Electronic Supplementary Material (ESI) for Green Chemistry.

Solar tracker integrated microreactor for real-time sunlight induced ketene formation and API synthesis

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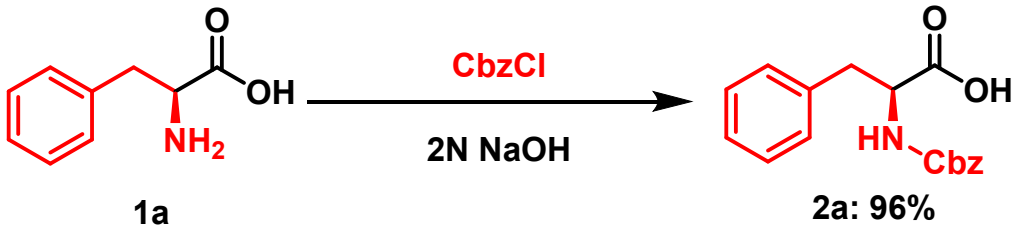
1. General

1.1. Materials: Most of the reagents and chemicals are bought from Sigma-Aldrich and used as such without any further purification. Common organic chemicals and salts were purchased from AVRA chemicals, India. Water used for the experiments was deionized water (18.2 mS conductivity). All work-up and purification procedures were carried out with reagent-grade solvents in air. Analytical thin-layer chromatography (TLC) was performed using analytical chromatography silica gel 60 F254 precoated plates (0.25 mm). The developed chromatogram was analysed by UV lamp (254 nm). PTFE (id = 500 μm) tubing, T-junction, high-purity PFA tubing was purchased from Upchurch IDEX HEALTH &SCIENCE. The photo-batch reactor bought from Lelesil Mumbai India was slightly modified for the continuous flow reaction. Visible light (Blue, Red, Green LED) reactor was bought from the Smartchemsynth Machine Pvt. Ltd, Hyderabad.

1.2. Analysis: High-resolution mass spectra (HRMS) were obtained from a JMS-T100TD instrument (DART) and Thermo Fisher Scientific Exactive (APCI). Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker 600, 500, 400 or 300 MHz in $CDCl_3$ or DMSO-d_6 solvent. Chemical shifts for ^1H NMR are expressed in parts per million (ppm) relative to tetramethylsilane (δ 0.00 ppm). Chemical shifts for ^{13}C NMR are expressed in ppm relative to $CDCl_3$ (δ 77.0 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, dd = doublet of doublets, t = triplet, q = quartet, quin = quintet, sext = sextet, m = multiplet), coupling constant (Hz). GC/MS analysis was conducted on an Shimadzu technology GCMS-QP2010 instrument equipped with a HP-5 column (30 m × 0.25 mm, Hewlett-Packard) and inbuilt MS 5975C VL MSD system with triple axis detector.

2. Synthesis of starting materials:

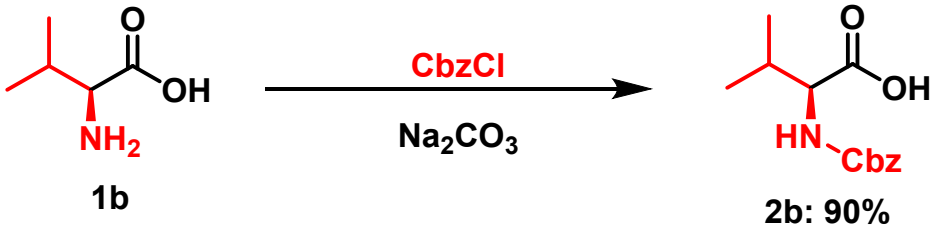
2.1 General procedure of compound 2a.



1a **2a: 96%**

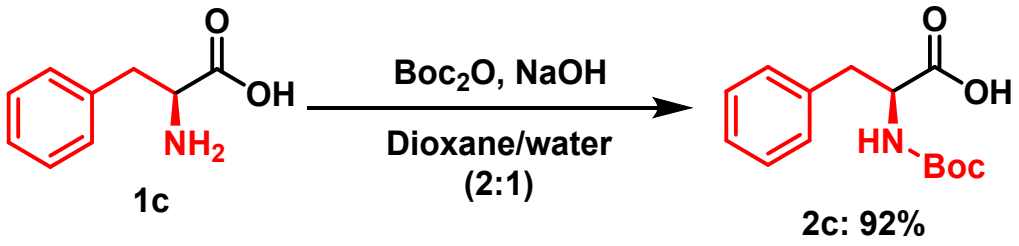
L- Phenylalanine (10.0 g, 60.5 mmol) was dissolved in 2*N* NaOH (50 mL), freshly distilled benzyloxy carbonyl chloride (10.5 mL, 73.3 mmol) was added drop wise under stirring condition and temperature was maintained at 0 °C. After the addition of the carbonyl chloride further reaction mixture was stirred at 0 °C for 30 min. and at room temperature for 1 h. The reaction mixture was washed with diethyl ether (50 mL), acidified to pH = 4 with 2*N* HCl and extracted with ethyl acetate (3 x 80 mL), the combined organic layer was washed with water (50 mL), brine (50 mL), dried (Na_2SO_4) and concentrated to give yield (17.4 g, 96%) as a white solid and **mp**: 85 – 87 °C. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.35 – 7.14 (m, 10H), 5.18 (d, *J* = 7.1 Hz, 1H), 5.10 (s, 2H), 4.72 – 4.67 (m, 1H), 3.23 – 3.08 (m, 2H). **^{13}C NMR (125 MHz, $CDCl_3$)** δ 175.7, 156.0, 136.2, 135.6, 129.5, 128.8, 128.7, 128.4, 128.2, 127.4, 67.3, 54.7, 37.9. **IR (ν_{max}):** 3334.57, 3035.75, 1711.05, 1521.24 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{17}H_{17}NNaO_4$** $[M+Na]^+$ 322.1055, Found 322.1064. Analytical data are well matched with reported literature.[1]

2.2 General procedure of compound 2b.



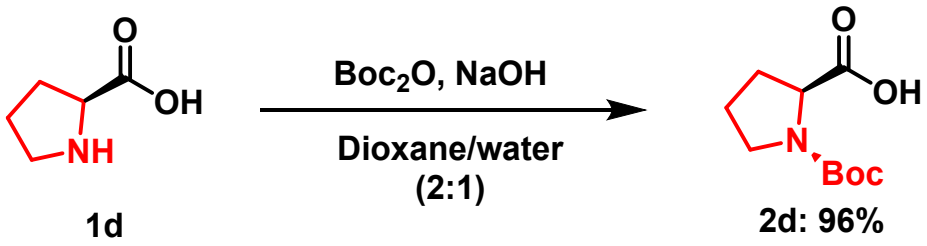
A 500 mL two neck round-bottom flask equipped with a magnetic stir bar was charged with distilled H_2O (60 mL) and Na_2CO_3 (8.13 g, 76.83 mmol). The resulting solution was cooled to 0 °C in an ice bath and further added the L-Valine (9g, 76.83). The solution was stirred to get the homogeneous solution. Further, benzyloxy carbonyl chloride (12.1 mL, 84.5 mmol) was added in dropwise manner. The reaction mixture was stirred at room temperature for 12 h. After completion of the reaction, the solution was extracted with Et_2O (3 × 50 mL), and the organic layers were discarded. The aqueous layer was again cooled to 0 °C in an ice bath, and concentrated HCl was added dropwise until the pH = 2. The aqueous solution was extracted with Et_2O (3 × 100 mL). The organic layers were combined, dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuum to give the product, yield **2b** (17.4g, 90%) as a white solid and **mp**: 60-62 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** 7.37 – 7.32 (m, 5H), 5.34 (d, *J* = 8.4 Hz, 1H), 5.12 (s, 2H), 4.37 – 4.33 (m, 1H), 2.27 – 2.18 (m, 1H), 0.96 (dd, *J* = 30.0, 6.8 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 176.78, 156.56, 136.22, 128.68, 128.38, 128.28, 67.35, 58.99, 31.15, 19.13, 17.47. **IR (ν_{max}):** 3427.34, 3214.15, 2978.42, 1712.31, 1508.37 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{13}H_{18}NO_4$** $[M+H]^+$ 252.1230, Found 252.1222. Analytical data are well matched with reported literature.[2]

2.3 General procedure of compound 2c.



L-Phenylalanine (8 g, 48.4 mmol) was dissolved in dioxane-water mixture (120:60) and the reaction mixture was stirred at room temperature for 5 min. (partly soluble). Addition of 1M NaOH (60 ml) to this mixture dissolves the reactant thereby affording a miscible solution. The reaction mixture was then cooled to 0 °C and stirred for 5 min. Finally, $(Boc)_2O$ (13.5 mL, 58.08 mmol) was added and the reaction mixture was allowed to stir at 0°C for 30 min. The reaction mixture was stirred at room temperature for 12 h. After completion of the reaction solution was extracted with Et_2O (3 × 80 mL), and the organic layers were discarded. The aqueous layer was again cooled to 0 °C in an ice bath, and concentrated HCl was added dropwise until the pH = 2. The aqueous solution was extracted with Et_2O (3 × 80 mL). The organic layers were combined, dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuum to give the product, yield **2c** (11.8 g, 92%) as a white solid; **mp**: 85 – 87 °C. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 9.91 (br. s, 1H), 7.31 – 7.17(m, 5H), 5.01 (d, *J* = 6.9 Hz, 1H), 4.64 – 4.39 (m, 1H), 3.21 – 3.05 (m, 2H), 1.35 (d, *J* = 63.1 Hz, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 176.28, 155.55, 135.95, 129.52, 128.75, 127.25, 80.48, 54.41, 37.90, 28.42, 28.14. **IR (ν_{max}):** 3427.34, 3214.15, 2978.42, 1712.31, 1508.37 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{14}H_{18}NNaO_4$** $[M+Na]^+$ 288.1212, Found 288.1207. Analytical data are well matched with reported literature.[3]

