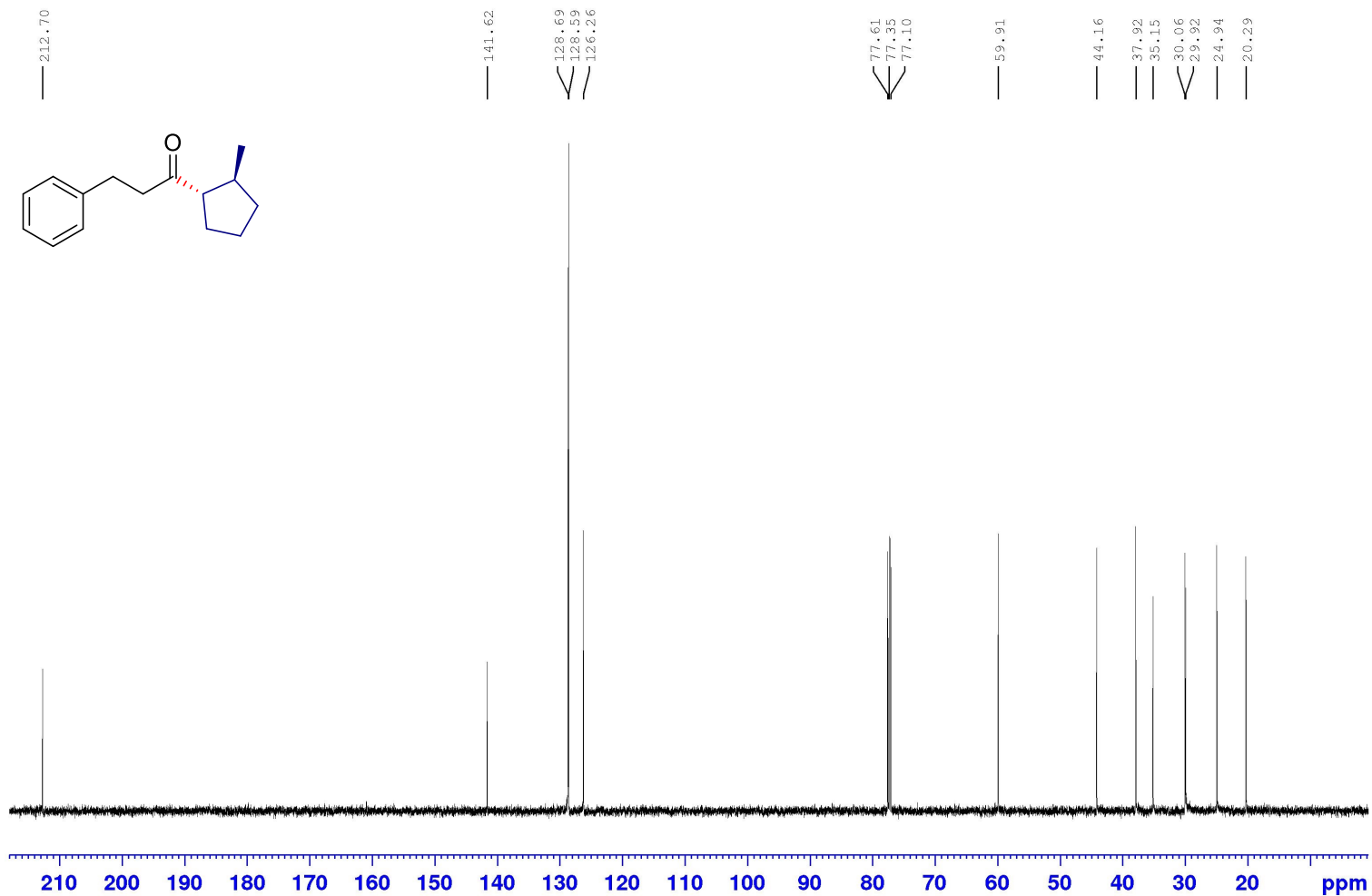
¹H NMR (500 MHz, CDCl₃) Spectrum of 1-cyclohexyl-3-phenylpropan-1-one (3a)



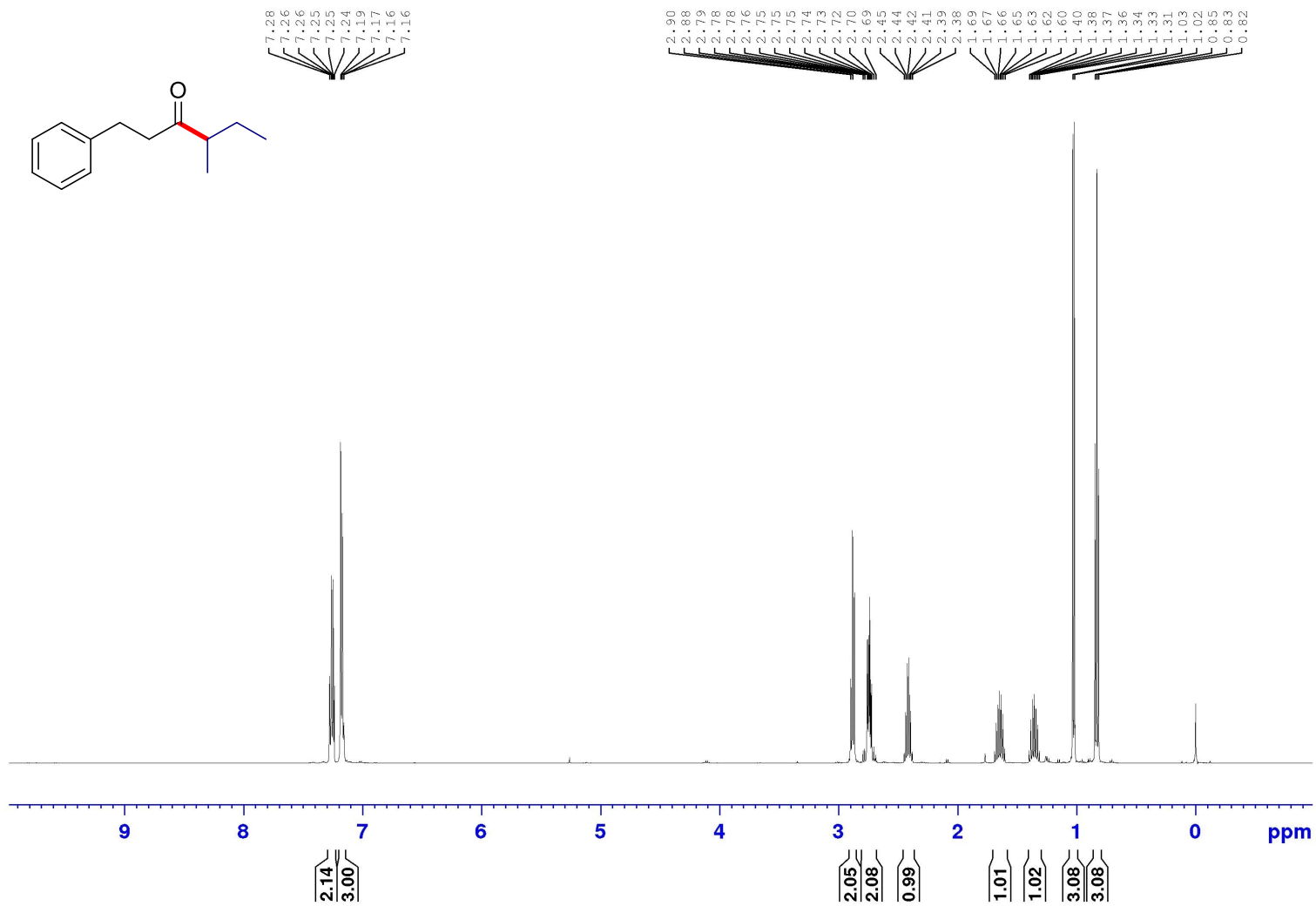
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of 1-cyclohexyl-3-phenylpropan-1-one (3a)



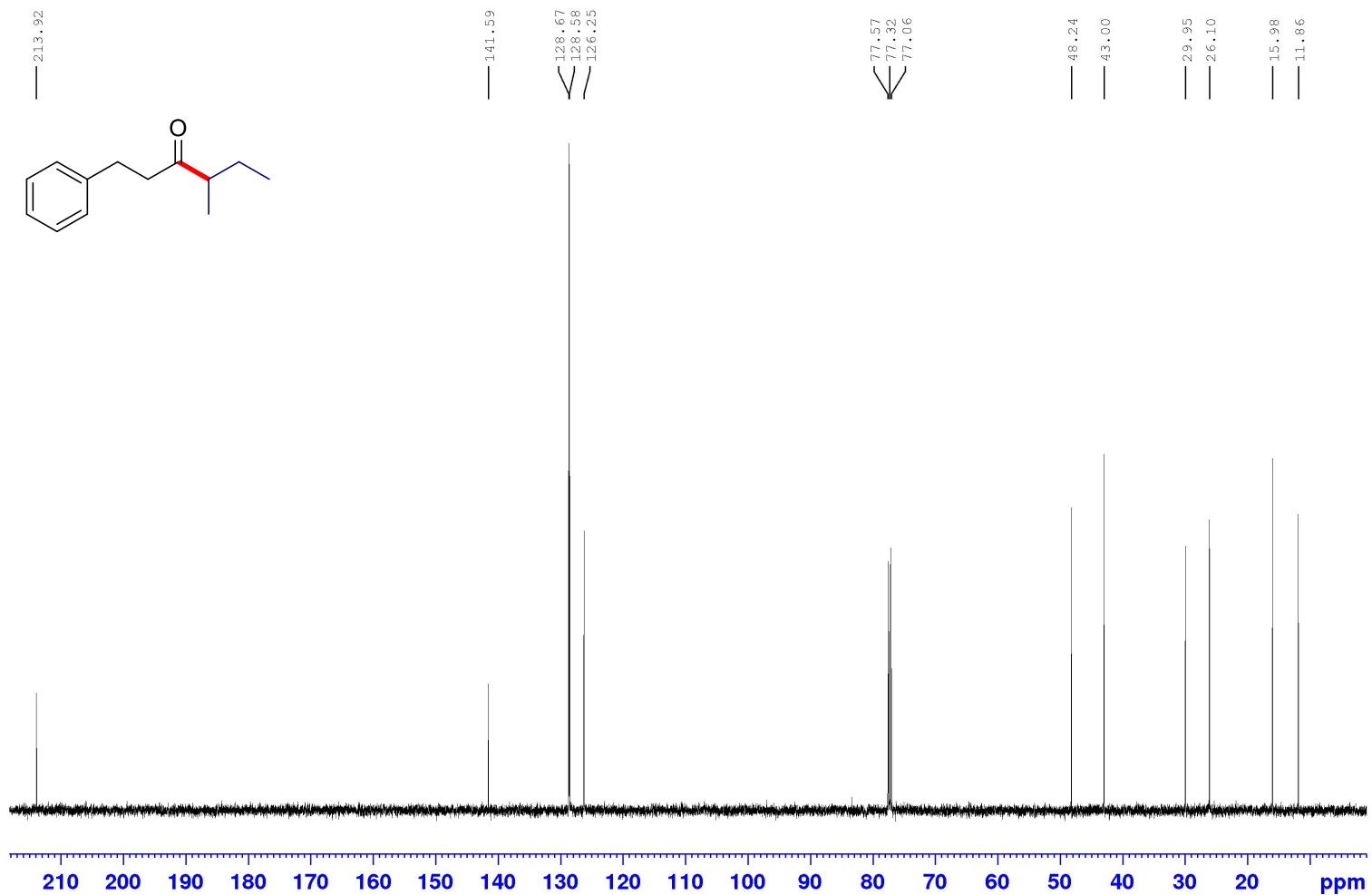
¹H NMR (500 MHz, CDCl₃) Spectrum of 1-(2-methylcyclopentyl)-3-phenylpropan-1-one (3b)



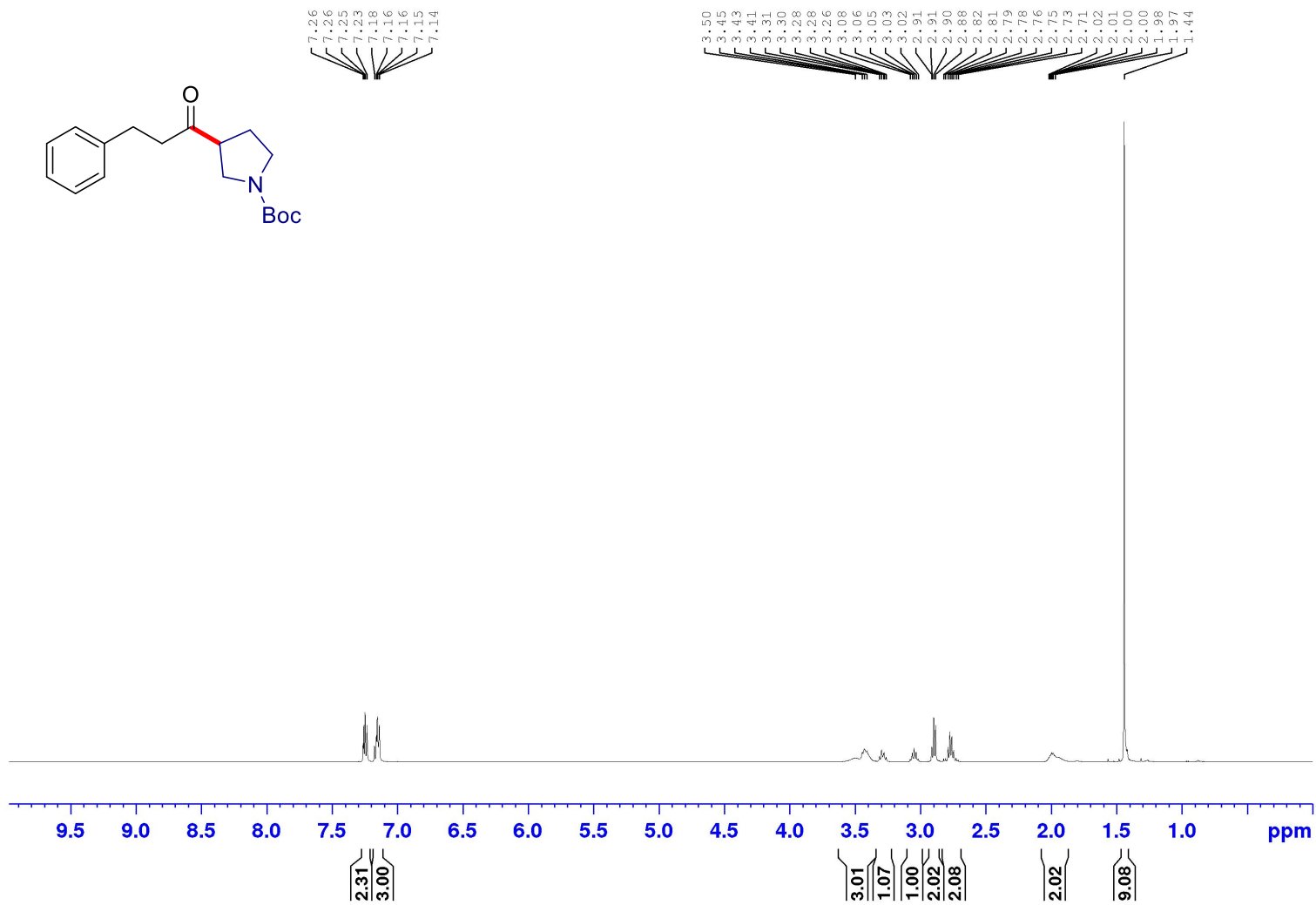
^{13}C NMR (125.8 MHz, CDCl_3) Spectrum of 1-(2-methylcyclopentyl)-3-phenylpropan-1-one (3b)



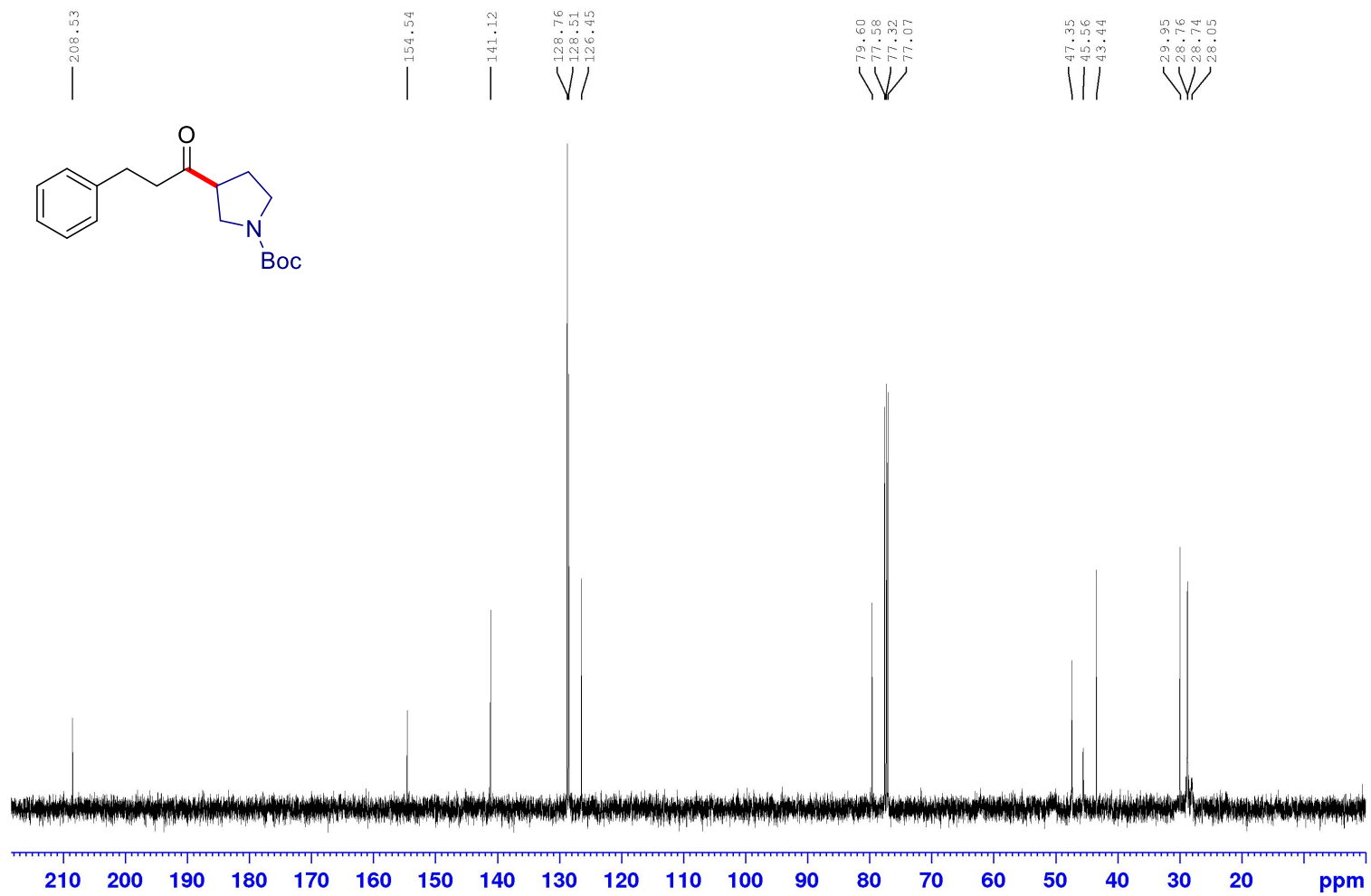
¹H NMR (500 MHz, CDCl₃) Spectrum of 4-methyl-1-phenylhexan-3-one (3c)



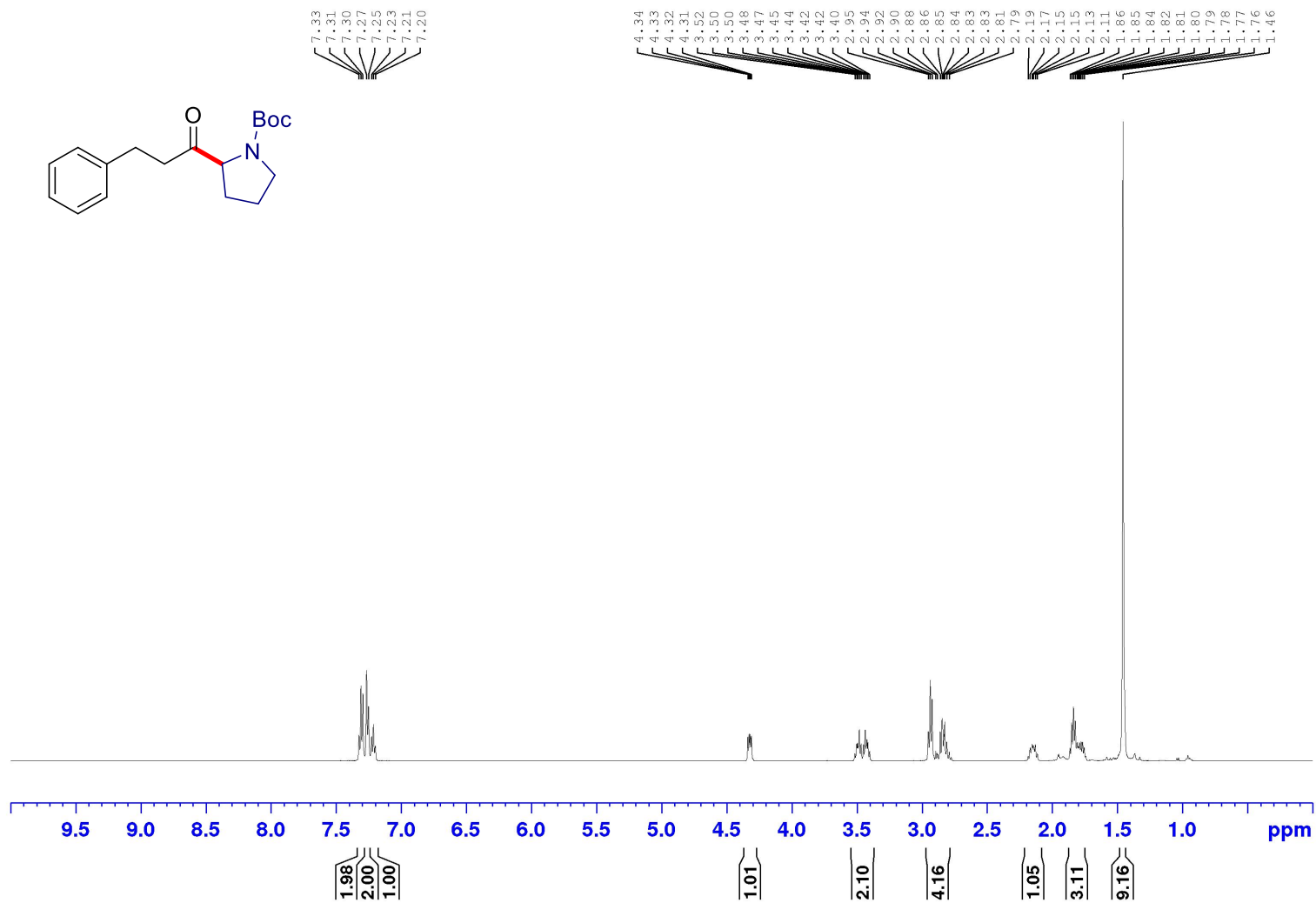
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of 4-methyl-1-phenylhexan-3-one (3c)



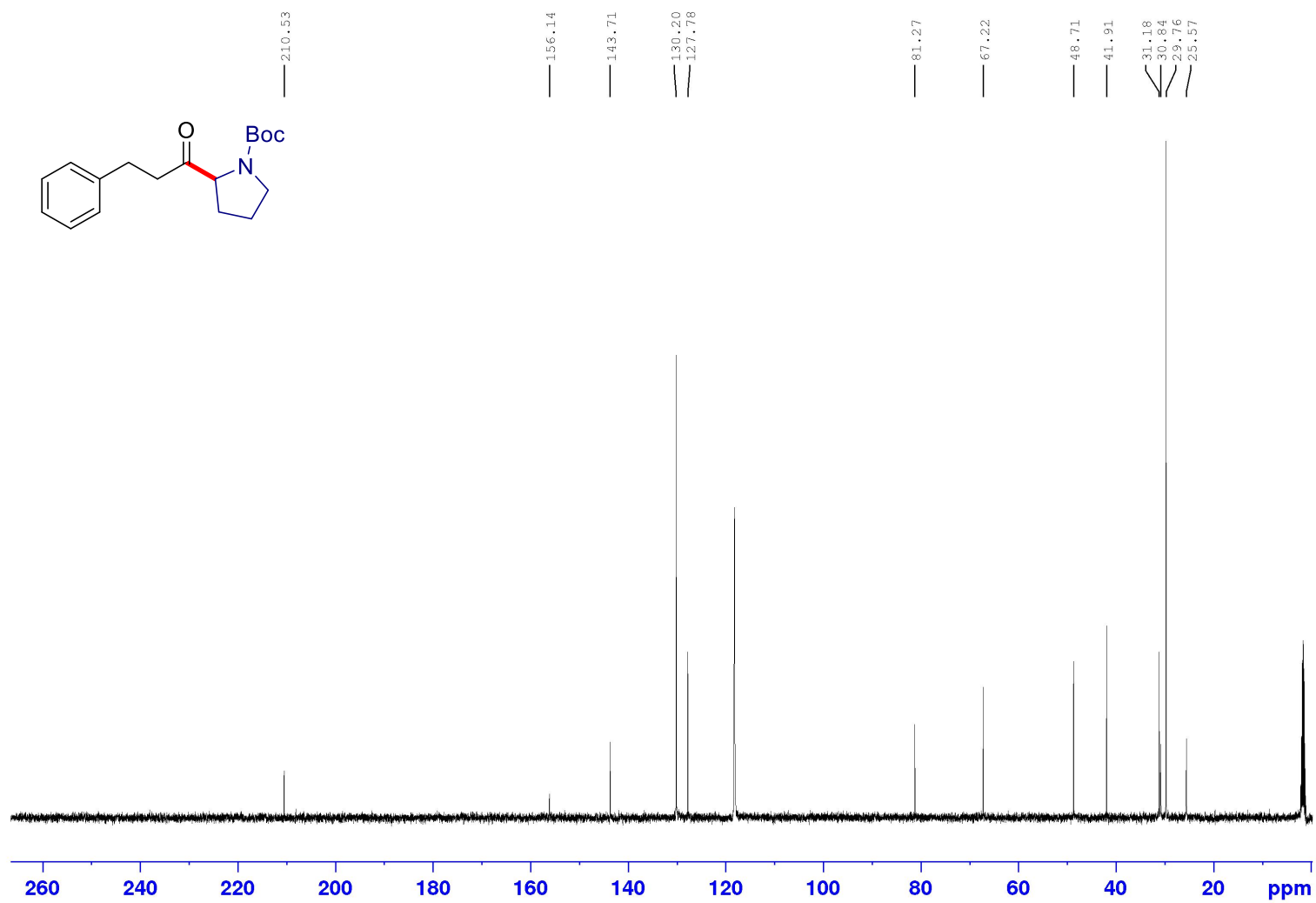
¹H NMR (500 MHz, CDCl₃) Spectrum of *tert*-butyl 3-(3-Phenylpropanoyl)pyrrolidine-1-carboxylate (3d)



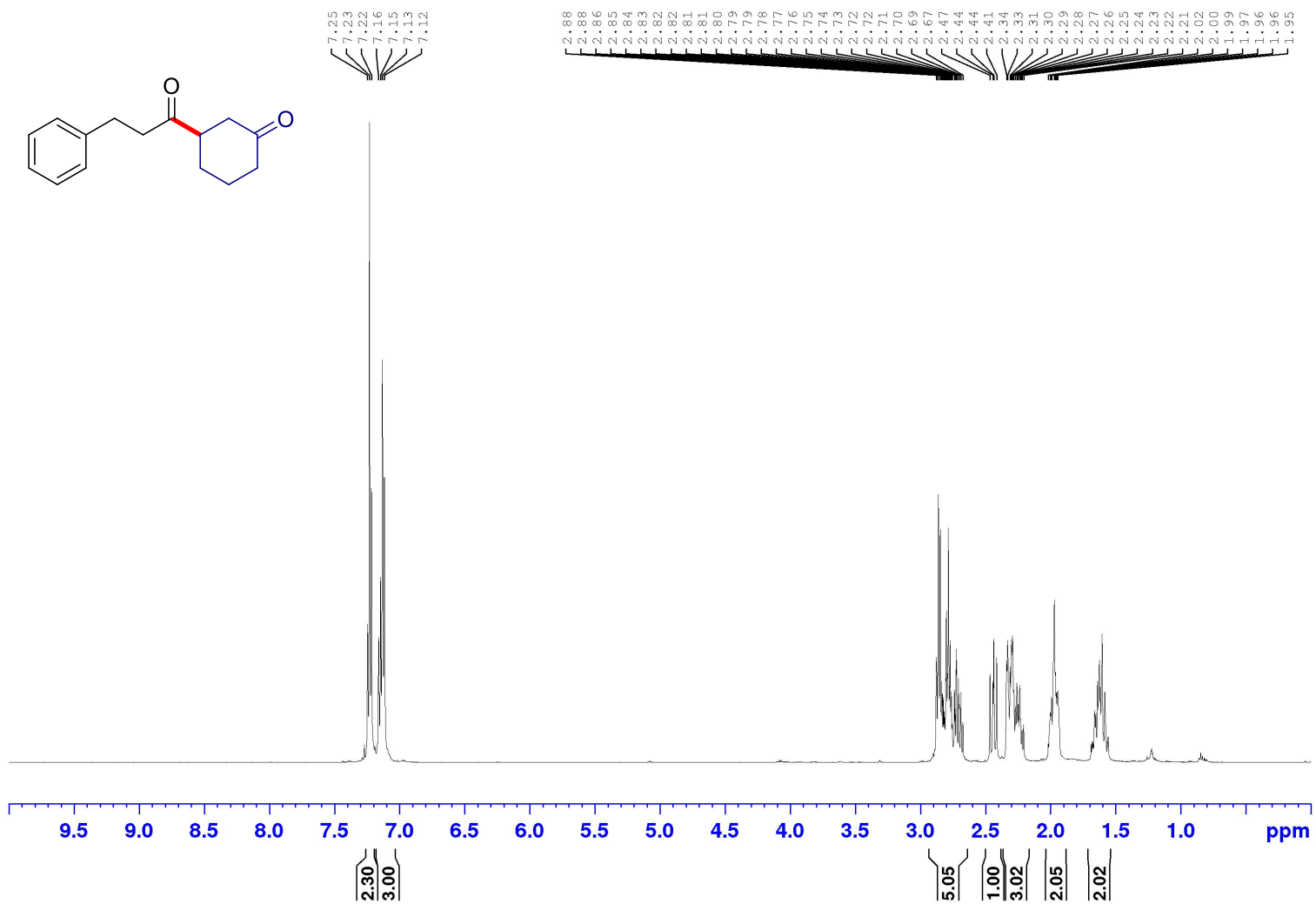
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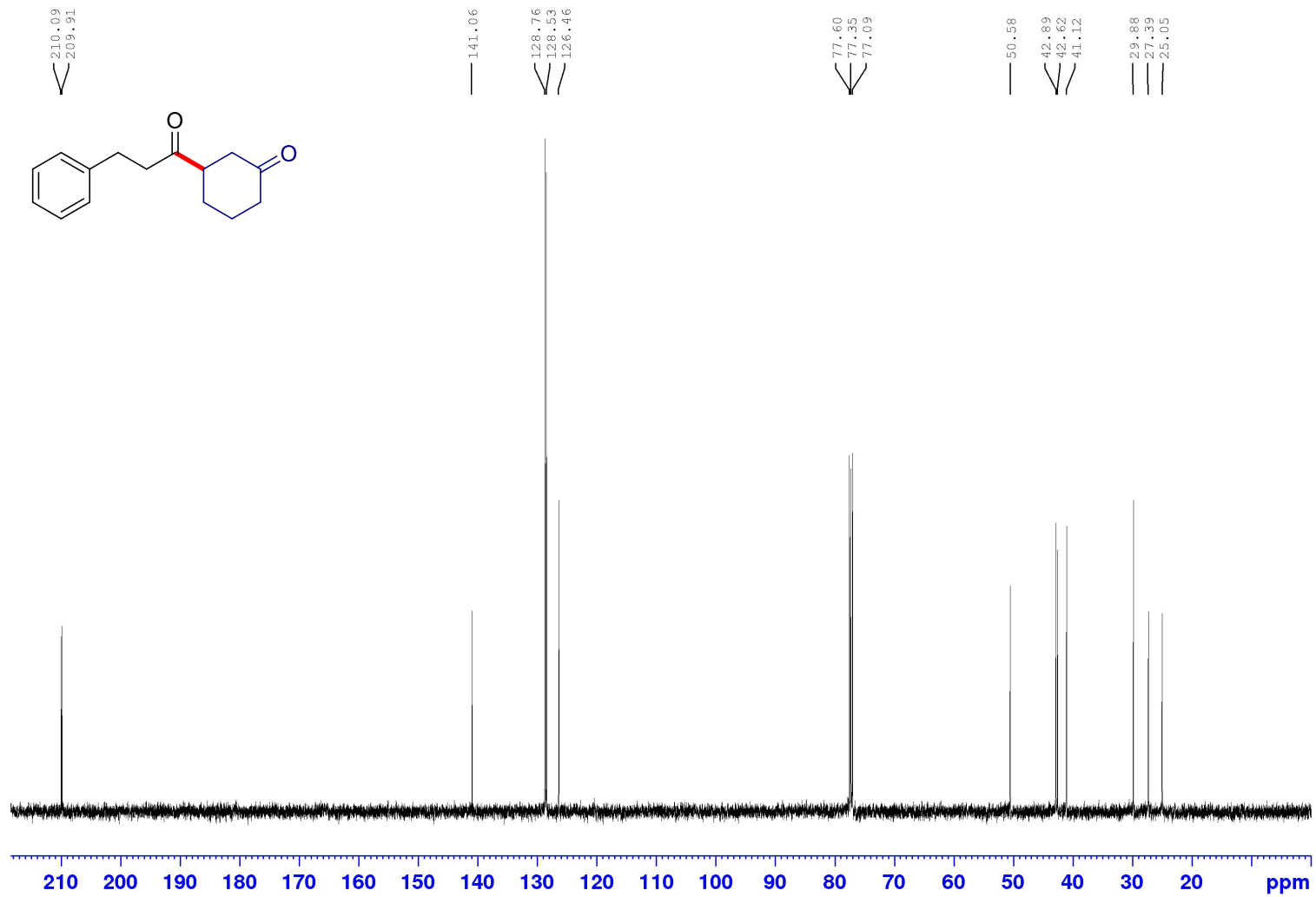
¹H NMR (500 MHz, CDCl₃) Spectrum of *tert*-butyl-2-(3-phenylpropanoyl)pyrrolidine-1-carboxylate (3e)



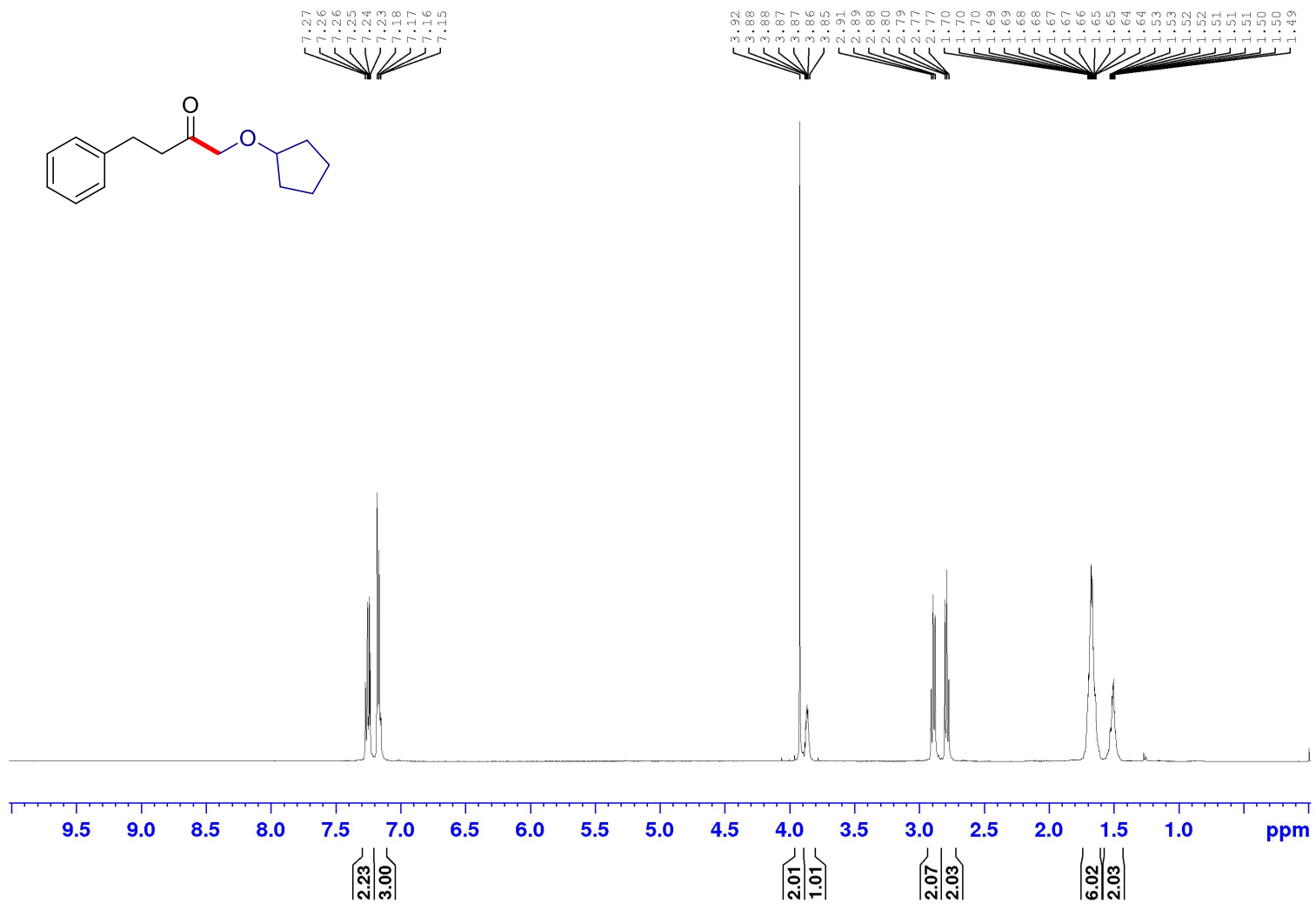
^{13}C NMR (125.8 MHz, CDCl_3) Spectrum of *tert*-butyl-2-(3-phenylpropanoyl)pyrrolidine-1-carboxylate (3e)