2.4 General procedure of compound 2d.



To a solution of (L)-proline (7.5 g, 65.1 mmol) in aq. NaOH (1M, 131 mL) and dioxane 40 ml and 20 mL of water at 0 °C was added Boc_2O (17.4 mL, 77 mmol) portion wise over 20 min. The resulting mixture was stirred at 0 °C for 30 min, then allowed to warm to 23 °C and stirred overnight. The organic solvent was removed under reduced pressure. The remaining aqueous solution was acidified to pH ~2 with aq. $KHSO_4$ (1 M). The aqueous solution was extracted with $CHCl_3$ (3 x 150 mL). The combined organic layers were washed with brine (150 mL), dried over Na_2SO_4, and concentrated by rotary evaporation to afford carbamate (13.5 g, 96% yield, as a white solid, which was sufficiently pure to be used for the next step. **mp**: 132 – 134 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 10.17 (br. s, 1H), 4.35– 4.21 (m, 1H), 3.58 – 3.32 (m, 2H), 2.29–2.22 (m, 1H), 2.14 – 2.02 (m, 1H), 2.00 – 1.85 (m, 2H), 1.44 (d, *J* = 23.1 Hz, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 178.90, 175.65, 156.16, 153.91, 81.22, 80.36, 59.06, 58.94, 46.94, 46.34, 30.84, 28.79, 28.39, 28.28, 24.31, 23.66. **IR (ν_{max}):** 3462.94, 2978.03, 2885.26, 1696.47 cm^{-1}. Analytical data are well matched with reported literature.[4]

3. Continuous flow diazo-methane (diazo-M) generator for multi-gram scale.

3.1 Fabrication of a pilot scale liquid-liquid separator: As illustrated in Figure **S1**, laser ablation on PTFE film was employed to fabricate the proposed dual channel device. First of all, layers of 1000 μm thick PTFE films were ablated by UV laser, to form 110 square well (12 mm width × 12 mm length) and direction layer contains the rectangular 109 wells (6 mm width × 15 mm length).[5] The 4-corners of each film were drilled for holes (1 mm dia.) to align the film patterns. After laser ablation, the films were cleaned by washing with acetone under ultrasonic and dried. Polytetrafluoroethylene (PTFE) membrane (Whatmann, 0.45 μm pore, 47 mm dia.) sandwiched by two sheets of PTFE film with identical dimension of microchannel were placed between Nylon frame holder, which were aligned each other by inserting metal bolt through the holes at the film corners. Finally, Nylon frame holder was tightly pressed by screw to seal the device with no leak.

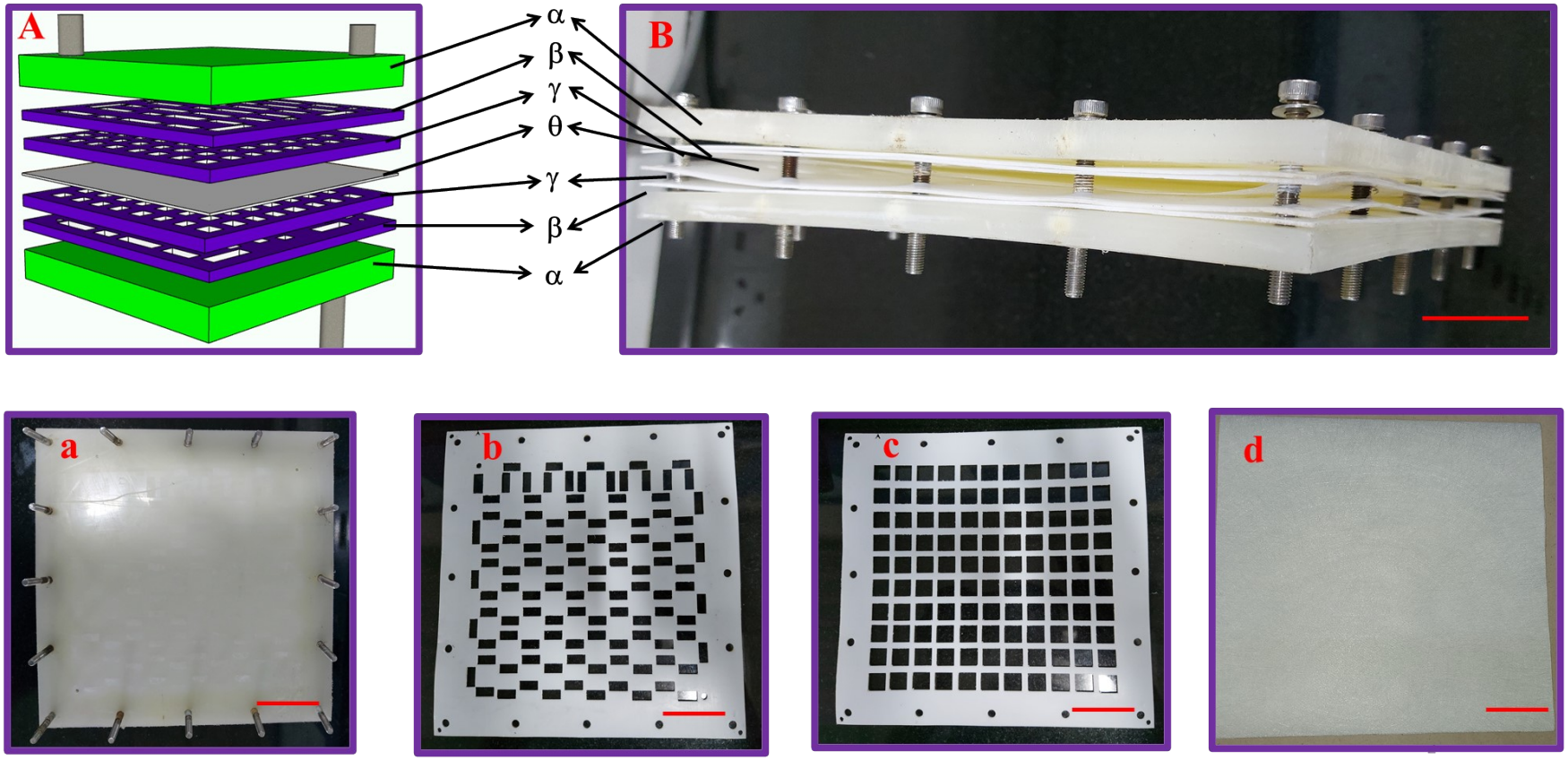
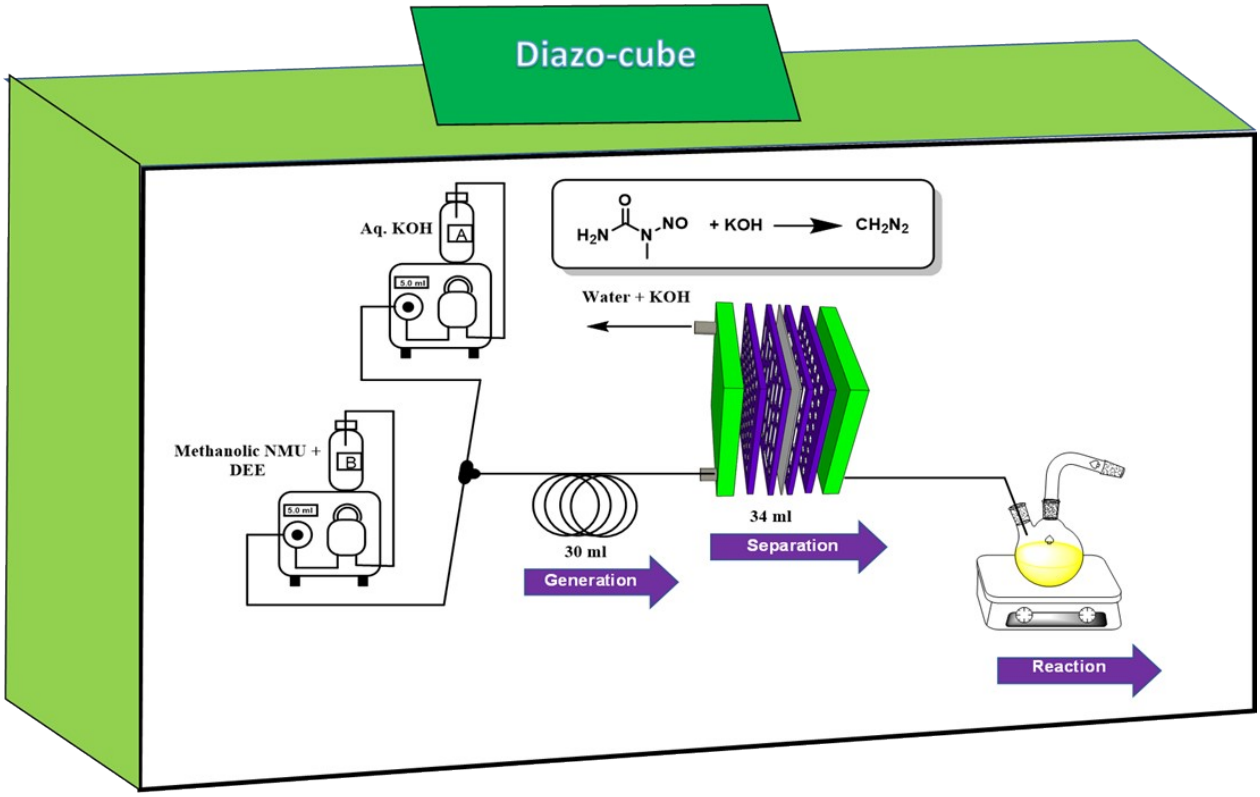


Figure S1. Schematic diagram of pilot scale liquid-liquid separator, (A) 3D model; (B) original photograph; (a) nylon fabric sheet (b) direction channel; (c) square well PTFE channel; (d) polypropylene coated PTFE membrane, bar represent the 5 cm.