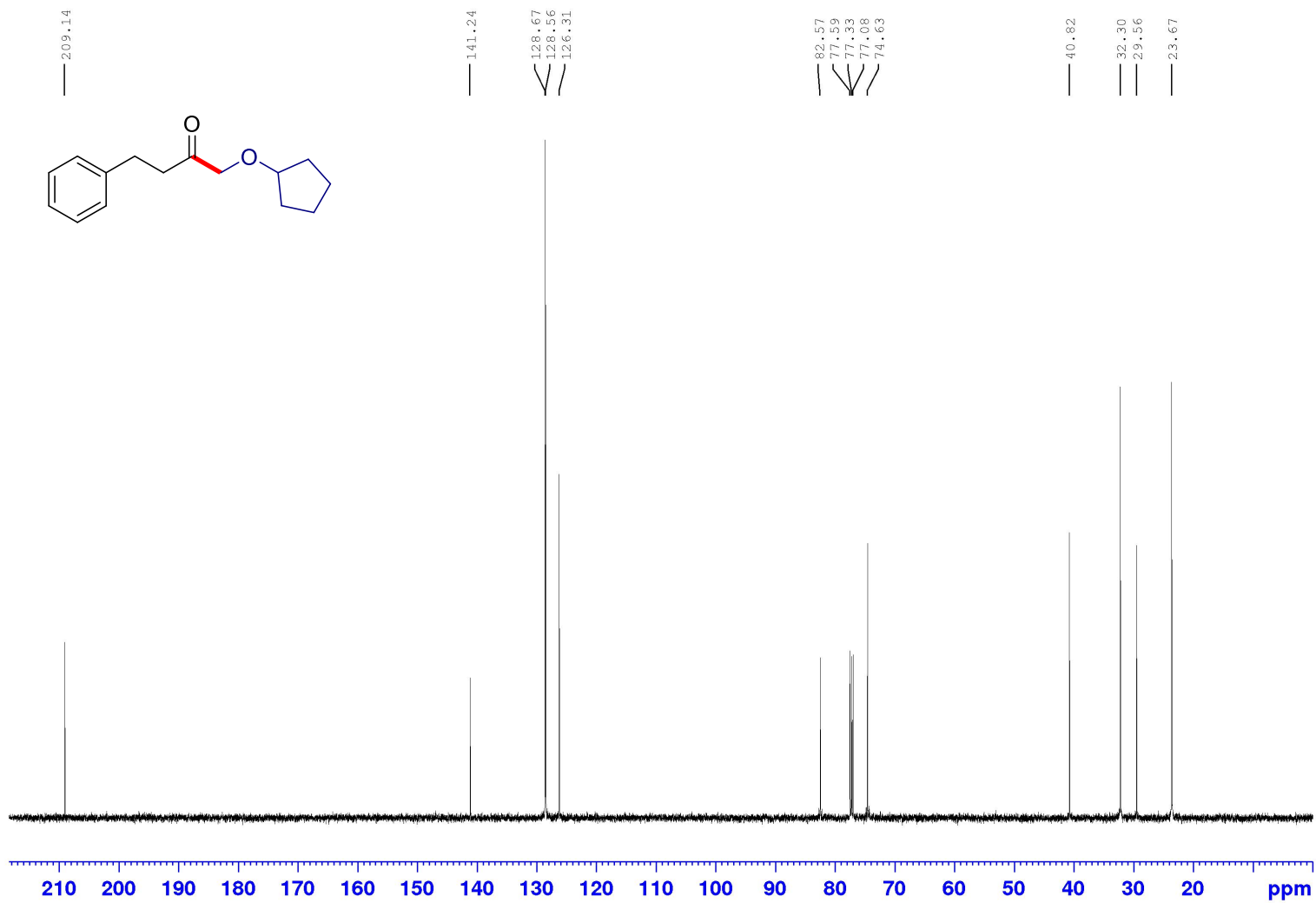
¹H NMR (500 MHz, CDCl₃) Spectrum of 3-(3-phenylpropanoyl)cyclohexan-1-one (3f)



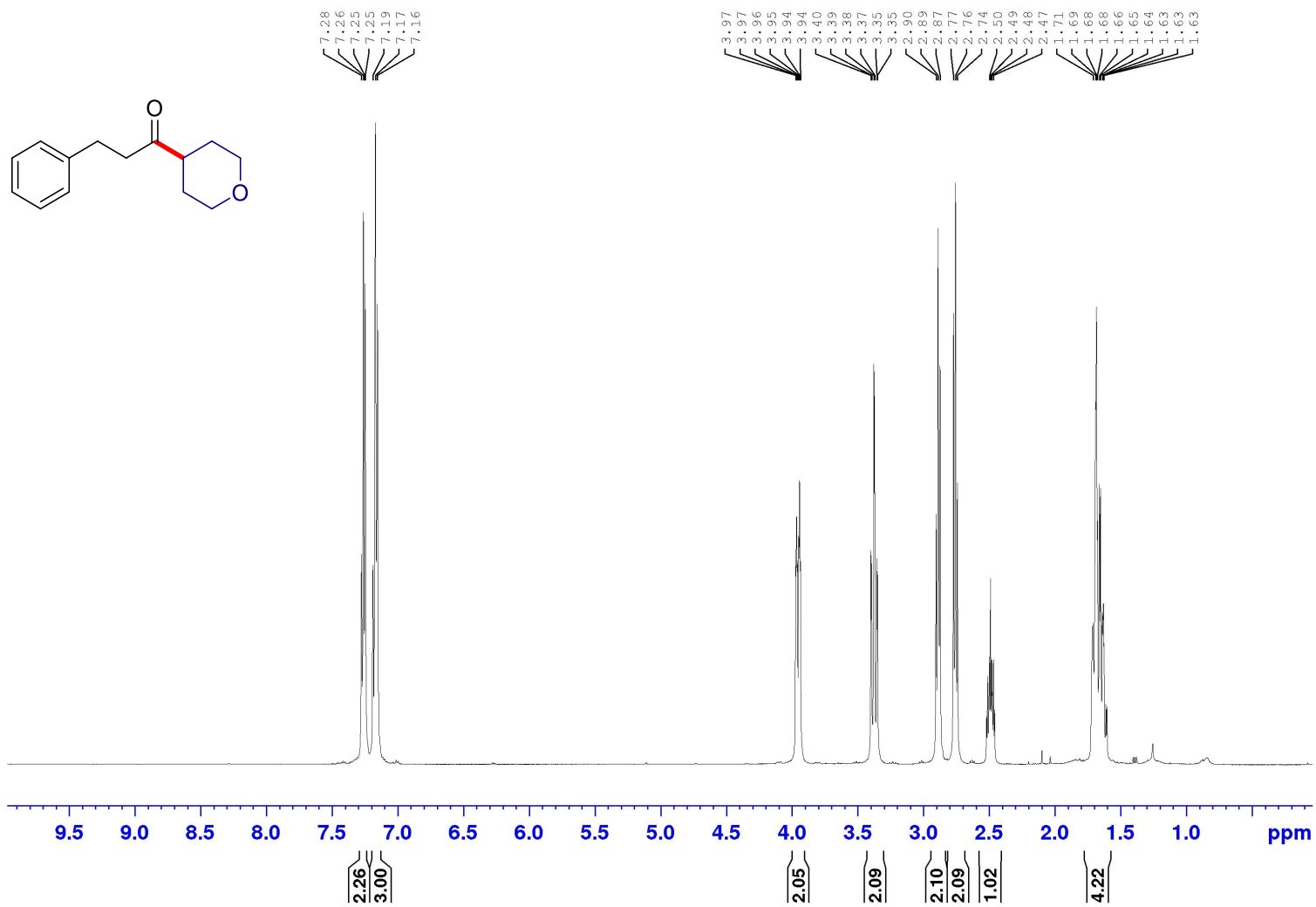
^{13}C NMR (125.8 MHz, CDCl_3) Spectrum of 3-(3-phenylpropanoyl)cyclohexan-1-one (3f)



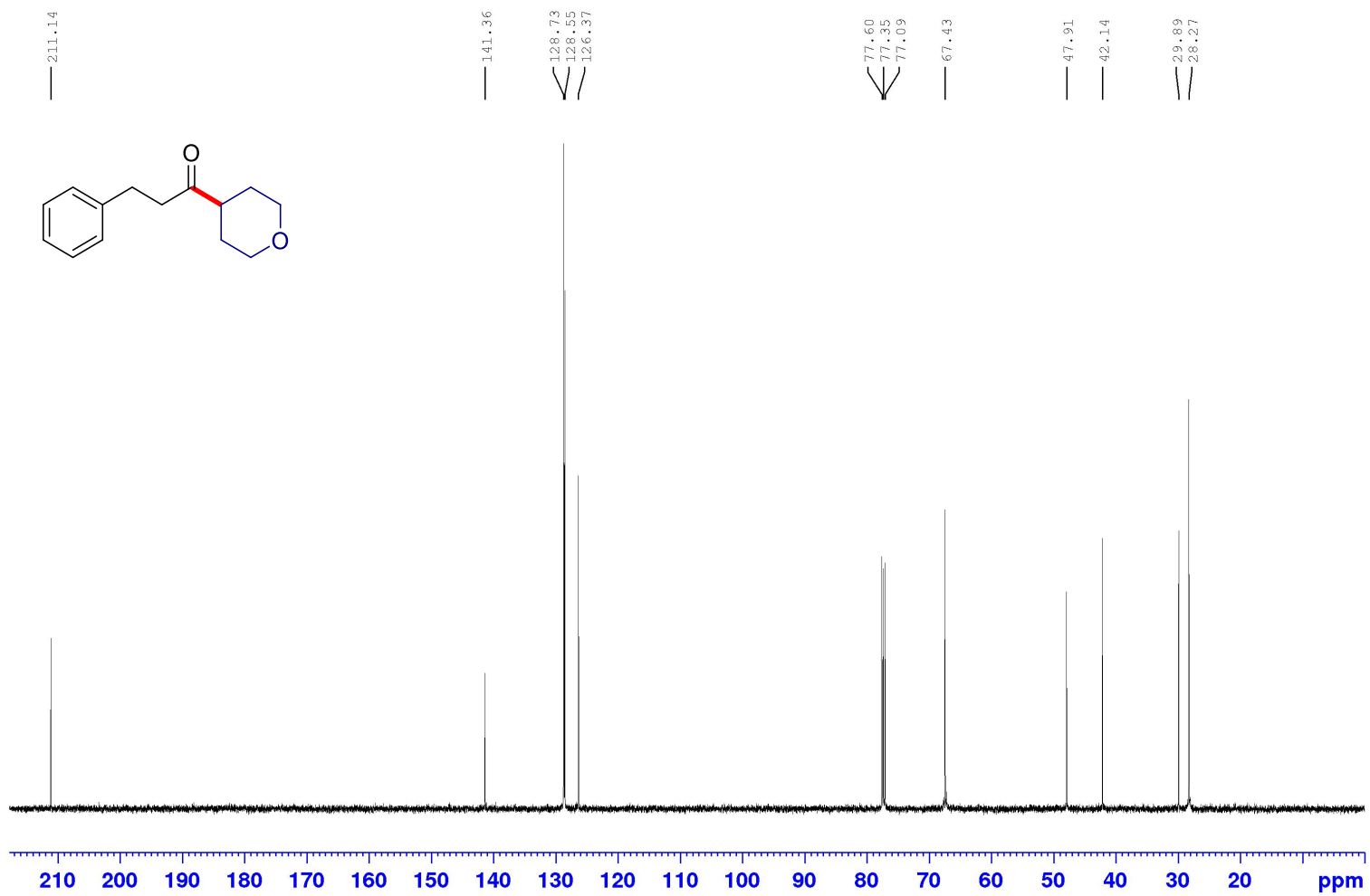
¹H NMR (500 MHz, CDCl₃) Spectrum of 1-(cyclopentyloxy)-4-phenylbutan-2-one (3g)



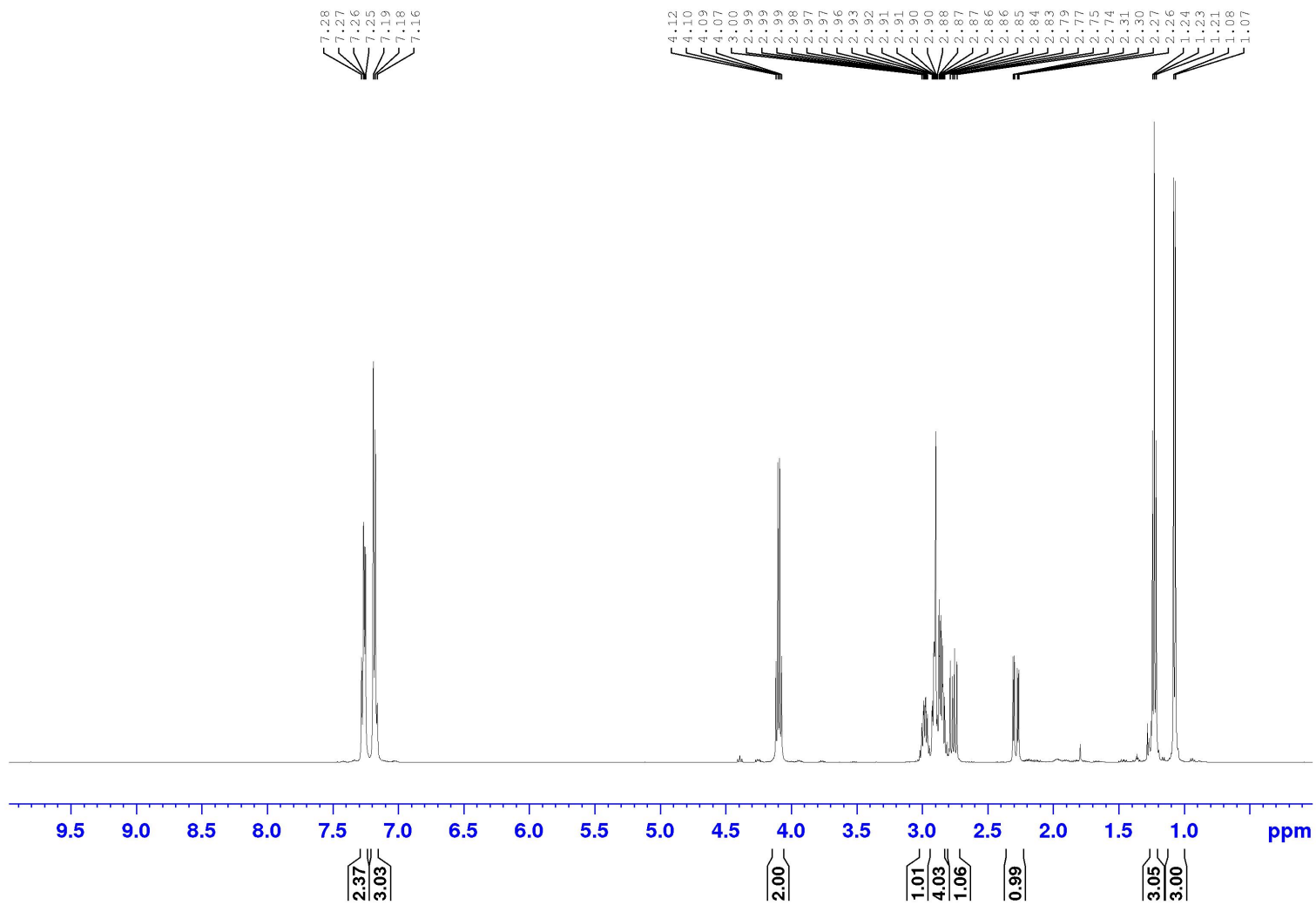
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of 1-(cyclopentyloxy)-4-phenylbutan-2-one (3g)



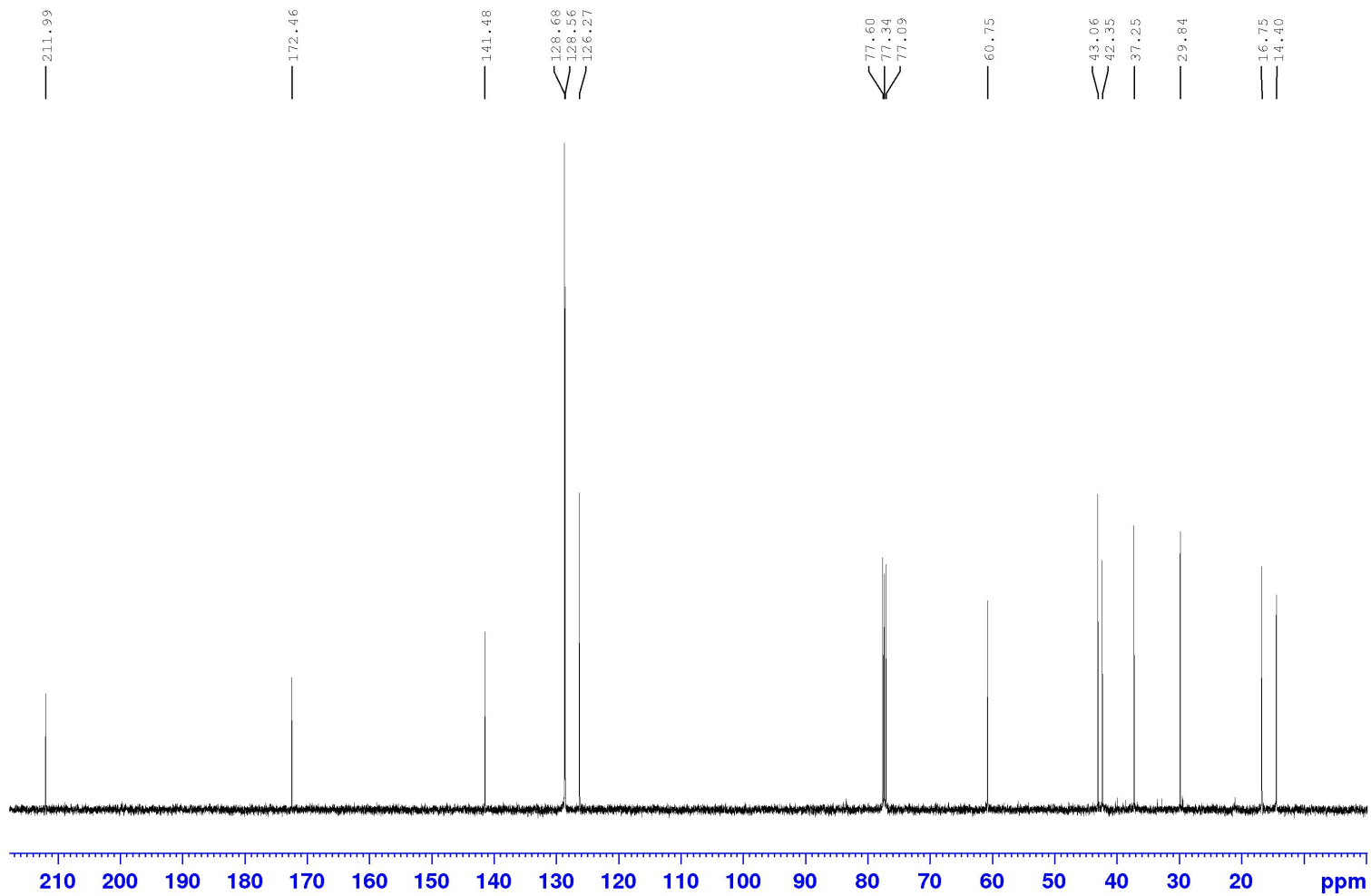
¹H NMR (500 MHz, CDCl₃) Spectrum of 3-phenyl-1-(tetrahydro-2H-pyran-4-yl)propan-1-one (3h)



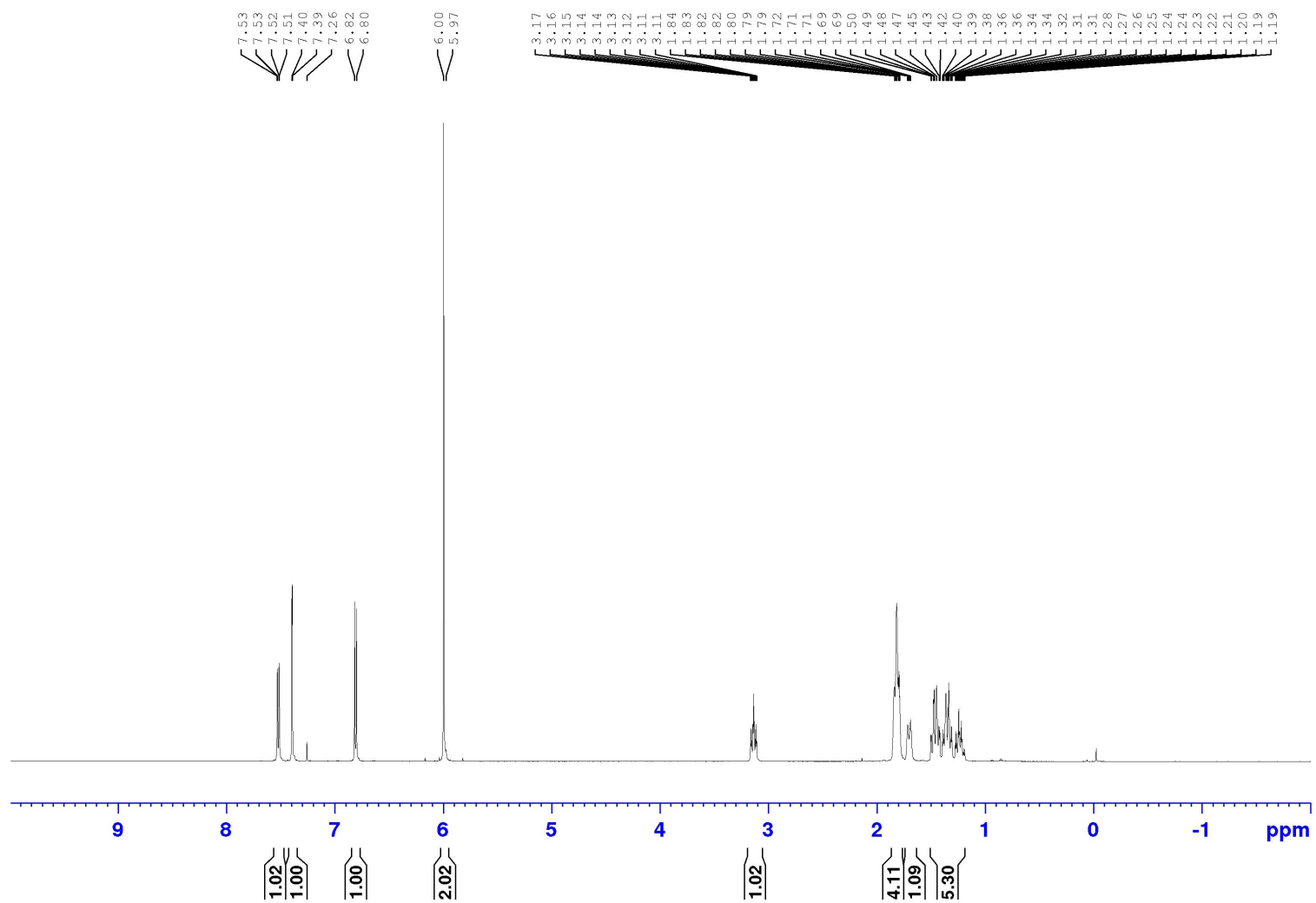
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of 3-phenyl-1-(tetrahydro-2H-pyran-4-yl)propan-1-one (3h)



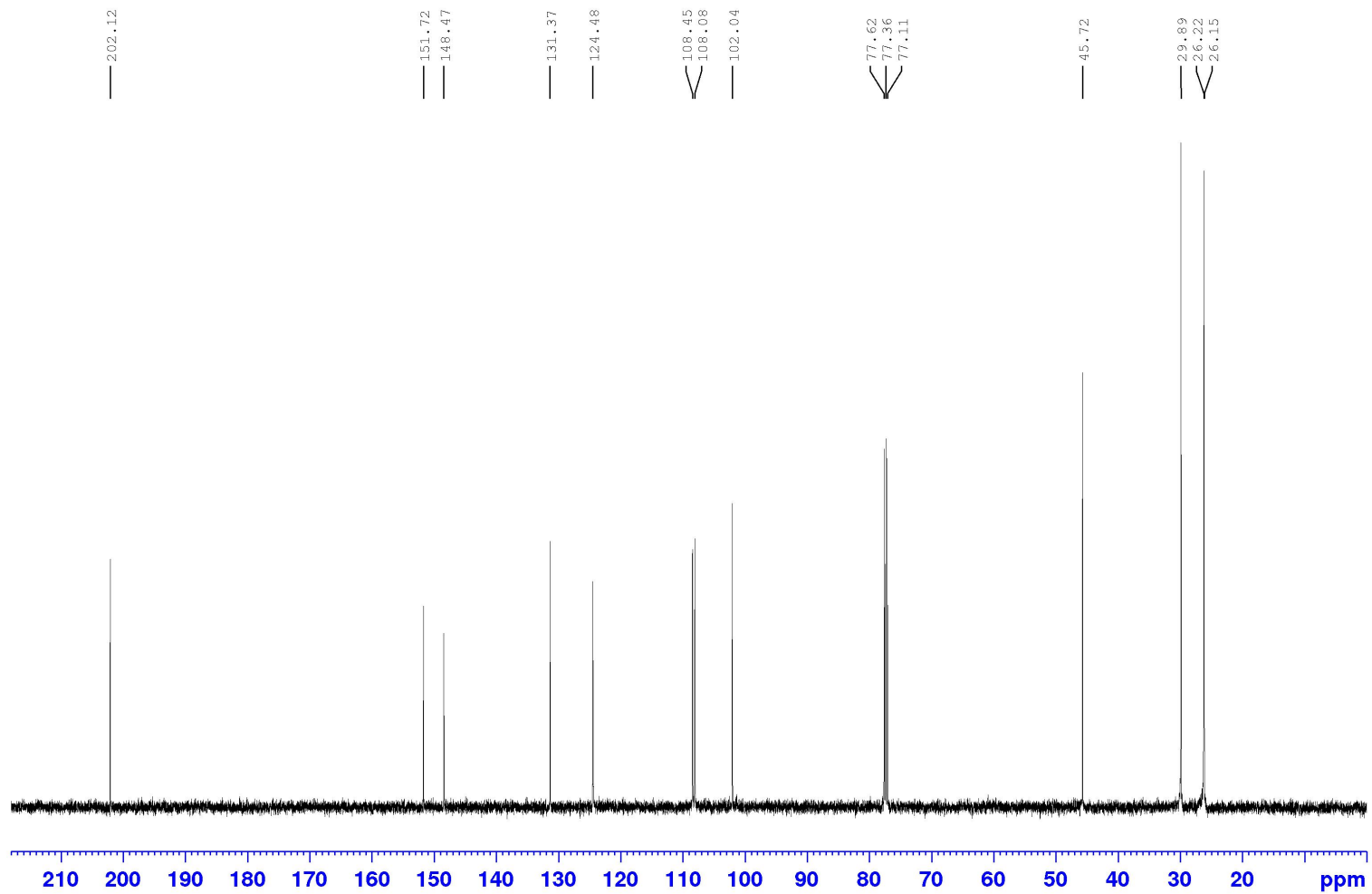
¹H NMR (500 MHz, CDCl₃) Spectrum of ethyl 3-methyl-4-oxo-6-phenylhexanoate (3i)



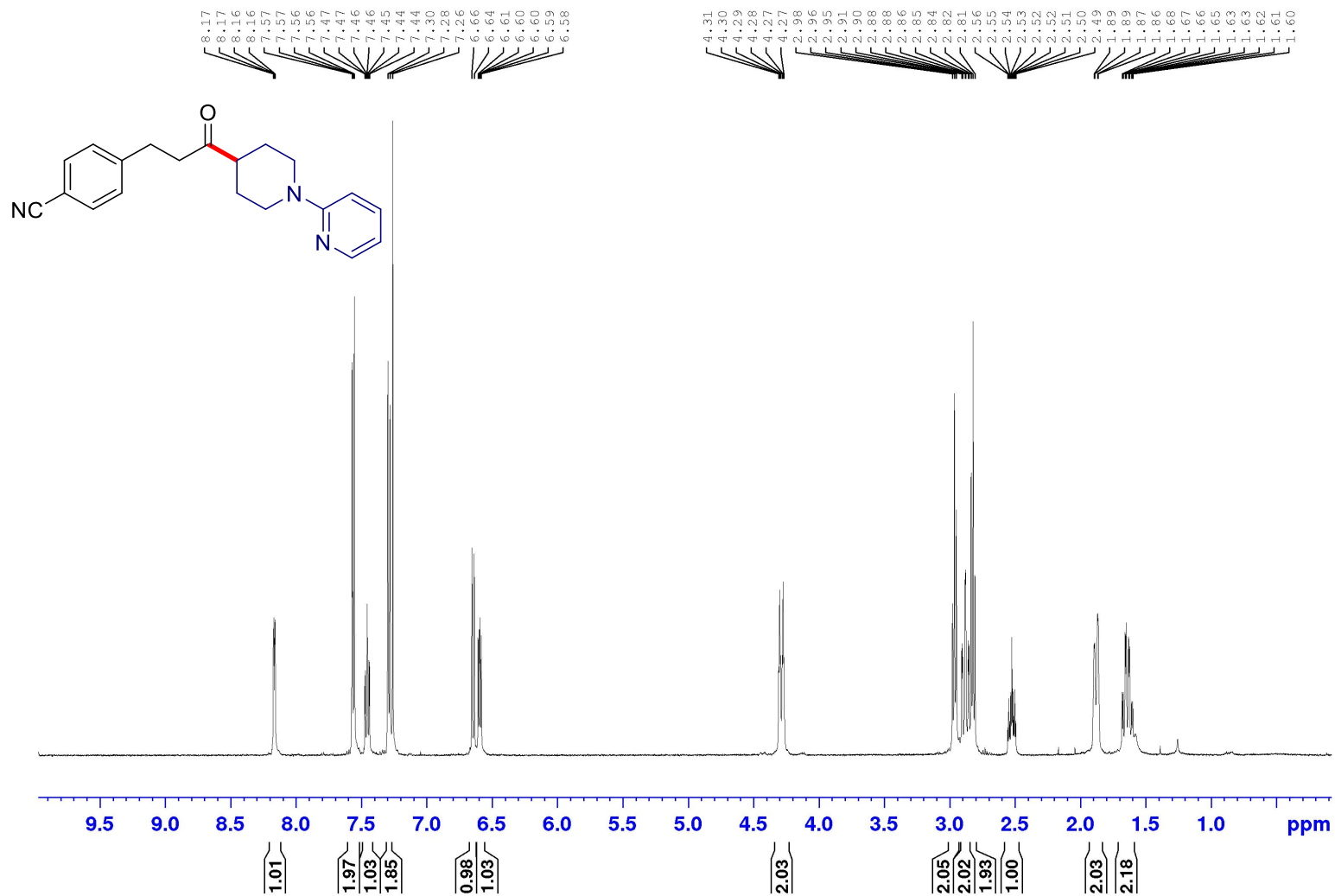
^{13}C NMR (125.8 MHz, CDCl_3) Spectrum of ethyl 3-methyl-4-oxo-6-phenylhexanoate (3i)



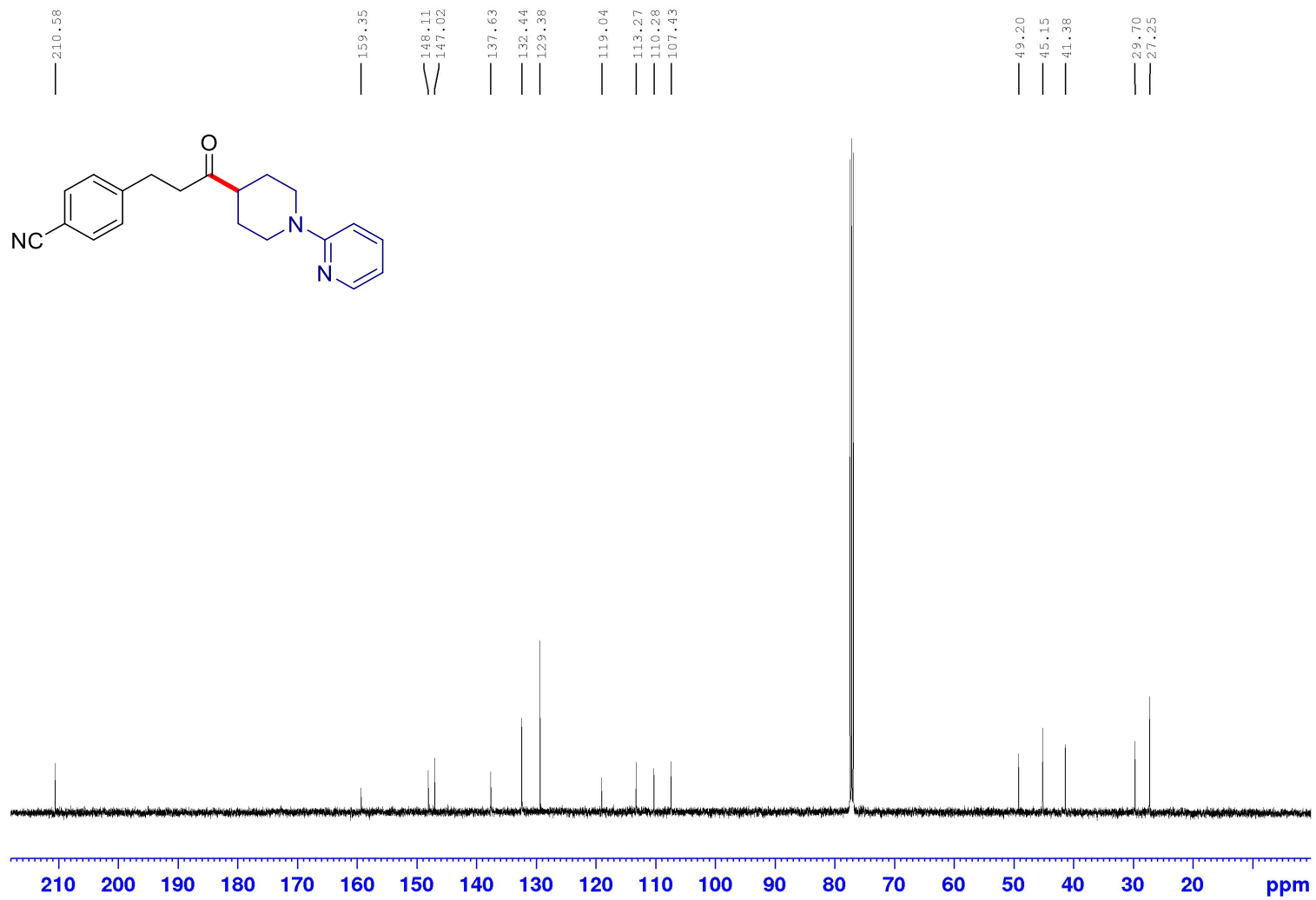
¹H NMR (500 MHz, CDCl₃) Spectrum of benzo[d][1,3]dioxol-5-yl(cyclohexyl)methanone (3j)



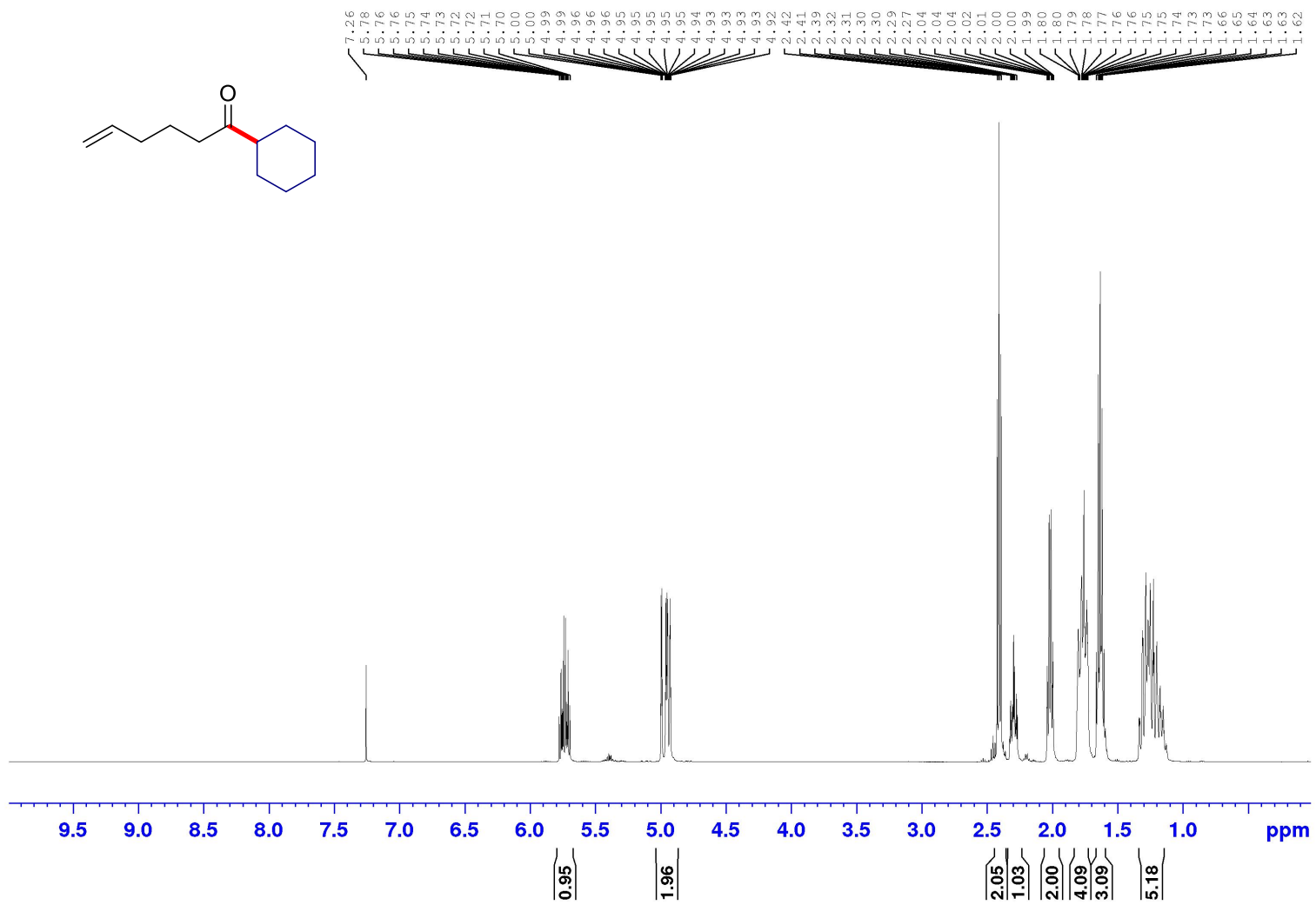
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of benzo[d][1,3]dioxol-5-yl(cyclohexyl)methanone (3j)