3.2 Integrated system for the diazo-insertion in to the organic acid.



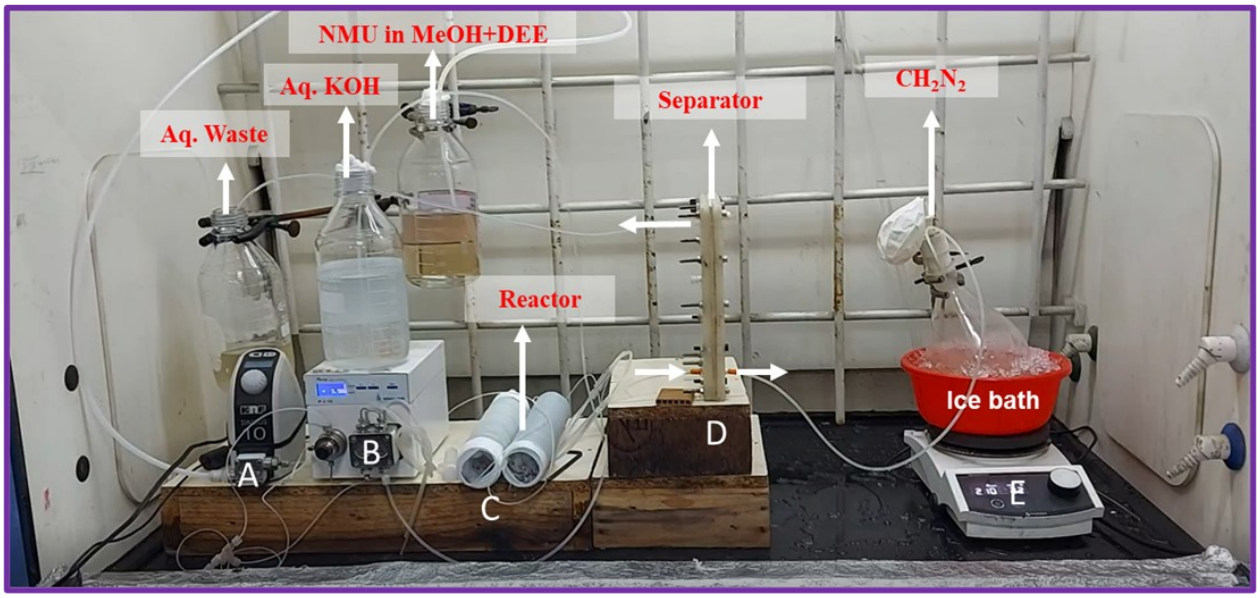
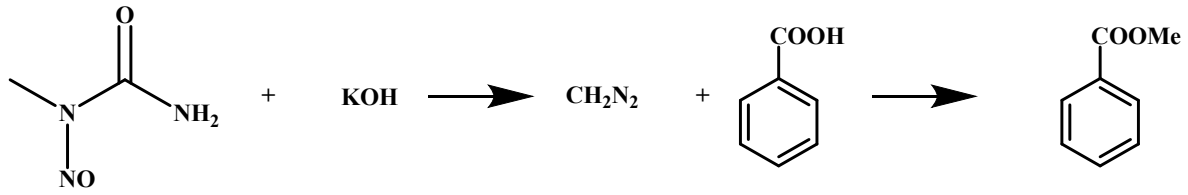


Figure S2. Integrated set-up for the continuous flow diazo-methane generation and insertion in to organic moiety.

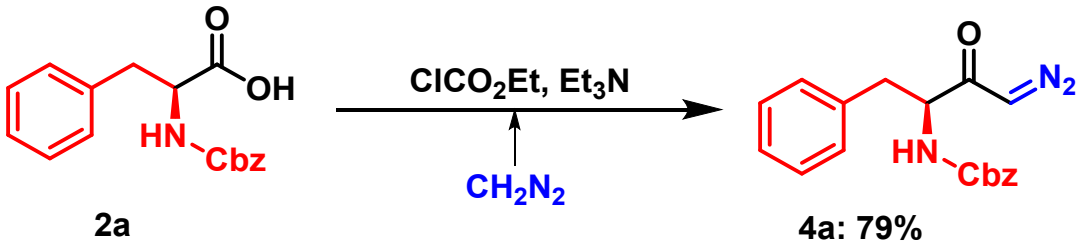
Table S1. Optimization of diazomethane synthesis in the continuous flow process.



Entry	Flow Rate (μL/min)		Retention time (min.)		Yield (%)
	NMU	KOH	Generation	Separation	
1	1000	1000	15	17	58
2	2500	2500	6	6.8	70
3	**5000**	**5000**	**3**	**3.4**	**86**
4	10000	10000	1.5	1.7	*

Reaction condition: NMU 0.162M in MeOH & diethyl ether in 1:2 ratios, KOH 30 wt% in water; diazomethane yields are based on benzoic acid titration. *Represent the over-pressure problem in KNF pump (max pressure 6 bar). A solution of NMU (N-Nitroso-N-methylurea) in MeOH : DEE with 1:2 ratio, (0.162 M) was taken in bottle connected with pump **A** and a solution of KOH in water (30 wt%) was taken in bottle connected with pump **B**. Both of reactants were introduced through **T-**mixer (T1) in a flow rate ratio of 1:1 (Table S1), and then passed through a PTFE tubing (id = 1000 μm, length = 38.25-meter, 34 mL) for the diazo-methane generation during **3** min of residence time and room temperature (Table S1, entry 3). After the successful completion, the aqueous and DEE was pumped as continuous flow droplets in house made pilot scale liquid-liquid separator. A residence time of 3.4 min, 1 bar pressure was found to be enough for the aqueous waste removal of the crude organic solution. The out flowed DEE reaction mixture was titrated with benzoic acid to get methyl benzoate and reveal the utility of 86% diazomethane (0.21 M in DEE, with productivity of 3.33 mL/min).

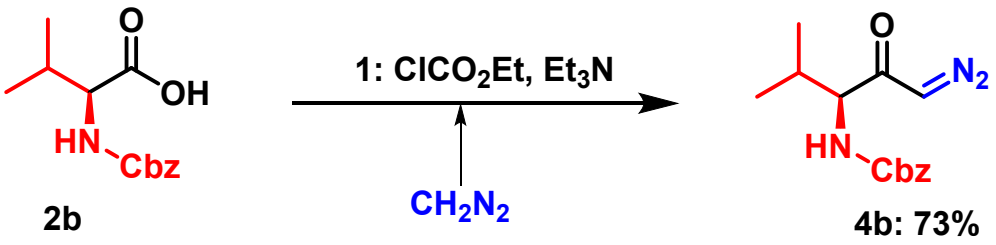
3.3. Optimization reaction platform for the benzyl (*S*)-(4-diazo-3-oxo-1-phenylbutan-2-yl) carbamate 4a synthesis.



2a **4a: 79%**

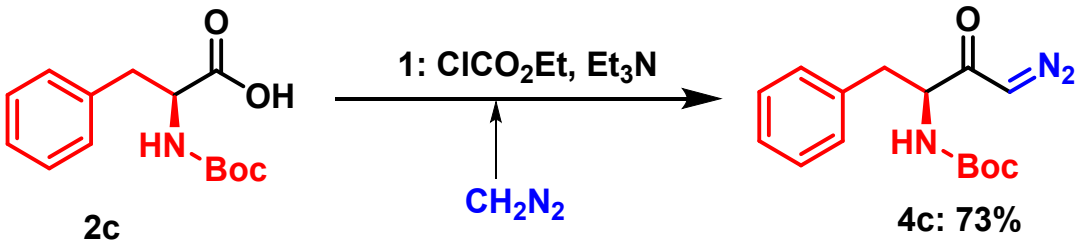
To an oven-dried 500 mL round bottom (RB) flask equipped with a Teflon coated magnetic stir bar, ((benzyloxy)carbonyl)-*L*-phenylalanine (17.4 g, 58.17 mmol) were added. Then dry THF (150 ml) was added using a syringe in argon atmosphere at -15 °C, and triethylamine (8.6 mL, 61.7 mmol) and ethyl chloroformate (5.9 mL, 61.7 mmol) were added dropwise. After stirring at this -15 °C temperature for 45 min, white precipitate was filtered, and the filtrate was transferred into a dried 1000 mL round bottom (RB) flask, the diazo-methane solution was added through the above designed diazo-generator for 50 min (1.35 equivalent). After diazo exposure for 50 min, the reaction mixture was further stirred for 6 h to complete the reaction. The reaction mixture was quenched and washed with $NaHCO_3$ (2 × 50 mL), then with brine (50 mL). The organic phase was dried over Na_2SO_4 and concentrated, the **4a** product was purified by flash chromatography, **R*f*** = 0.3 (30% Ethyl acetate / Hexane); yield **4a** (14.8 g, 79%) as a white off solid and **mp**: 85-87 °C**. ^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.37 –7.16 (m, 10H), 5.37 (d, *J* = 4.7 Hz, 1H), 5.20 (s, 1H), 5.08 (s, 2H), 4.48 (d, *J* = 5.4 Hz, 1H), 3.04 (d, *J* = 6.7 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 192.83, 155.83, 136.26, 136.12, 129.45, 128.82, 128.67, 128.35, 128.19, 127.23, 67.17, 58.97, 54.74, 38.62. **IR (ν_{max}):** 3316.90, 2953.83, 2108.67, 1712.33, 1526.57 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{18}H_{17}N_3ONaO_3$** $[M+Na]^+$ 346.1168, Found 346.1170. Analytical data are well matched with reported literature.[6]

Benzyl (*S*)-(1-diazo-4-methyl-2-oxopentan-3-yl) carbamate 4b.