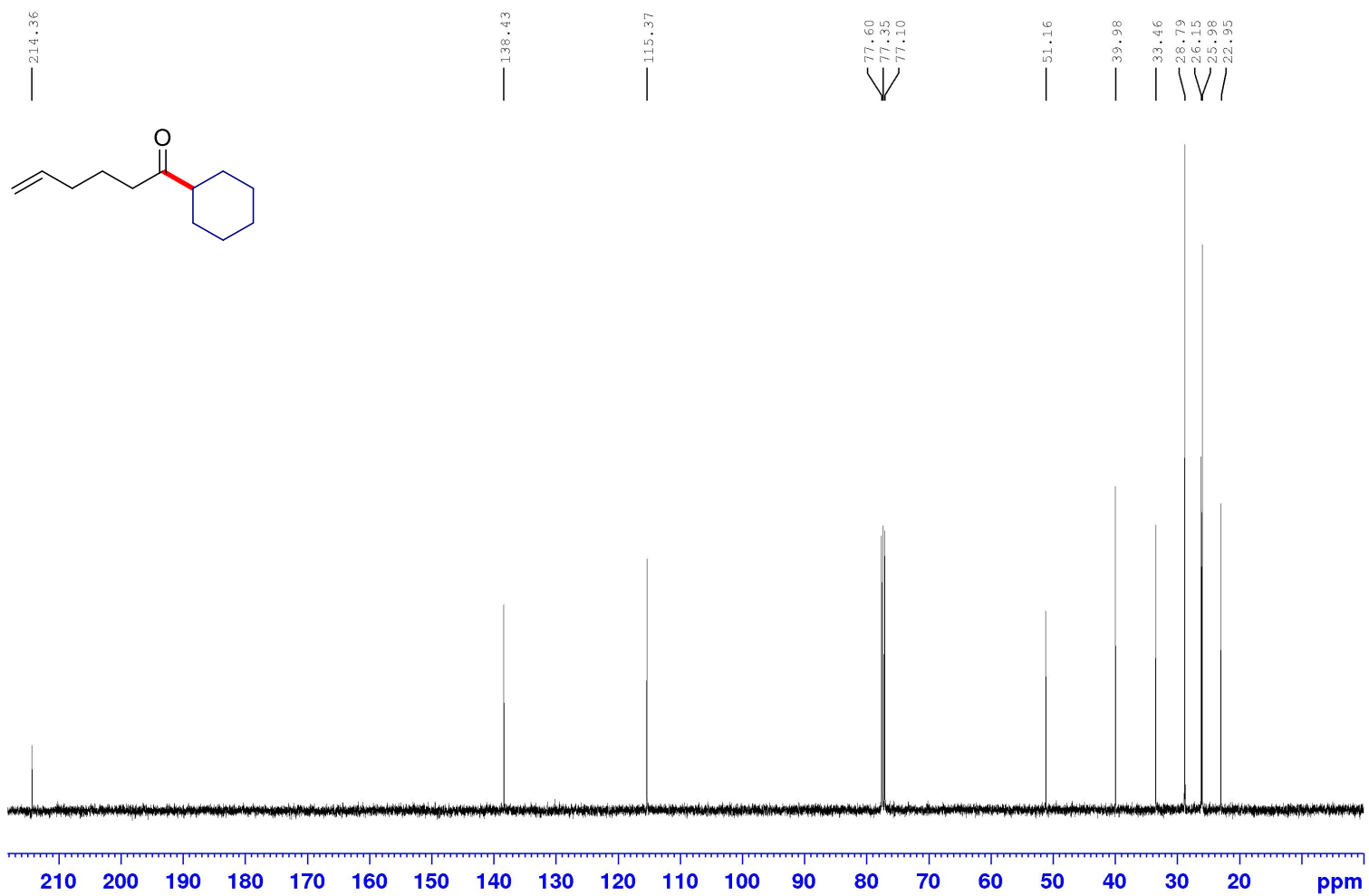
¹H NMR (500 MHz, CDCl₃) Spectrum of 4-(3-oxo-3-(1-(pyridin-2-yl)piperidin-4-yl)propyl)benzonitrile (3k)



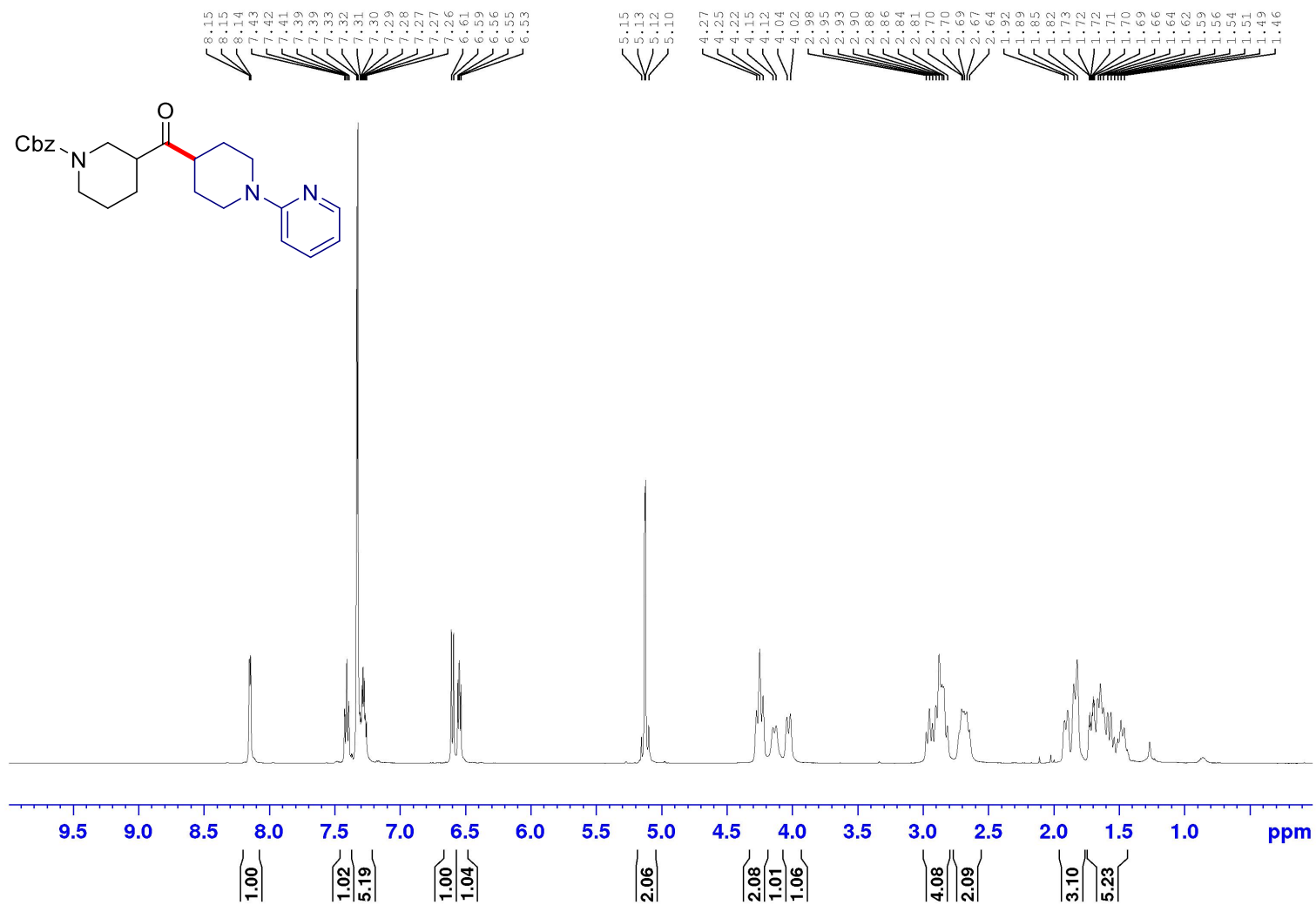
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of 4-(3-oxo-3-(1-(pyridin-2-yl)piperidin-4-yl)propyl)benzonitrile (3k)



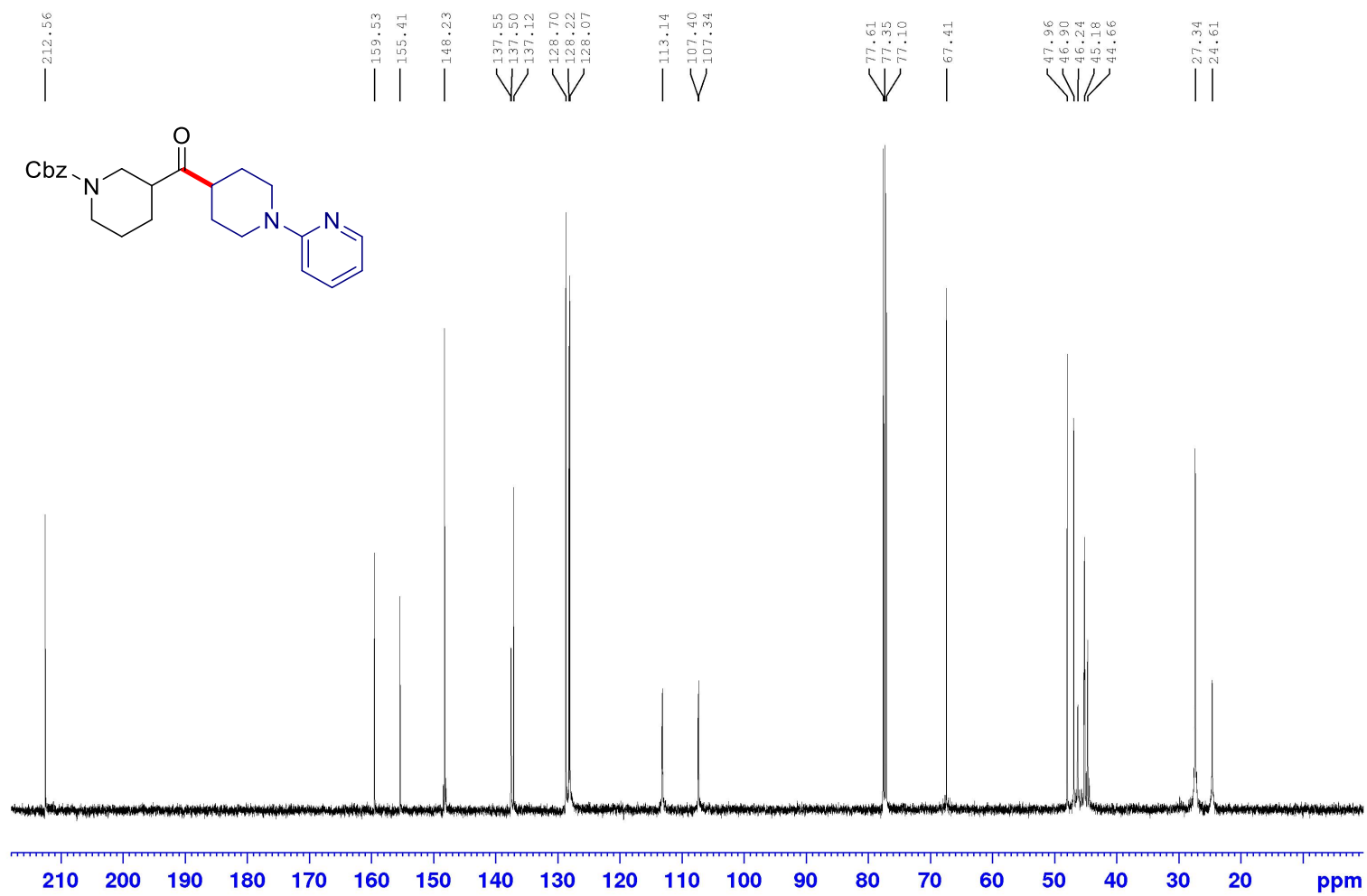
¹H NMR (500 MHz, CDCl₃) Spectrum of 1-cyclohexylhex-5-en-1-one (3l)



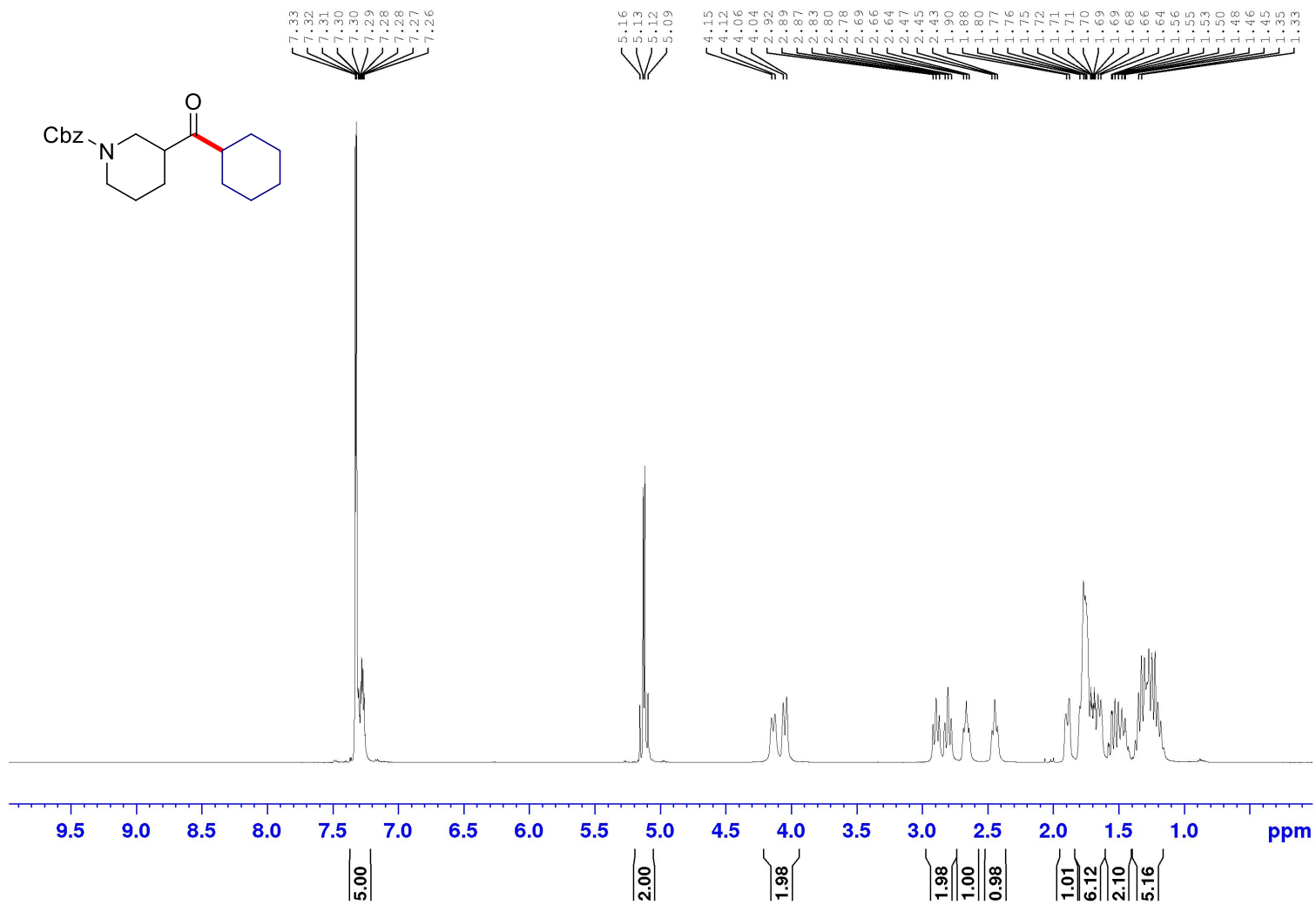
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of 1-cyclohexylhex-5-en-1-one (3l)



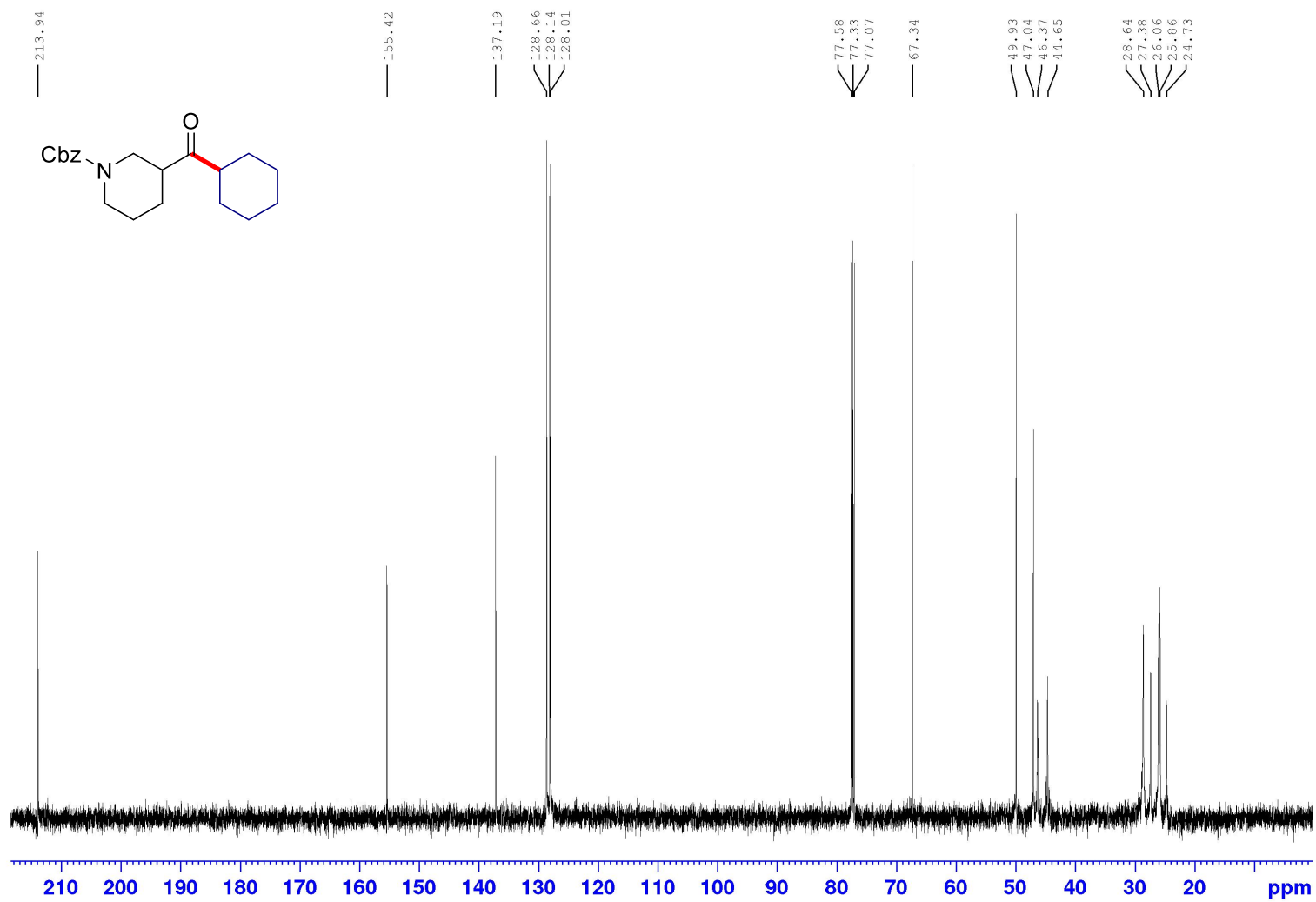
¹H NMR (500 MHz, CDCl₃) Spectrum of benzyl 3-(1-(pyridin-2-yl)piperidine-4-carbonyl)piperidine-1-carboxylate (3m)



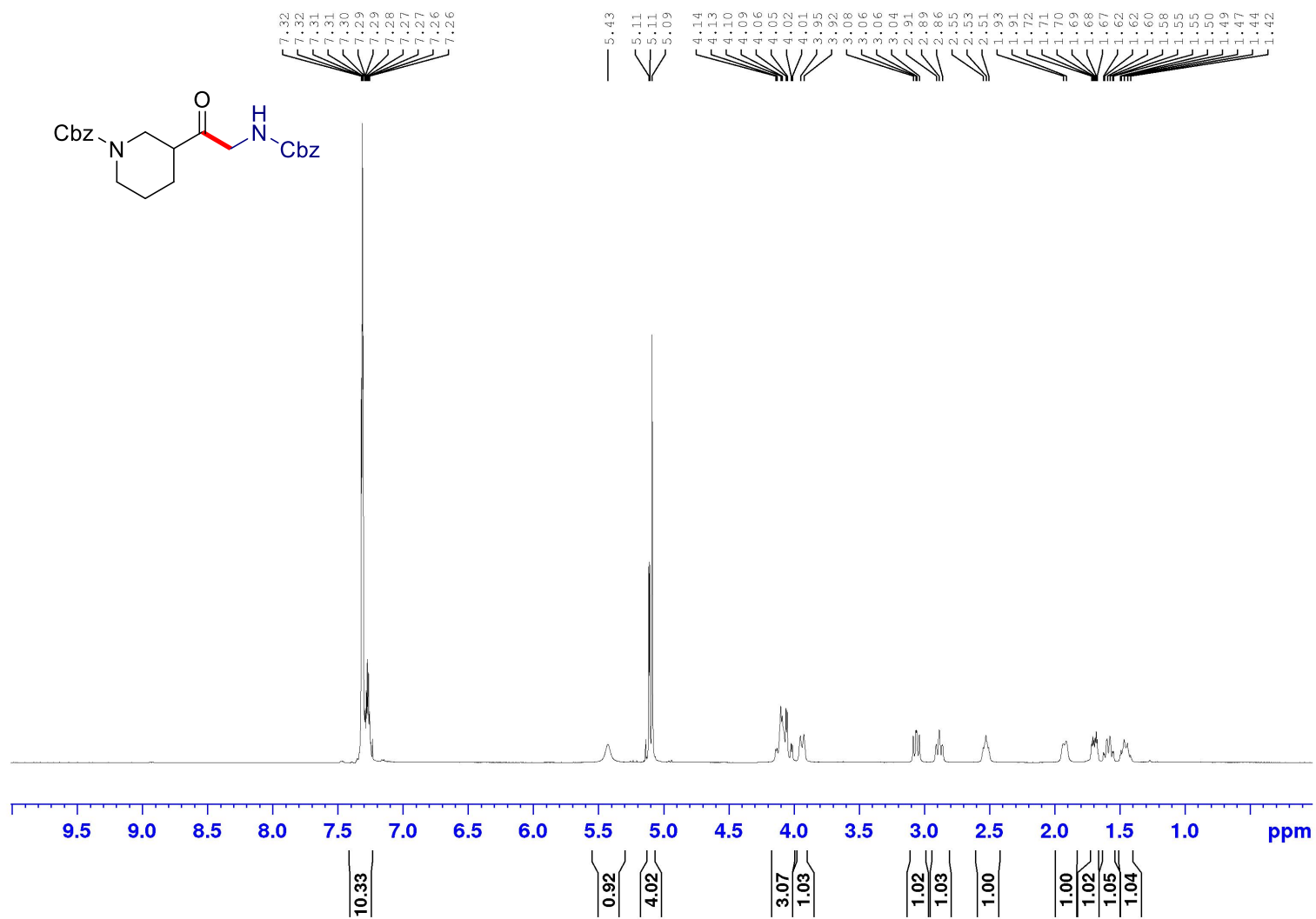
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of benzyl 3-(1-(pyridin-2-yl)piperidine-4-carbonyl)piperidine-1-carboxylate (3m)



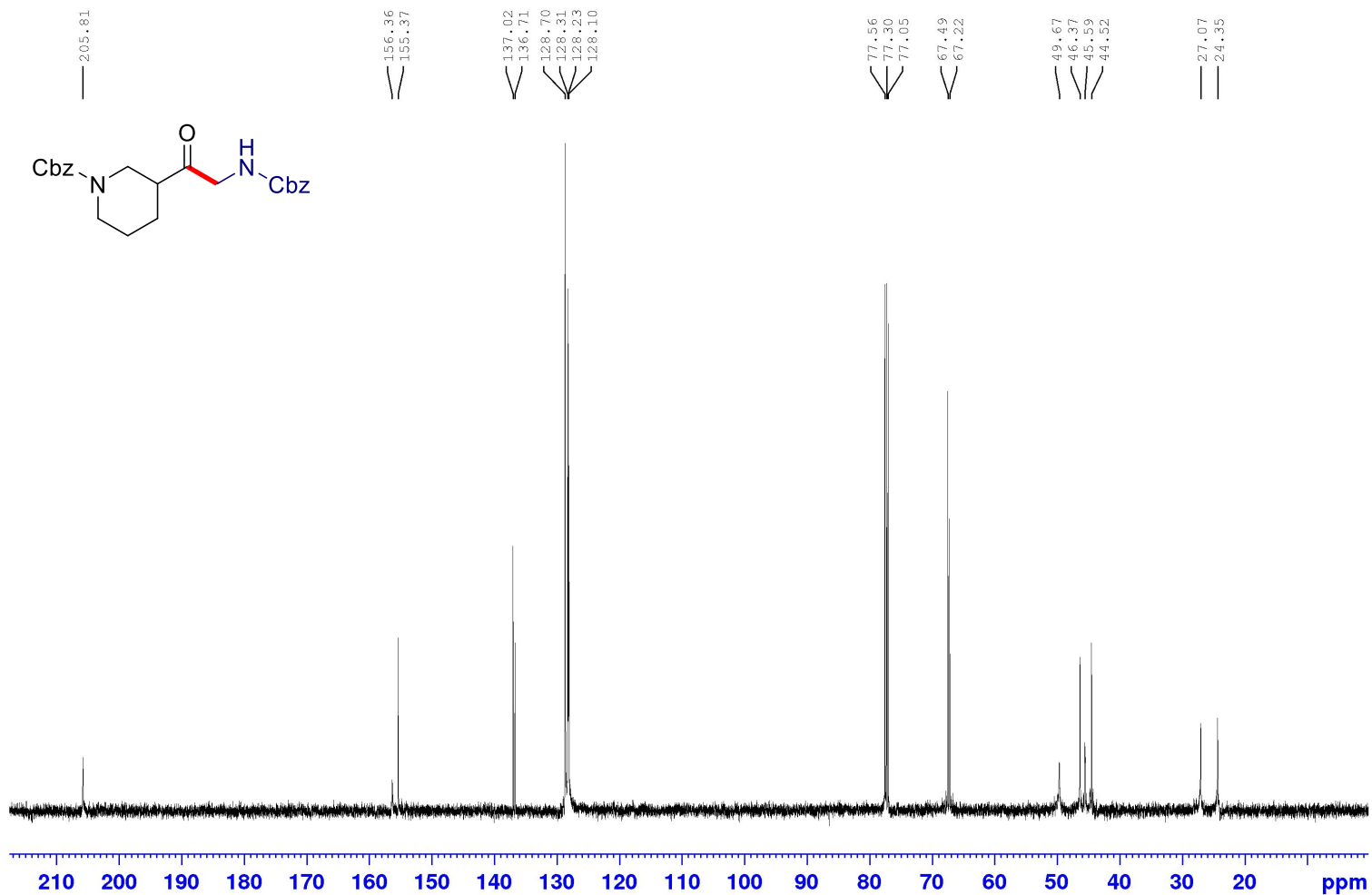
¹H NMR (500 MHz, CDCl₃) Spectrum of benzyl 3-(cyclohexanecarbonyl)piperidine-1-carboxylate (3n)



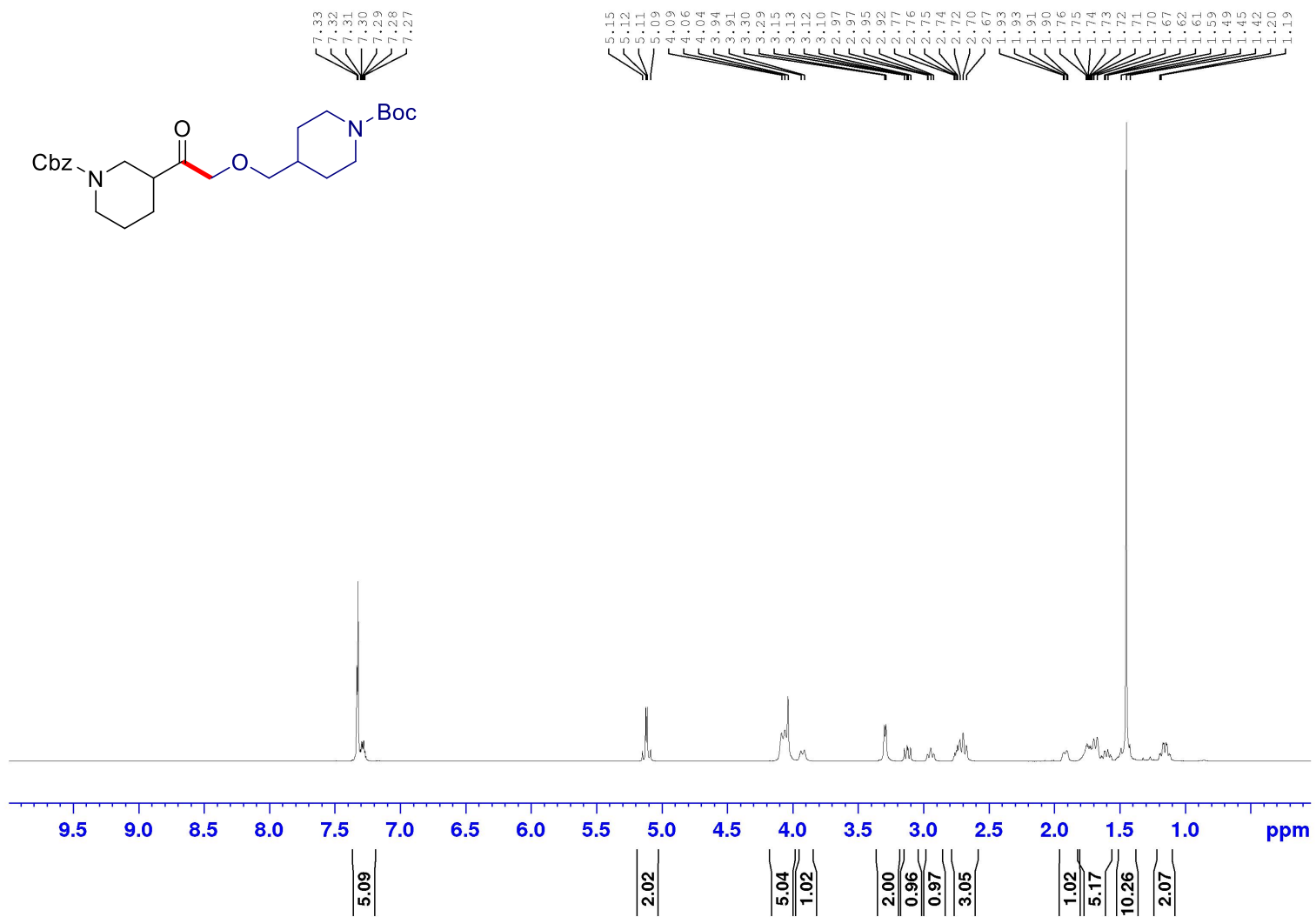
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of benzyl 3-(cyclohexanecarbonyl)piperidine-1-carboxylate (3n)



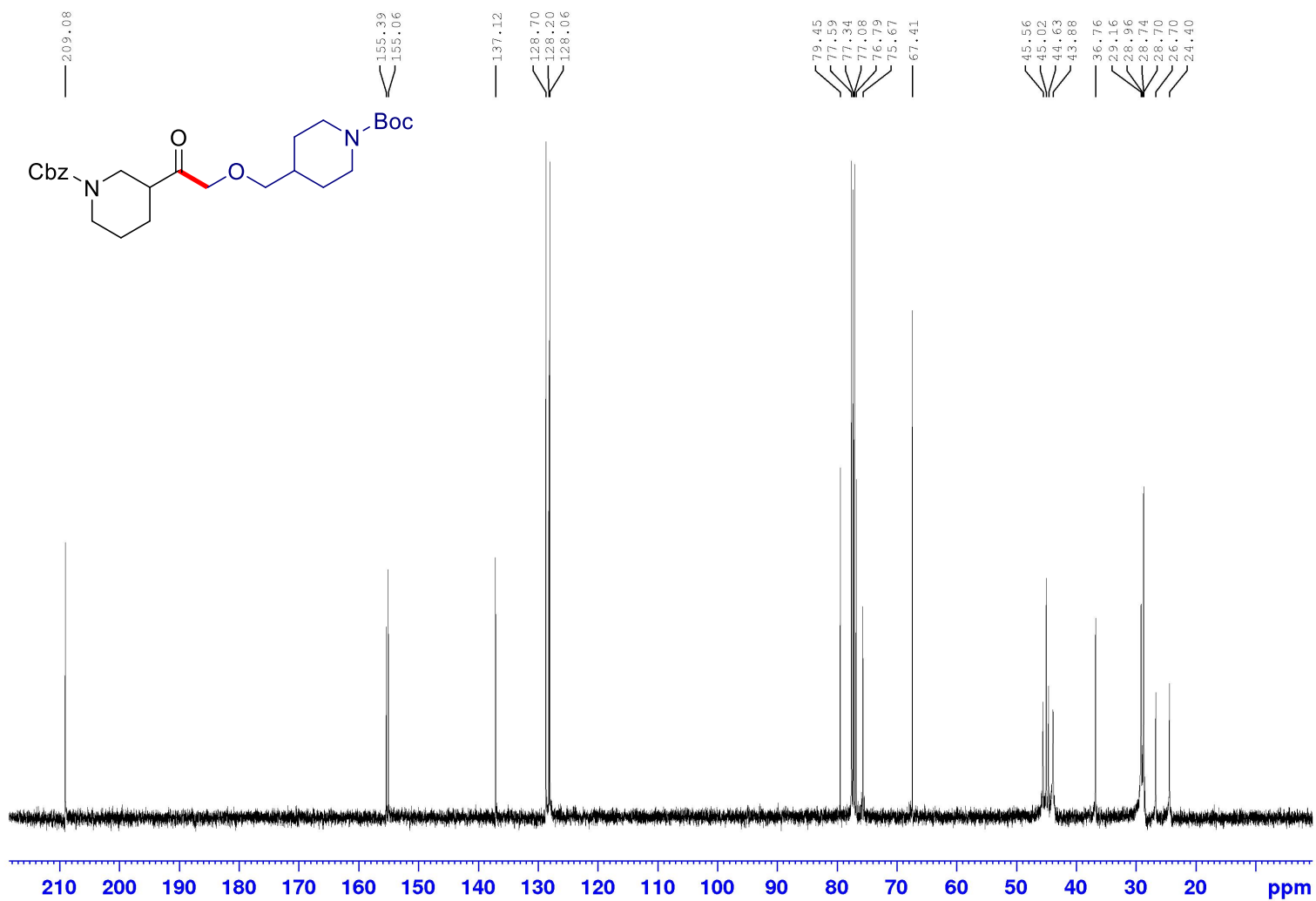
¹H NMR (500 MHz, CDCl₃) Spectrum of benzyl 3-(((benzyloxy)carbonyl)glycyl)piperidine-1-carboxylate (3o)