Similar procedure was adapted as described in **3.2** using **2b** to provide **4b**. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography, **R*f*** = 0.5 (50% ethyl acetate / hexane), yield **4b** (11.7 g, 73%) as an off-white solid. and **mp**: 63-65 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.39 – 7.30 (m, 5H), 5.40 (s, 2H), 5.10 (s, 2H), 4.14 (s, 1H), 2.13 – 2.05 (m, 1H), 0.99 (d, *J* = 6.8 Hz, 3H), 0.89 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 193.32, 156.47, 136.34, 128.69, 128.35, 128.23, 67.22, 62.97, 54.86, 31.30, 19.54, 17.45. **IR (ν_{max}):** 3326.19, 2966.87, 2108.96, 1725.71, 1641.27, 1528.70 cm^{-1}. **HRMS** (ESI): *m/z* calcd for **$C_{14}H_{16}N_3NaO_3$** $[M+Na]^+$ 298.1168, Found 298.1161. Analytical data are well matched with reported literature.[7]

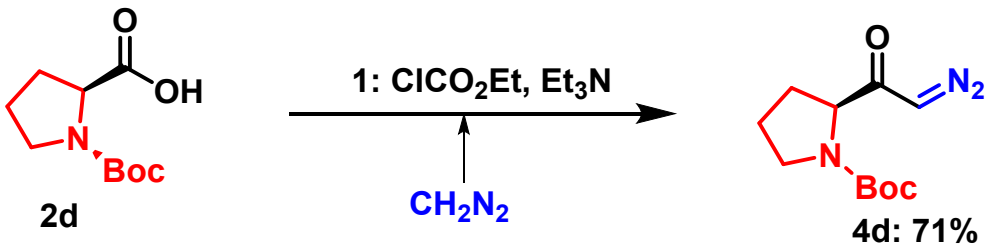
***tert*-Butyl (S)-(4-diazo-3-oxo-1-phenylbutan-2-yl) carbamate 4c.**



Similar procedure was adapted as described in **3.2** using **2c** to provide **4c**. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography, **R*f*** = 0.5 (20% ethyl acetate / hexane); yield of **4c** (12.3 g, 73%) as an off-white solid and **mp**: 88-90 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.32 – 7.18 (m, 5H), 5.22 (s, 1H), 5.11 (d, *J* = 5.2 Hz, 1H), 4.42 (d, *J* = 5.2 Hz, 1H), 3.03 (d, *J* = 6.9 Hz, 2H), 1.41 (s, 9H). **^{13}C NMR (125 MHz, $CDCl_3$)** δ

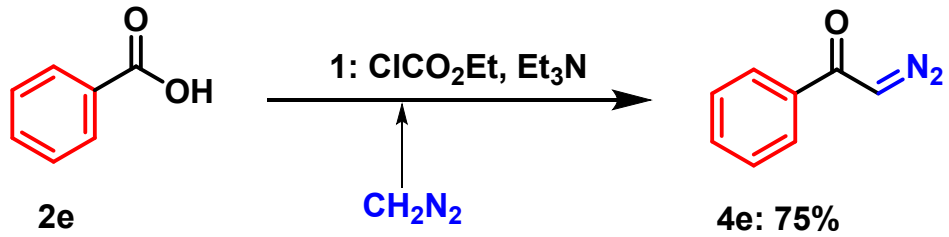
193.45, 155.27, 136.44, 129.47, 128.75, 127.10, 80.22, 58.63, 54.55, 38.66, 28.41. **IR (ν_{max}):** 3441.07, 2979.30, 2111.10, 1714.75, 1513.88 cm^{-1}. **HRMS (ESI):** *m/z* calcd for $C_{15}H_{20}N_3O_3$ $[M+H]^+$ 290.1499, Found 290.1503. Analytical data are well matched with reported literature.[8]

***tert*-Butyl (*S*)-2-(2-diazoacetyl) pyrrolidine-1-carboxylate 4d.**



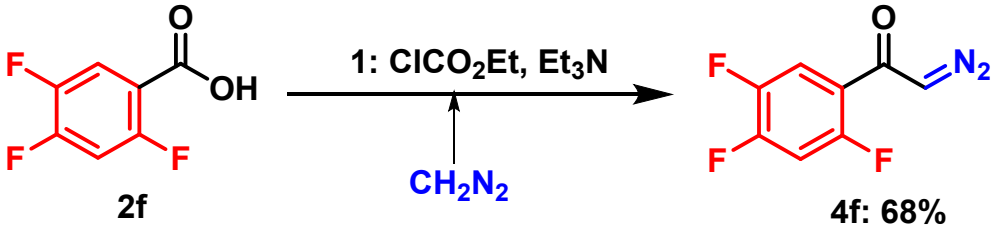
Similar procedure was adapted as described in **3.2** using **2d** to provide **4d.** The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography, **R*f*** = 0.5 (20% ethyl acetate / hexane); yield of **4d** (9.9 g, 71%). as a pale-yellow viscous liquid. **^{1}H NMR (400 MHz, $CDCl_3$ mixture of rotamers)** δ 5.45 (d, *J* = 35.7 Hz, 1H), 4.29 – 4.20 (m, 1H), 3.82 – 3.37 (m, 2H), 2.25 – 1.99 (m, 2H), 1.91 – 1.85 (m, 2H), 1.46 (d, *J* = 14.4 Hz, 9H). **^{13}C NMR (101 MHz, $CDCl_3$ mixture of rotamers)** δ 196.08, 195.14, 154.81, 154.13, 80.45, 80.14, 64.46, 63.58, 53.13, 52.02, 47.09, 46.77, 31.25, 29.68, 28.36, 24.39, 23.69. **IR (ν_{max}):** 2980.64, 2108.08, 1699.24, 1451.67 cm^{-1}. **HRMS (ESI):** *m/z* calcd for $C_{11}H_{18}N_3O_3$ $[M+H]^+$ 240.1342, Found 240.1339. Analytical data are well matched with reported literature.[9]

2-Diazo-1-phenylethan-1-one 4e.



Similar procedure was adapted as described in **3.2** using **2e** to provide 4e. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography, **R*f*** = 0.5 (10% ethyl acetate / hexane); yield of **4e** (6.4 g, 75%) as an orange oil. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.77 – 7.75 (m, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.5 Hz, 2H), 5.90 (s, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 186.47, 136.82, 132.85, 128.80, 126.84, 54.29. **IR (ν_{max}):** 2109.69, 1619.77 cm^{-1}. Analytical data are well matched with reported literature.[6]

2-Diazo-1-(2,4,5-trifluorophenyl) ethan-1-one 4f.



Similar procedure was adapted as described in **3.2** using **2f** to provide **4f**. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography, **R*f*** = 0.4 (20% ethyl acetate / hexane); yield of **4f** (7.9 g, 68%) as a viscous liquid., **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.89 – 7.82 (m, 1H), 7.02 – 6.96 (m, 1H), 6.08 (s, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 179.73, 158.12, 158.03, 155.61, 155.53, 154.14, 154.01, 153.87, 151.57, 151.43, 151.29, 148.68, 148.65, 148.55, 148.52, 146.22, 146.19, 146.10, 146.07, 121.09, 120.94, 118.50, 118.31, 106.77, 106.56, 106.47, 106.26, 58.79, 58.60. **IR (ν_{max}):** 3070.88, 2114.26, 1621.68, 1595.51, 1370.06 cm^{-1}. **^{19}F NMR (377 MHz, $CDCl_3$)** δ -112.57 (dd, *J* = 16.2, 7.8 Hz), -125.70 (dd, *J* = 21.6, 7.6 Hz), -140.66 (dd, *J* = 21.6, 16.4 Hz).