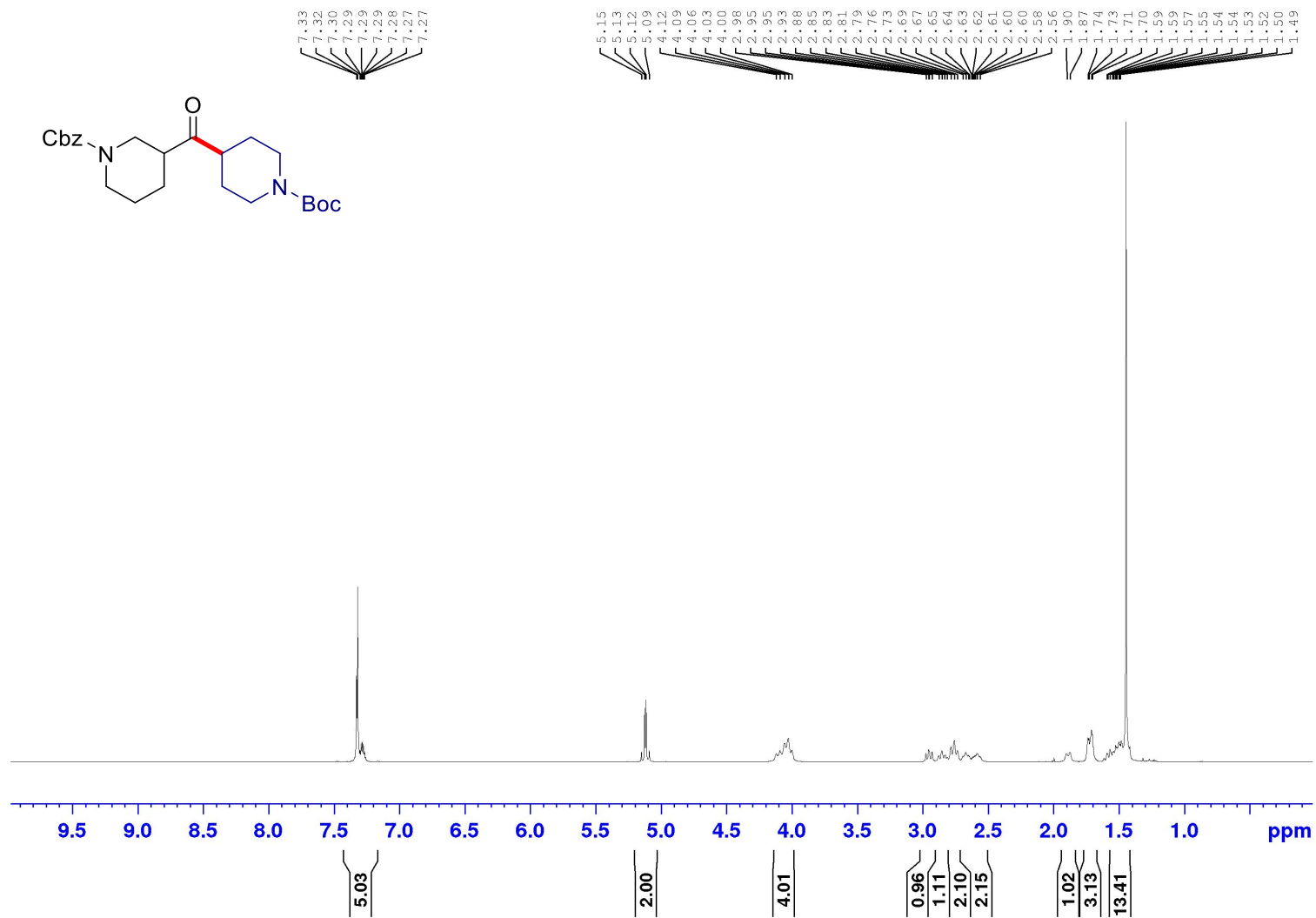
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of benzyl 3-(((benzyloxy)carbonyl)glycyl)piperidine-1-carboxylate (3o)



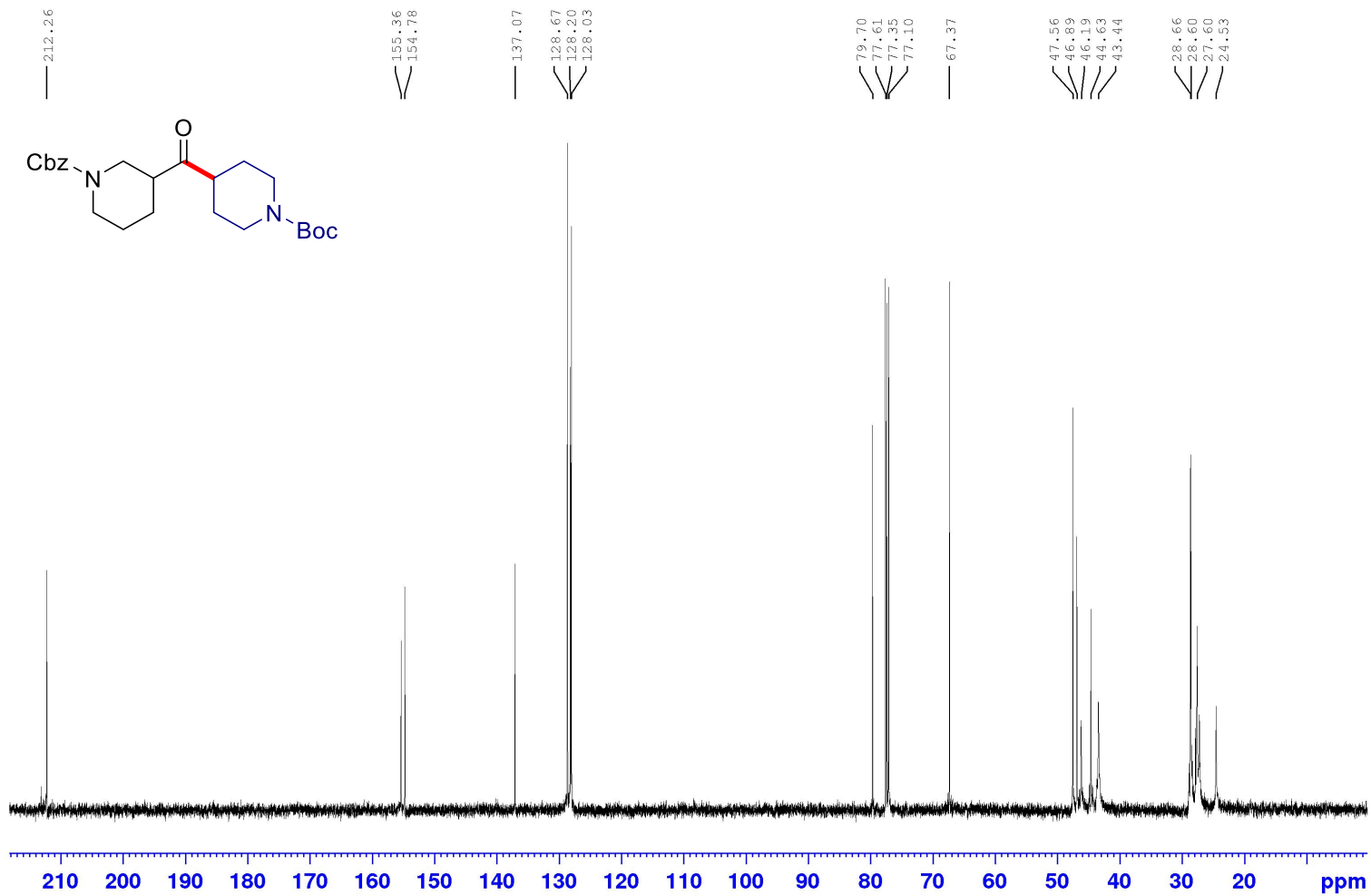
¹H NMR (500 MHz, CDCl₃) Spectrum of benzyl 3-(2-((1-(tert-butoxycarbonyl)piperidin-4-yl)methoxy)acetyl)piperidine-1-carboxylate (3p)



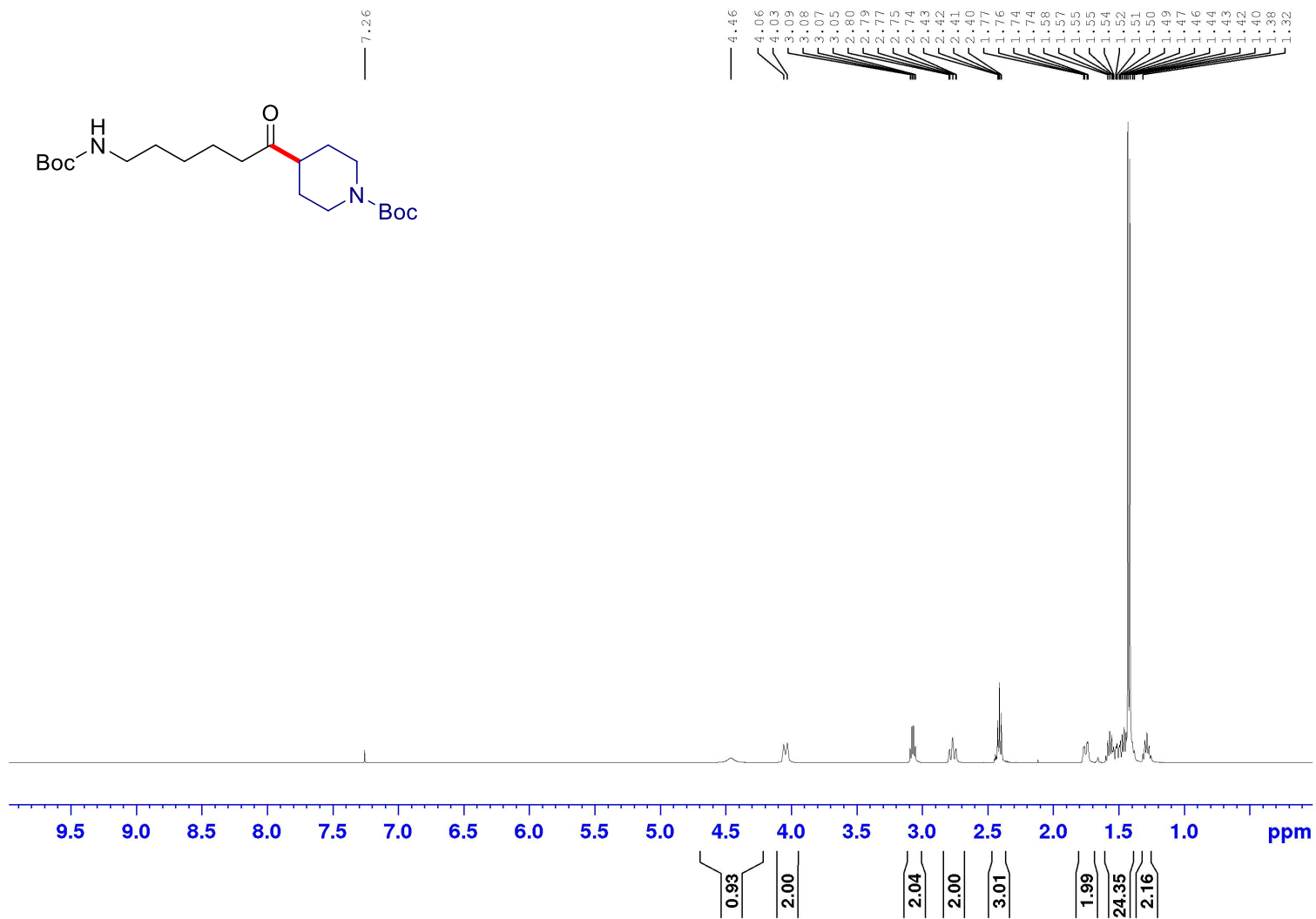
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of benzyl 3-(2-((1-(*tert*-butoxycarbonyl)piperidin-4-yl)methoxy)acetyl)piperidine-1-carboxylate (3p)



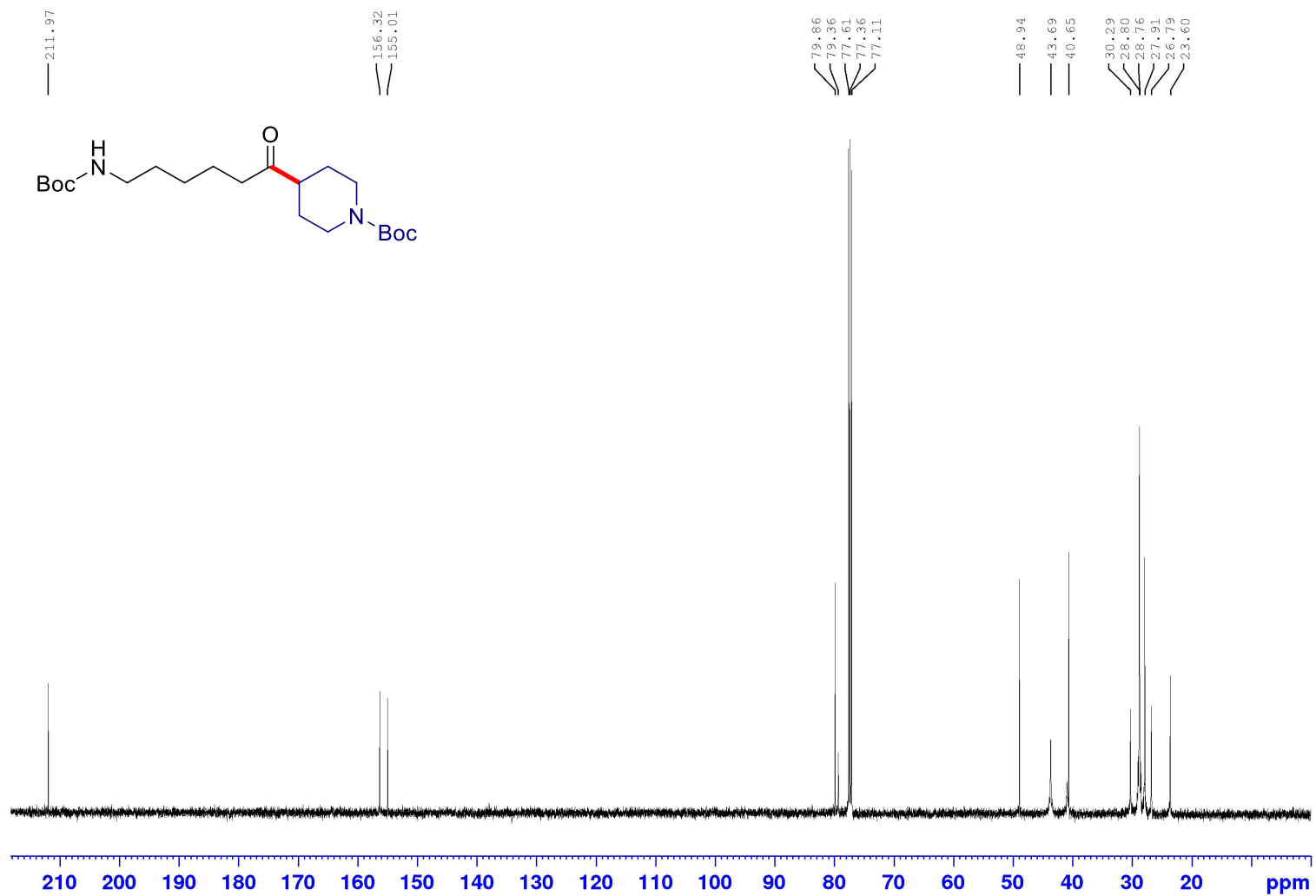
¹H NMR (500 MHz, CDCl₃) Spectrum of benzyl 3-(1-(*tert*-butoxycarbonyl)piperidine-4-carbonyl)piperidine-1-carboxylate (3q)



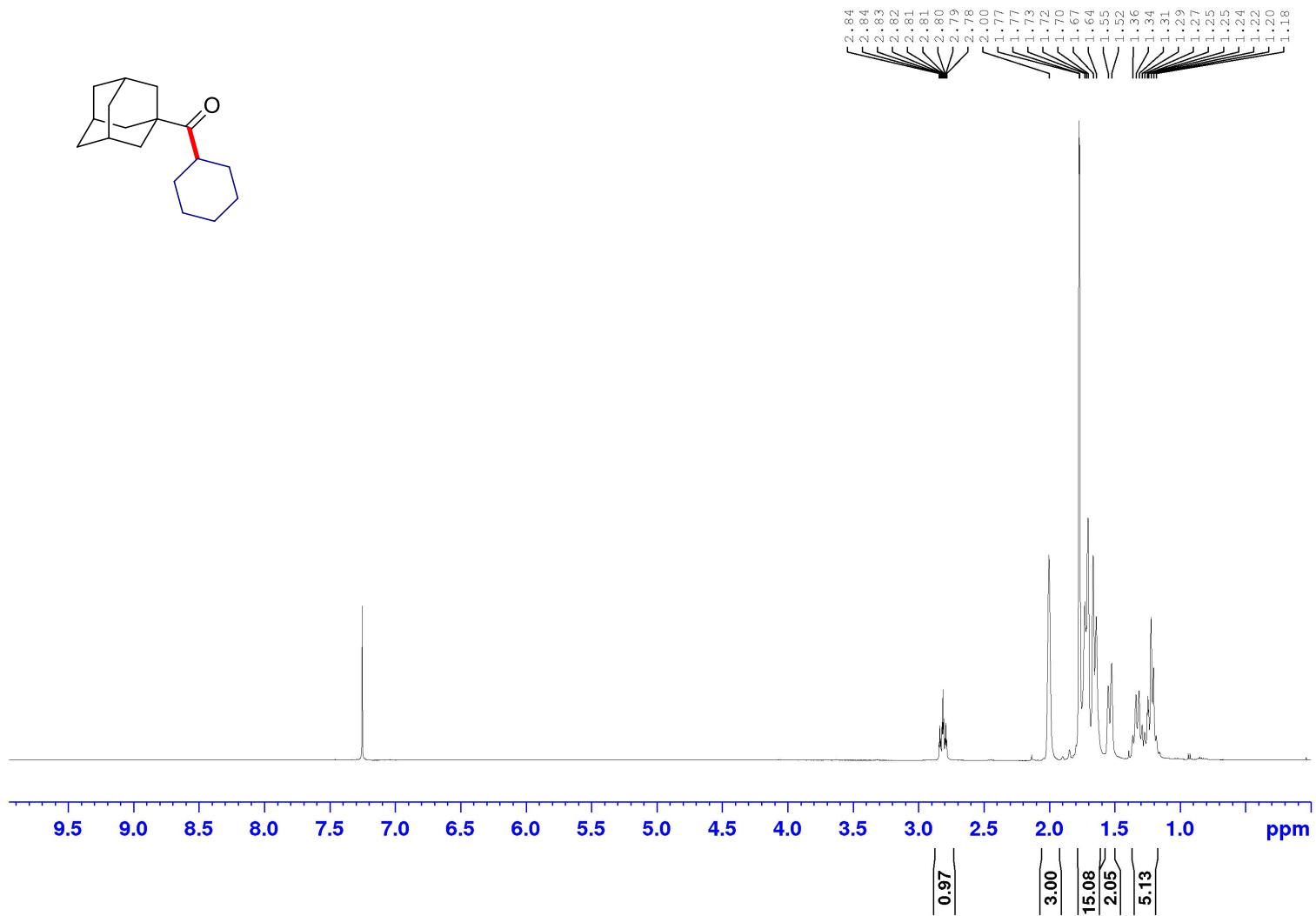
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of benzyl 3-(1-(*tert*-butoxycarbonyl)piperidine-4-carbonyl)piperidine-1-carboxylate (3q)



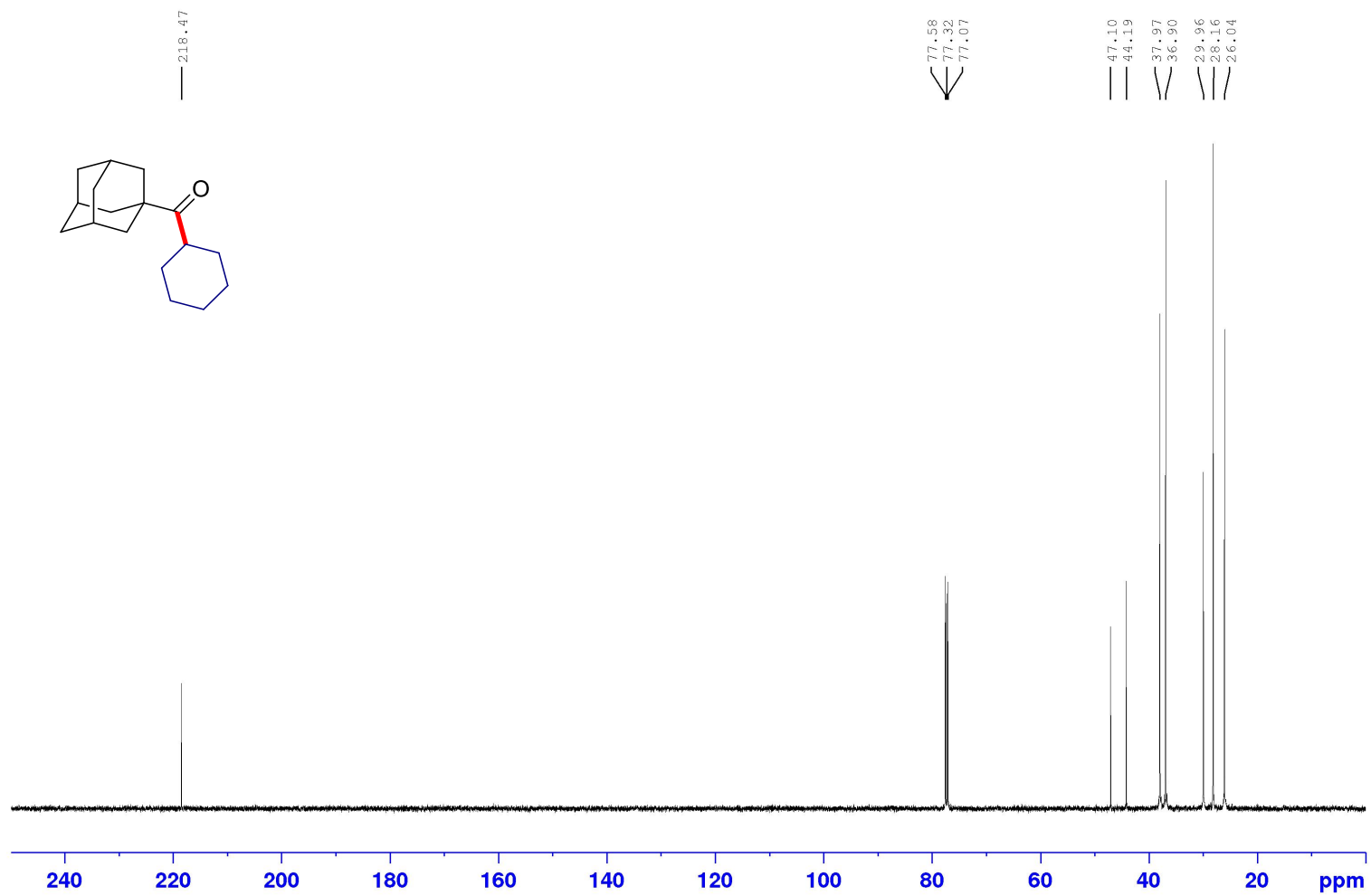
¹H NMR (500 MHz, CDCl₃) Spectrum of *tert*-butyl 4-(6-((*tert*-butoxycarbonyl)amino)hexanoyl)piperidine-1-carboxylate (3r)



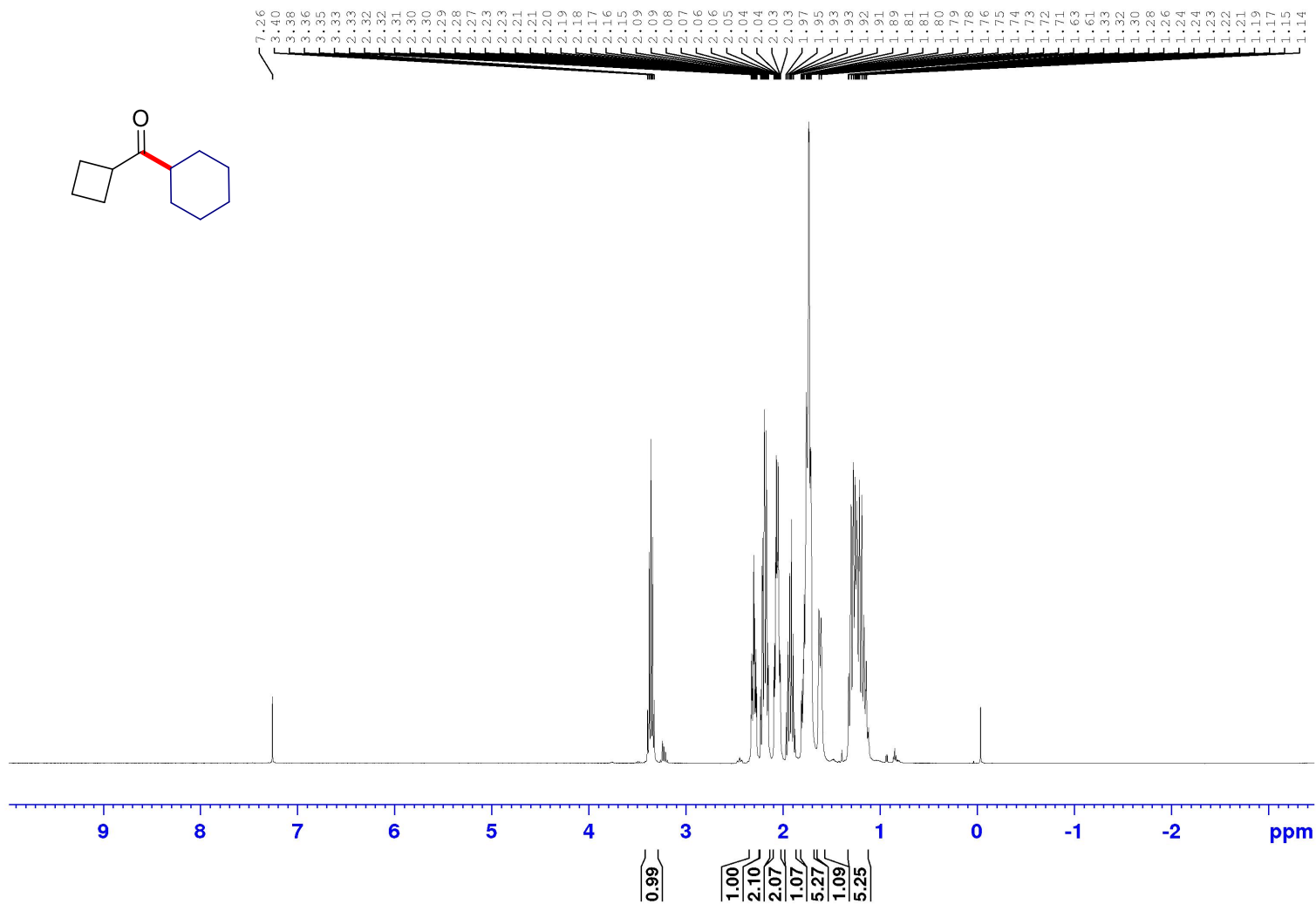
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of *tert*-butyl 4-(6-((*tert*-butoxycarbonyl)amino)hexanoyl)piperidine-1-carboxylate (3r)



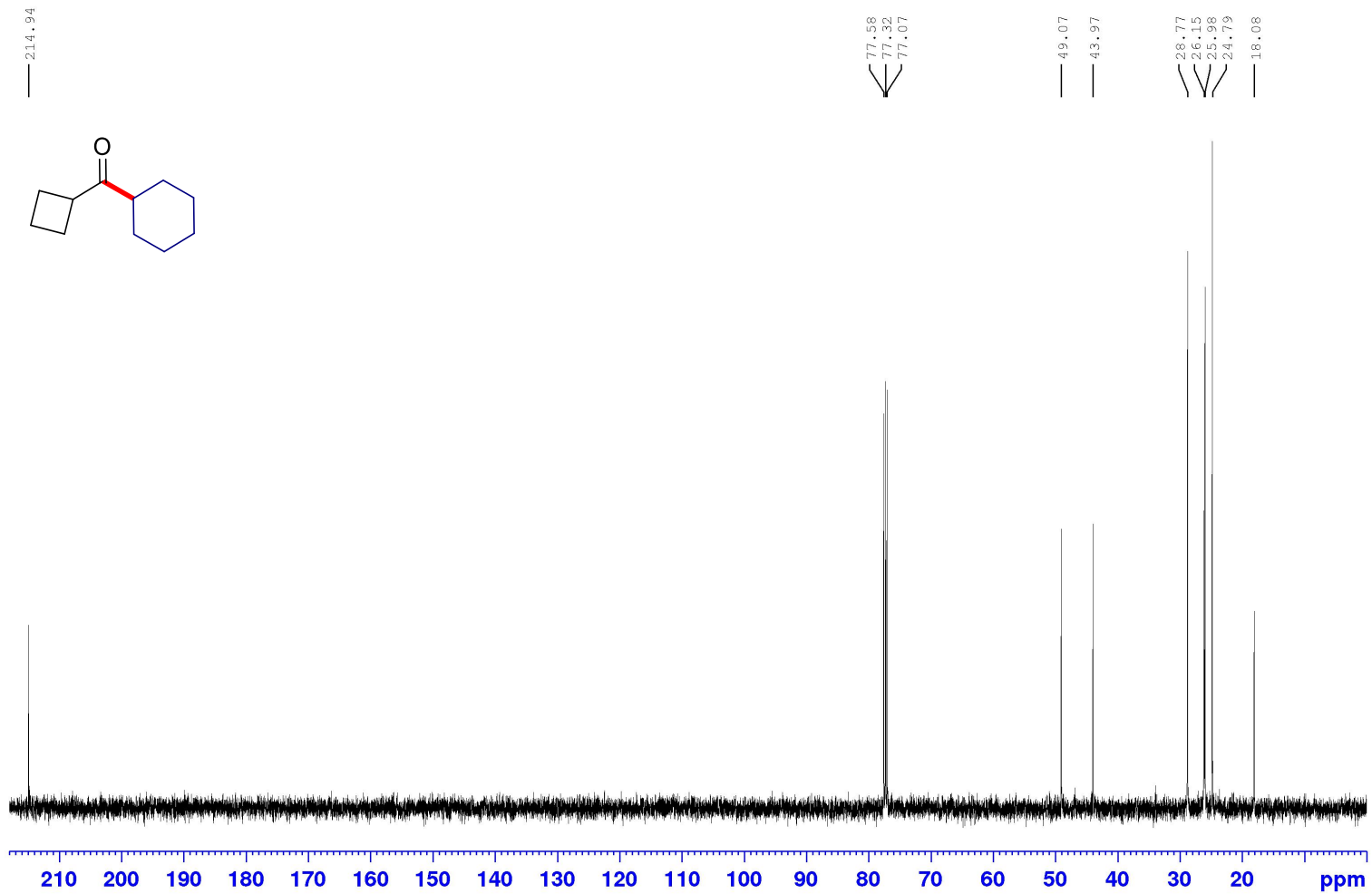
¹H NMR (500 MHz, CDCl₃) Spectrum of (adamantan-1-yl)(cyclohexyl)methanone (3s)



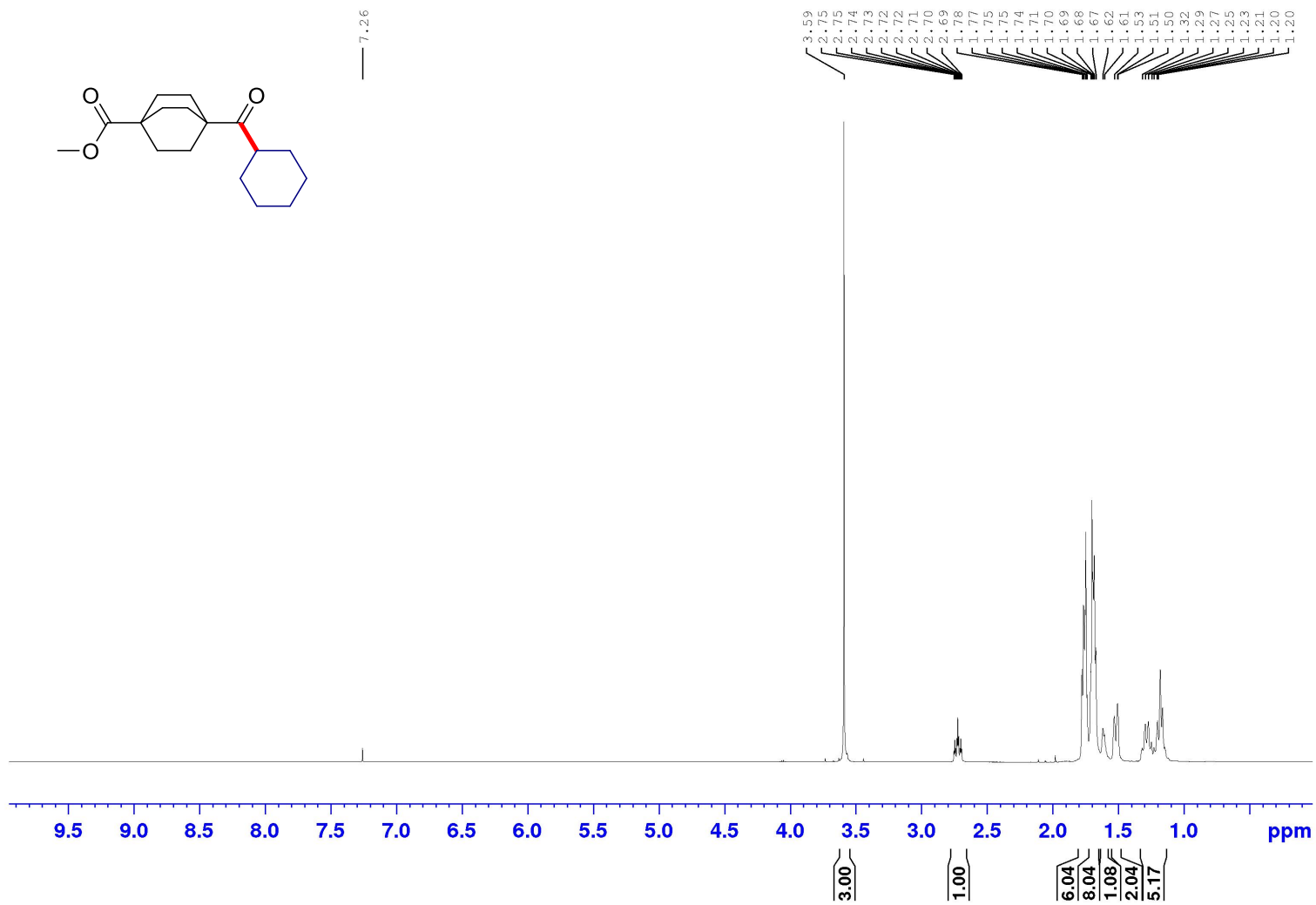
¹³C NMR (125.8 MHz, CDCl₃) Spectrum of (adamantan-1-yl)(cyclohexyl)methanone (3s)



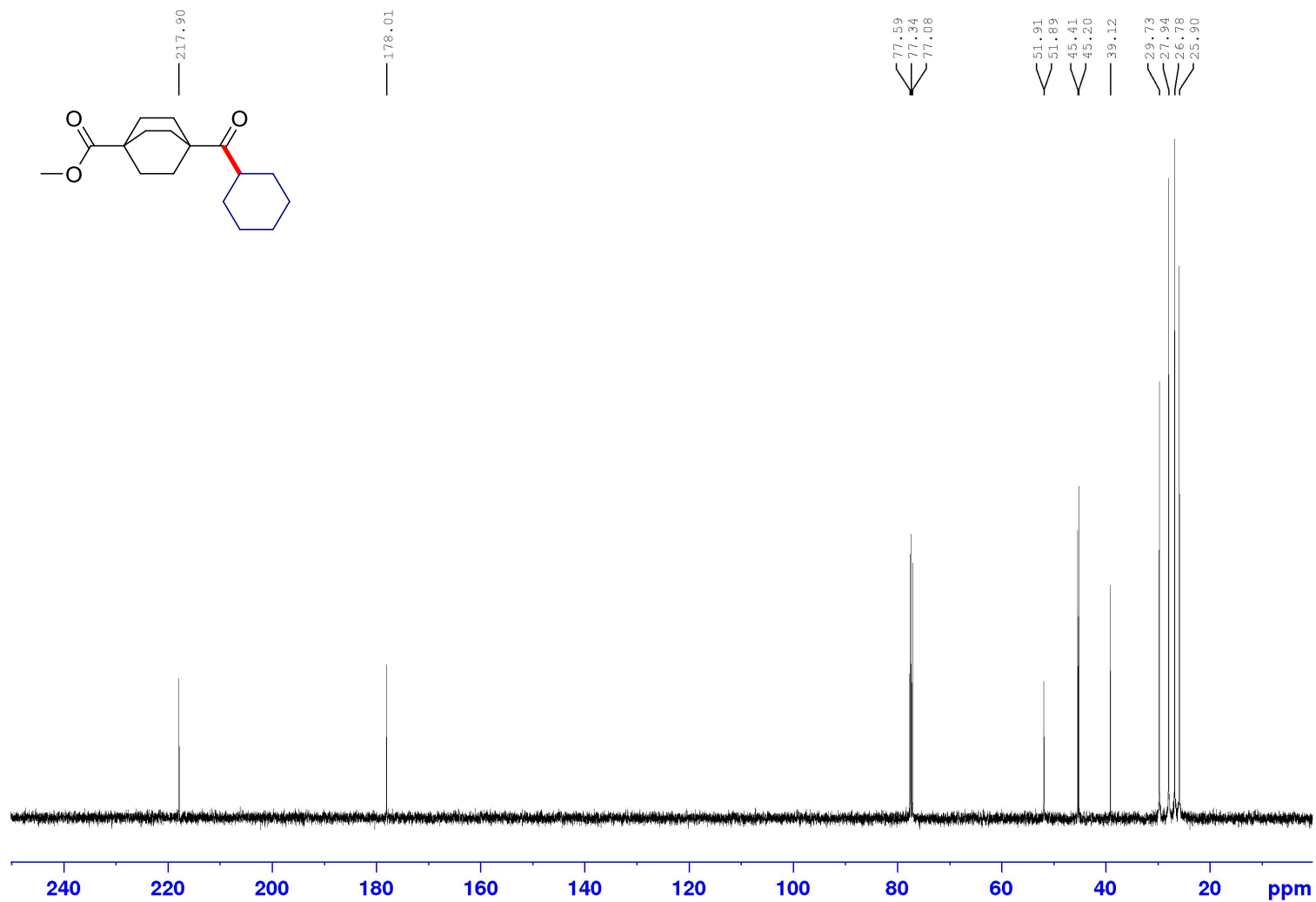
¹H NMR (500 MHz, CDCl₃) Spectrum of cyclobutyl(cyclohexyl)methanone (3t)



¹³C NMR (125.8 MHz, CDCl₃) Spectrum of cyclobutyl(cyclohexyl)methanone (3t)



¹H NMR (500 MHz, CDCl₃) Spectrum of methyl 4-(cyclohexanecarbonyl)bicyclo[2.2.2]octane-1-carboxylate (3u)



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