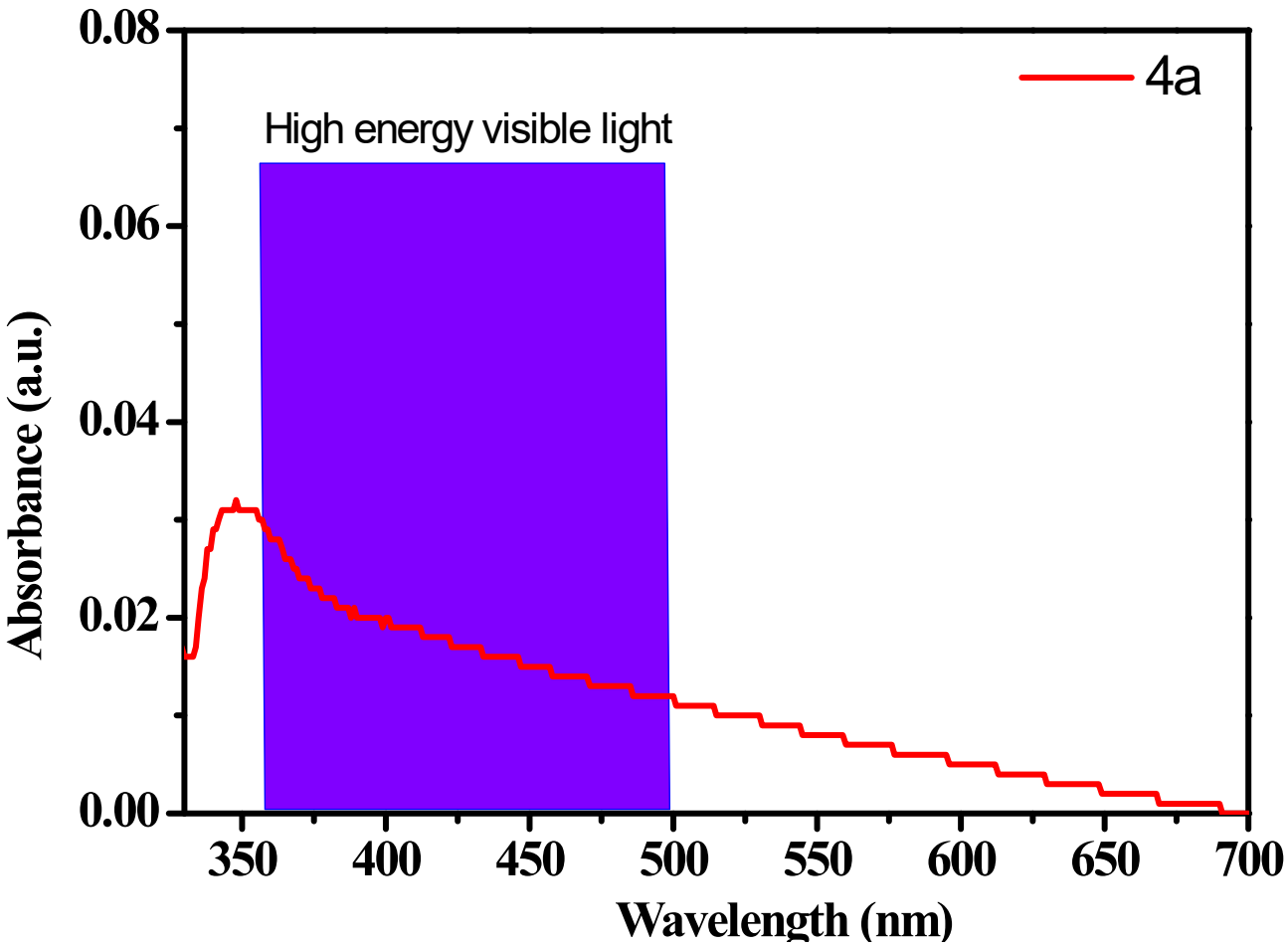


Figure S3. UV-Visible absorption spectroscopy of the synthesized **4a**.

4. Homologation reaction under controlled laboratory condition: 4a (50 mg; mol ratio 1), ethanol (6.25 mL; mol ratio 734), and varied solvent (mol ratio 446), were added to 50 mL round bottom flask. Then the RB was sealed by a septum and air bubble was exchanged with nitrogen gas and was then placed in an LED light source (Figure S4). The reaction mixture was allowed to various light irradiation for 24 h. The reaction mixture was concentrated under vacuum and further the product was purified using flash column chromatography on 100-200 mesh silica gel with hexane/ethyl acetate as eluent.

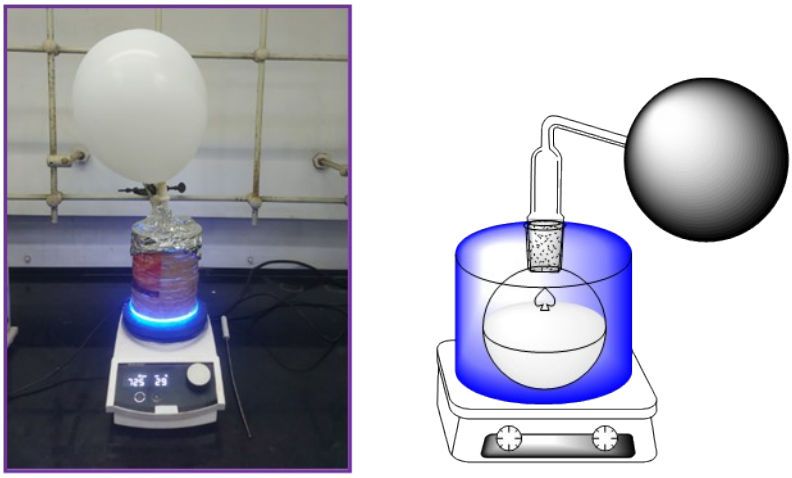
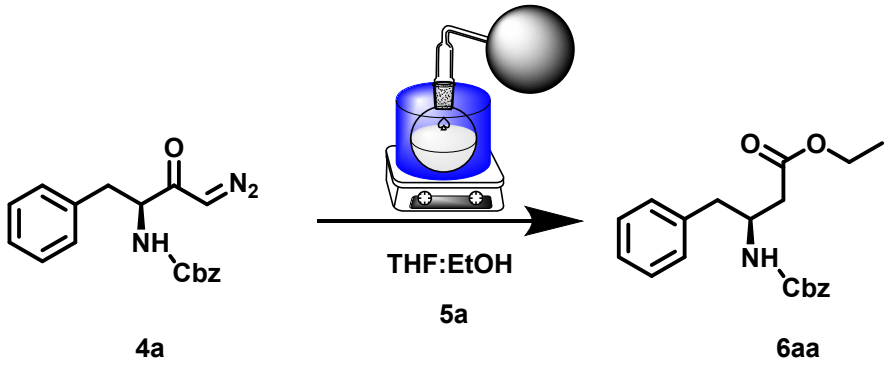


Figure S4. Batch process background and photo-light reaction.

Table S2. Solvent screen for the controlled-homologation photochemical reaction.



4a 6aa

Entry	Solvent	LED light source	Yield%
1[a]	THF	Red	NA
2[b]	THF	Green	NA
3[c]	THF	Blue	30
4[d]	THF	Blue	2
5[c]	Acetonitrile	Blue	15
6[c]	Toluene	Blue	20
7[c]	Hexane	Blue	NA
8[c]	Ethyl acetate	Blue	20
9[c]	DMF	Blue	25
10[c]	DCM	Blue	23
11[c]	Chloroform	Blue	20
12[c]	DMSO	Blue	10
13[c]	1,4-Dioxane	Blue	20
14[c]	Acetone	Blue	27

Reaction conditions: stock solution containing **4a**: **5a**: THF with molar ratio of 1:734:446 (50 mg **4a**: 6.25 mL **5a**: 5.65 mL THF); Reaction time 24 h, reaction temperature 25±5 °C. a) Red LED light intensity 7831 lux; b) green LED light intensity 13822 (lux); c) blue LED light intensity 15695 lux; yields are based on isolated yields and light intensity error ±400 lux are acceptable.

4.1. Controlled continuous flow homologation reaction condition.

A stock solution containing **4a**: **5a**: **THF** with molar ratio of 1:734:446 was taken in 20 mL BD syringe connected with syringe pump (Table S2). The solution phase was passed through a perfluoroalkoxy (PFA) coil reactor $^{10\ \text{volume}\ =\ 2.0\ \text{mL}}$. The tubing reactor was wrapped over the polystyrene sheet and to avoid the light exposure to out-side of photo-reactor was fully covered with aluminium foil. The flat reactor was irradiated by 30W LED was placed above to reactor. The product mixture was collected at the end of the photo-flow reactor into a flask. As mentioned in Table 1, various reaction parameters (retention time, temperature, LED) were regulated to optimize reaction performance.

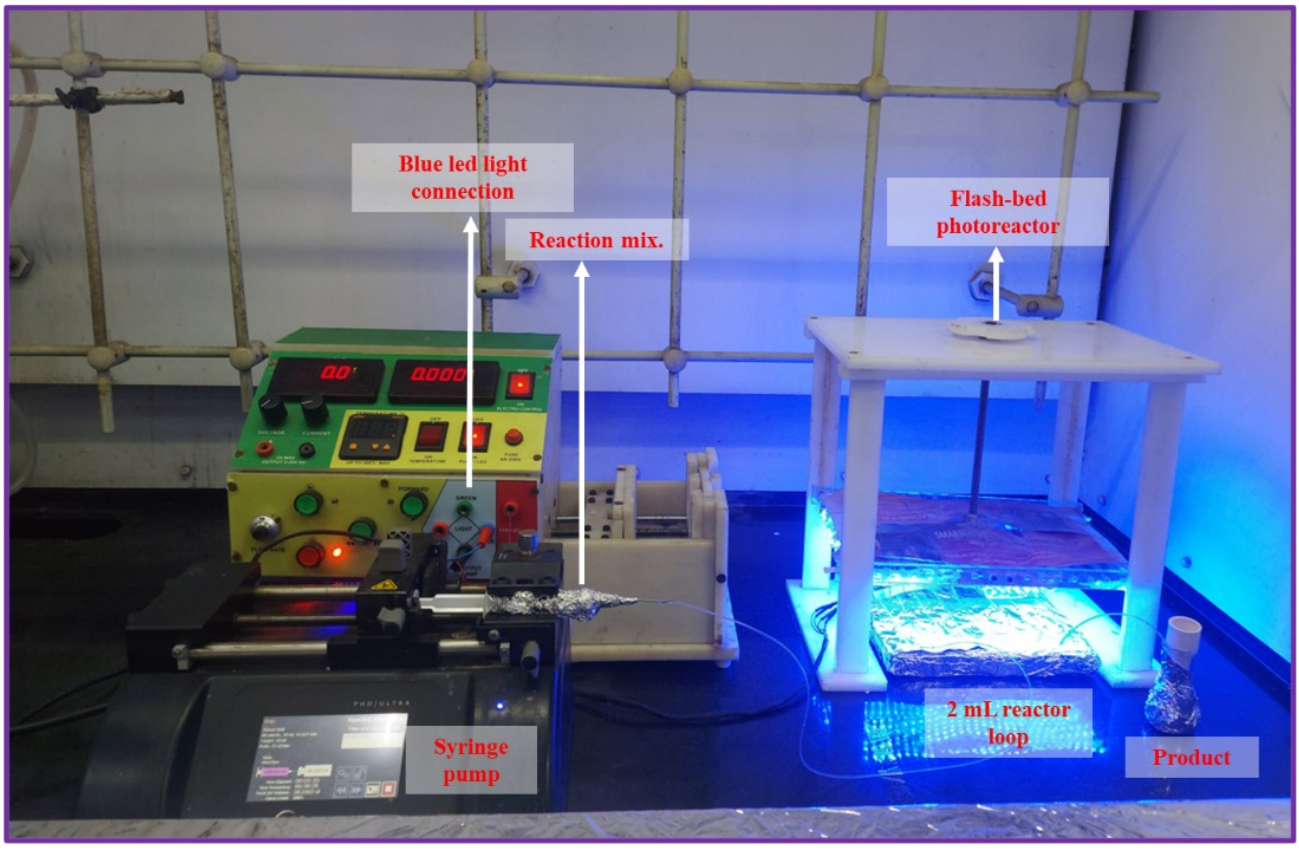


Figure S5. Schematic diagram of controlled continuous flow laboratory experiment.

4.2. Real-time reaction control for direct homologation of the diazo compound under fluctuating irradiance (cloudy or rainy days).

Optimization of solar panel angular position: To measure the light intensity, we were used the lux light meter app in to the Samsung A50 mobile phone, and further cross-verified with Samsung A71 mobile phone (Figure S6a). By using the acrylic sheet and laser cutter, we have fabricated the mobile stand with 10-degree displacement as shown in Figure S6b, and place the mobile for the light intensity measurement. Recorded light intensity has shown in Table S3 and Figure 1a. To compare the light intensity in zero degree (pond condition) and 50-degree angular position shown in Figure 1b.

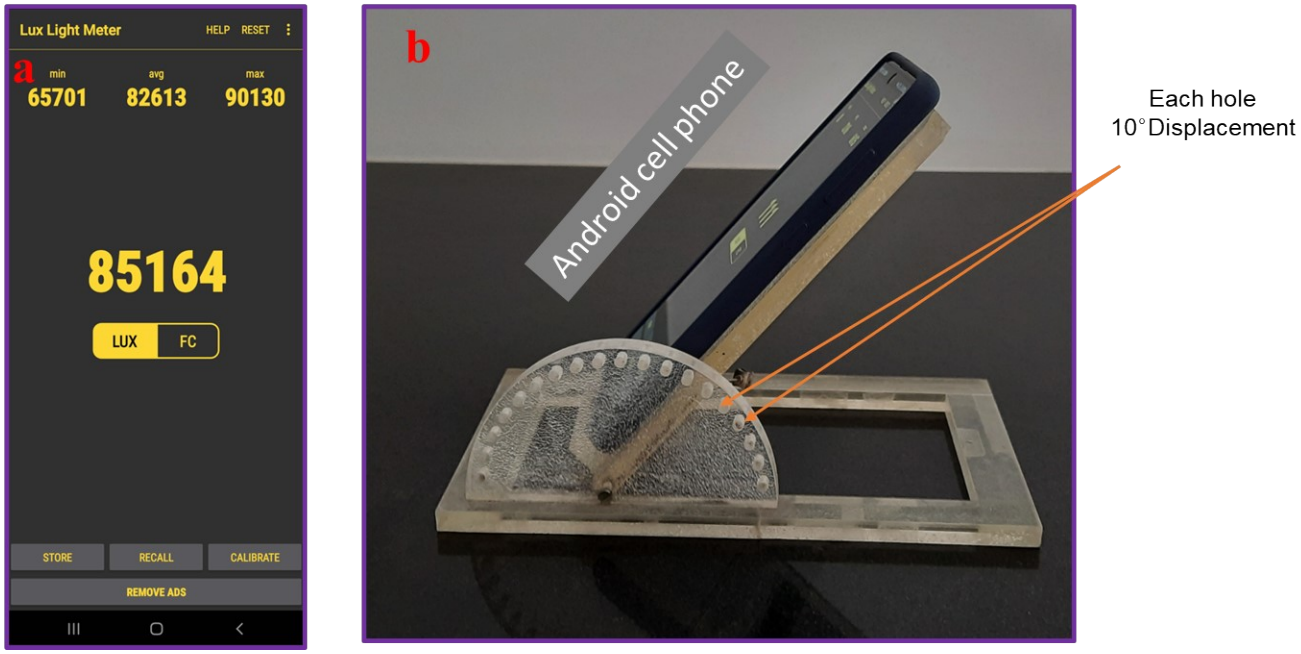


Figure S6. Schematic diagram of (a) Lux light meter app; (b) radial mobile stand.

Table S3. Optimization data of light intensity in lux.

Time	0°	10°	20°	30°	40°	50°	60°	70°	80°	90°
9:00	52700	70000	83000	235000	255000	268000	27000	255000	233000	35000
10:00	76000	92000	260000	291000	304400	313000	310000	300000	274000	23000
11:00	260000	275000	290000	288000	290000	276000	256000	228000	80000	42000
12:00	274000	309000	320000	330000	322000	323000	290000	256000	189000	35000
13:00	268000	281000	300000	312000	316000	316000	279000	250000	188000	36000
14:00	200000	245000	248000	245000	93000	90000	81000	65000	40000	35000
15:00	59000	77000	88000	100000	270000	280000	290000	283000	267000	98000
16:00	25000	40000	52000	67000	79000	91000	90000	94000	92000	88000
17:00	7800	11900	16900	20000	24000	26000	28900	29000	29000	25000
± 500 lux error are acceptable.										

Solar panel flow reactor fabrication: The solar panel reactor body was made with the acrylic sheet. At first all the component as shown in (Figure S7 a) were designed and fabricated through the laser cutter. After fabrication, all the parts were connected to each other by using the screw (Figure S7 b). The stepper motor 18 Kg-cm was bought from the local market and installed to panel for the rotation. To set the solar panel flow reactor angle, the manual rotor has been built. To fabricate the solar panel reactor, we have used foam sheet, Aluminium foil, PFA tubing, and cello-tape. Our polystyrene foam (length 50 cm X width 50 cm X height 6 cm) was covered by aluminium foil (0.016 mm) for reflecting of sun light (Figure S7 c). Manually, PFA tubing mounted over the aluminium covered surface (Figure S7 d). Finally, blue polyethylene film filter covered the solar panel reactor to make the equivalent to the blue LED light source (Figure S7 e), 33% light intensity reduction has been observed after the covering with blue film.

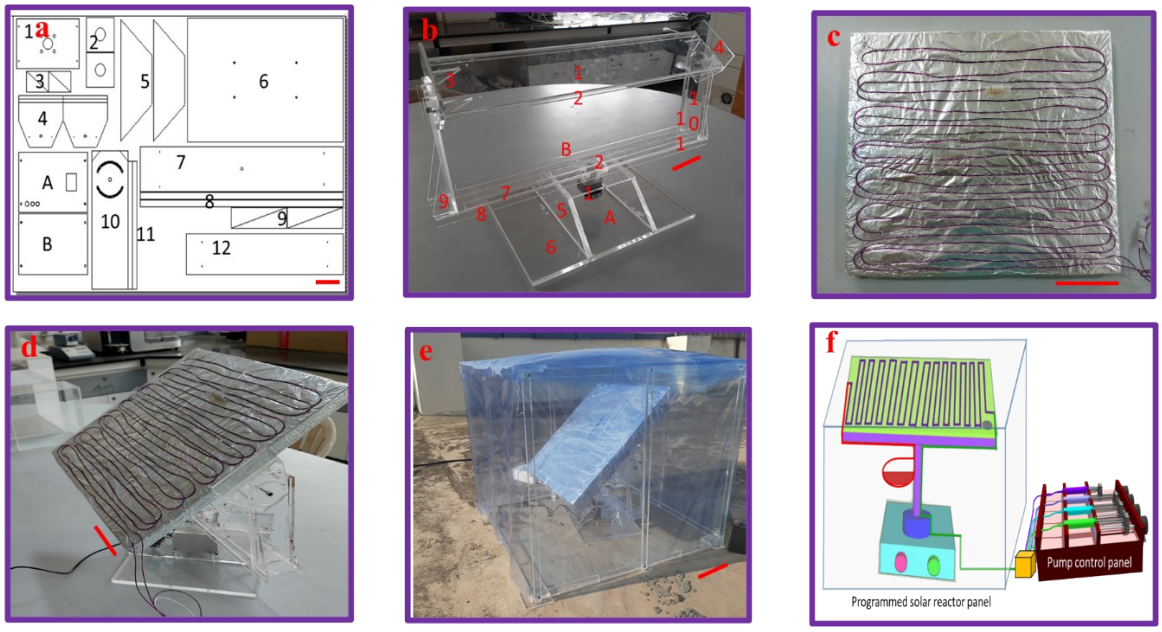


Figure S7. (a) Laser cutting design of solar panel; (b) acrylic body chassis of solar panel; (c) PFA tubing solar reactor; (d) pasting of solar reactor on acrylic body chassis of solar panel; (e) Solar panel under blue light film; (f) schematic diagram of photo reaction set-up, bar represent the 10 cm.

5. Electronic circuit design to integrate the light sensitive syringe pump.

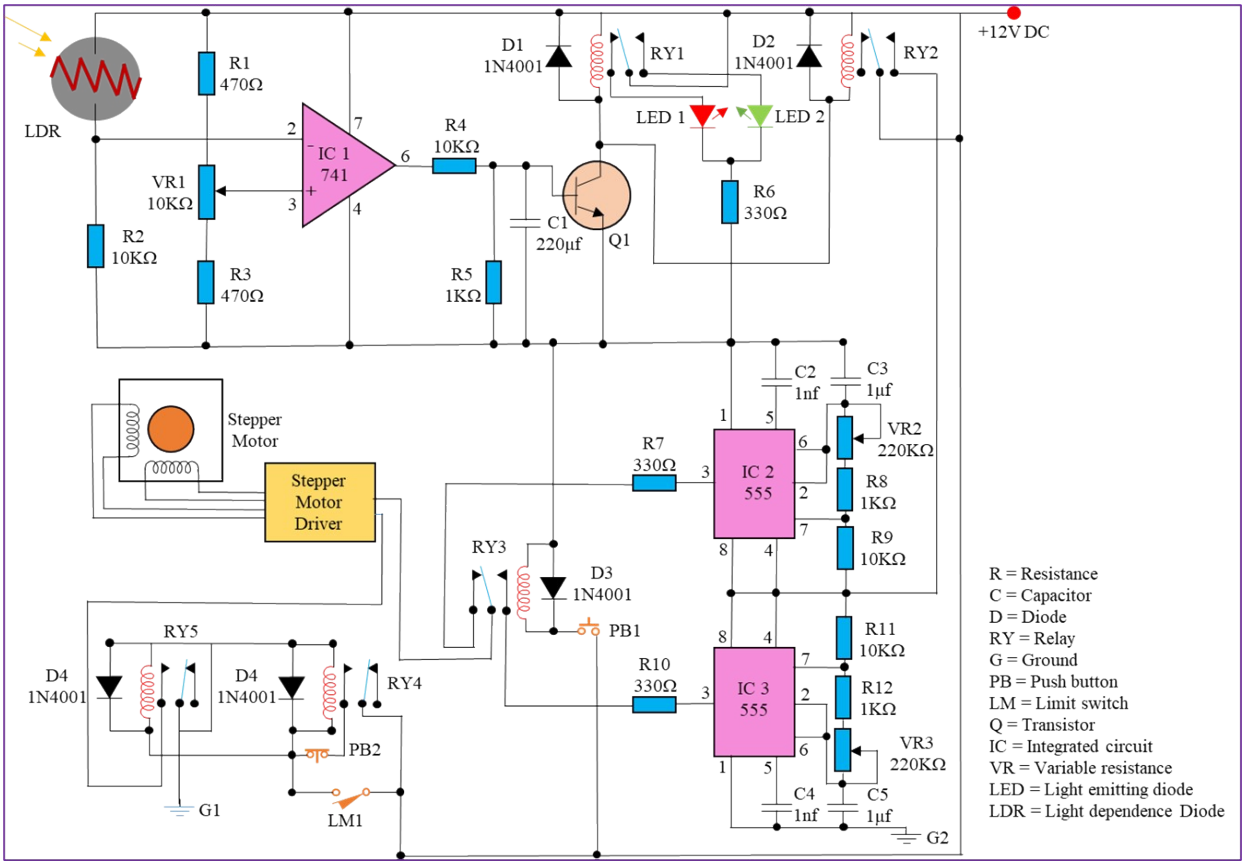


Figure S8. Electronic circuit of light-dependent resistor (LDR) based syringe pump.

The electronic parts were procured from the local market and arranged as per above design. This system is based on the principle of photo electric effect containing one LDR (light emitting diode), which is connected to 741-OP **IC1** to pin number **2** which is a non-inverting mode. When the incident light on LDR is zero, no positive signal is received from 741-OP **IC1** pin number **6**, but once light is interacting with LDR the input current is generated at 741-OP **IC1** pin number **2**, thus bringing 741-OP **IC1** to operational mode. This 741-OP **IC1** starts emitting the output signal at pin no **6**, yielding approx. 50-80 mA current. This output current is further reduced to approx. 15-20 mA by using 10KΩ resistance (**R4**) which was sequentially connected to NPN transistor (**Q1**) base pin. The transistor (**Q1**) starts working in forward biasing mode and turns on the relay (**RY1**) and relay (**RY2**). Relay (**RY1**) monitors the pump running status, which is

indicated through the LED indicators (red = off & green = on). Relay (**RY2**) is used for turning on the square wave generator (555-IC) **IC2** and (555-IC) **IC3** resulting in a positive current. The output frequency of (555-IC) **IC2** is adjusted by variable resistance (**VR2**). The output frequency signal at pin number **3** was passing through a 330Ω resistance (**R7**) which is connected to NC (normally closed) relay (**RY3**). Once relay (**RY3**) sends the current to stepper motor driver, syringe pump turns on. Due to this motor rotation, lead screw rotates causing the injection piston to introduce the reaction mixture in the photo reactor tubing. To complete the reaction, certain light intensity was needed and in this regard variable resistance (VR1) is connected through LDR circuit to control the pump movement under the fluctuating light. If, during the process, due to weather conditions, sunlight exposure is limited, and the optimised LUX value is not achieved, (741-IC) **IC1** instantaneously stops giving output signal to pin number 6, and thus the injection pump stops.

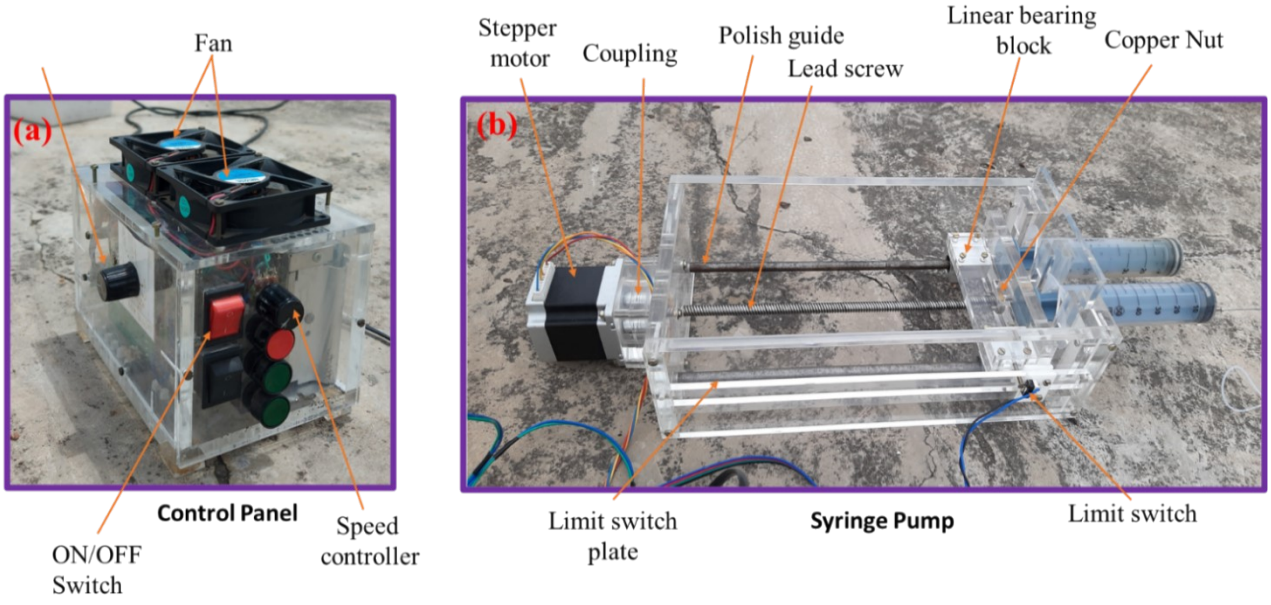


Figure S9. light-dependent resistor (LDR) controlled system; (a) control panel; (b) syringe pump.

The syringe in the pump can be inserted or removed by pressing push button (**PB1**) to adjust the lead screw plate. The push button (**PB1**) turns on the relay (**RY3**) due to which (555-IC) **IC3** generates high frequency signal at terminal **3**. This terminal being connected to step (clock) pin of stepper motor driver rotates it in a high speed. The forward and backward rotation of the stepper motor is controlled by the limit switch (**LM1**), which turns on the relay (**RY4**). NO (normally open) terminal of relay (**RY4)** is connected to push button (**PB2**) which usually remains in NC (Normally closed) mode. This NC mode keeps the relay (**RY5**) turned on. The common terminal of relay (**RY5**) is connected to the ground (**G1**) so ground negative supply is passing to NO (normally open) terminal of **RY5**. The NO terminal is connected to stepper motor driver direction pin due to which the stepper motor starts rotating in reverse direction. The push button (**PB2**) can be used to resume the rotation of stepper motor in forward direction. This forward rotation takes place because the (**PB2**) button breaks the continuity of Relay (**RY4**) by switching the relay (**RY5**) off.

5.1. Electronic circuit design for solar tracker control.

Table S4. The connection of A4899 driver to a stepper motor and the Arduino.

A4988 Stepper Driver	Connection
VMOT	+12 V DC
GND	Motor Ground
SLP	RESET
RST	SLP
VDD	5 Volt
GND	Logic ground
STP	Pin 3
DIR	Pin 2
1A, 1B, 2A, 2B	Stepper Motor

1. The motor power supply is connected to GND and VMOT (top right).
2. The two coils of the stepper motor are connected to 1A, 1B and 2A, 2B (see below).
3. The GND pin (lower right) is connected to the ground pin of the microcontroller and VDD is connected to 5V.
4. The STP (step) and DIR (direction) pin are connected to digital pin 3 and 2 respectively.
5. The SLP pin is an active low input. Meaning, pulling this pin low puts the driver in sleep mode, minimizing the power consumption. RST is also an active low input. When pulled low,
6. The EN (enable) pin can be left disconnected, it is pulled low by default. When this pin is set high the driver is disabled.

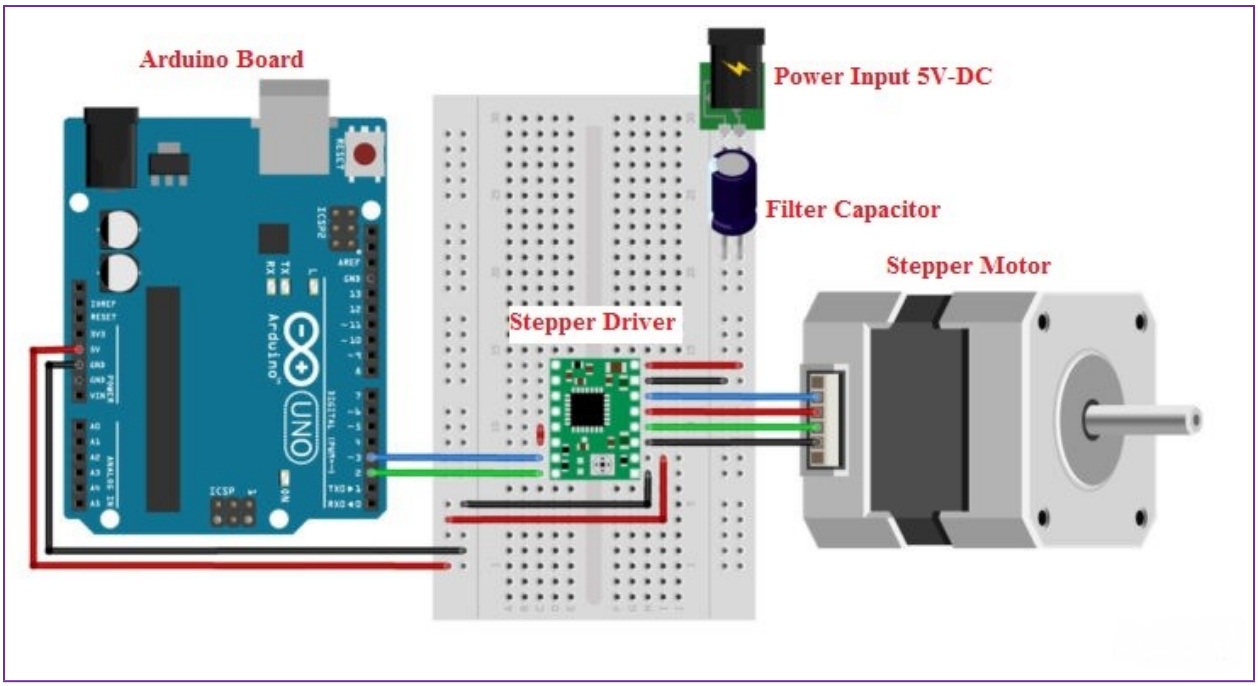


Figure S10. Electrical scheme of the circuit for the solar panel movement control system.

5.2. Integrated solar panel reactor design.

Solar panel Arduino code. The following source file has been made for the solar panel motor movement.

```
// Drive Stepper motor using A4988 stepper motor driver
// for more info visit iknowvations.in

// first define the pins
const int DirPin = 4;    // this pin defines direction CW or CCW
const int StepPin = 5;   // pulse this pin to move one step
const int SPR = 100;     // Steps per revolution

void setup()
{
  // Make pins as Outputs
  pinMode(StepPin, OUTPUT);
  pinMode(DirPin, OUTPUT);
}
void loop()
{
  // 1 let us rotate shaft clockwise
  digitalWrite(DirPin, HIGH);   // defines the direction to clockwise

  // Pulse the step pin
  for(int x = 0; x < SPR/100; x++)
  {
    digitalWrite(StepPin, HIGH);
    delayMicroseconds(1000);
    digitalWrite(StepPin, LOW);
    delayMicroseconds(1000);
 }
  delay(432000); // Short delay of one second
 // 2 let us rotate shaft clockwise
  digitalWrite(DirPin, HIGH);   // defines the direction to clockwise

  // Pulse the step pin
  for(int x = 0; x < SPR/100; x++)
  {
    digitalWrite(StepPin, HIGH);
    delayMicroseconds(1000);
    digitalWrite(StepPin, LOW);
    delayMicroseconds(1000);
 }
  delay(432000); // Short delay of one second
```

```
  // 3 let us rotate shaft clockwise
 digitalWrite(DirPin, HIGH);   // defines the direction to clockwise

 // Pulse the step pin
 for(int x = 0; x < SPR/100; x++)
 {
   digitalWrite(StepPin, HIGH);
   delayMicroseconds(1000);
   digitalWrite(StepPin, LOW);
   delayMicroseconds(1000);
}
 delay(432000); // Short delay of one second

  // 4 let us rotate shaft clockwise
 digitalWrite(DirPin, HIGH);   // defines the direction to clockwise

 // Pulse the step pin
 for(int x = 0; x < SPR/100; x++)
 {
   digitalWrite(StepPin, HIGH);
   delayMicroseconds(1000);
   digitalWrite(StepPin, LOW);
   delayMicroseconds(1000);
}
 delay(432000); // Short delay of one second

  // 5 let us rotate shaft clockwise
 digitalWrite(DirPin, HIGH);   // defines the direction to clockwise

 // Pulse the step pin
 for(int x = 0; x < SPR/100; x++)
 {
   digitalWrite(StepPin, HIGH);
   delayMicroseconds(1000);
   digitalWrite(StepPin, LOW);
   delayMicroseconds(1000);
}
 delay(432000); // Short delay of one second
```

```
  // 6 let us rotate shaft clockwise
 digitalWrite(DirPin, HIGH);   // defines the direction to clockwise

 // Pulse the step pin
 for(int x = 0; x < SPR/100; x++)
 {
   digitalWrite(StepPin, HIGH);
   delayMicroseconds(1000);
   digitalWrite(StepPin, LOW);
   delayMicroseconds(1000);
}
 delay(432000); // Short delay of one second

  // 7 let us rotate shaft clockwise
 digitalWrite(DirPin, HIGH);   // defines the direction to clockwise

 // Pulse the step pin
 for(int x = 0; x < SPR/100; x++)
 {
   digitalWrite(StepPin, HIGH);
   delayMicroseconds(1000);
   digitalWrite(StepPin, LOW);
   delayMicroseconds(1000);
}
 delay(432000); // Short delay of one second

  // 8 let us rotate shaft clockwise
 digitalWrite(DirPin, HIGH);   // defines the direction to clockwise

 // Pulse the step pin
 for(int x = 0; x < SPR/100; x++)
 {
   digitalWrite(StepPin, HIGH);
   delayMicroseconds(1000);
   digitalWrite(StepPin, LOW);
   delayMicroseconds(1000);
}
 delay(432000); // Short delay of one second
```

```
  // 9 let us rotate shaft clockwise
 digitalWrite(DirPin, HIGH);   // defines the direction to clockwise

 // Pulse the step pin
 for(int x = 0; x < SPR/100; x++)
 {
   digitalWrite(StepPin, HIGH);
   delayMicroseconds(1000);
   digitalWrite(StepPin, LOW);
   delayMicroseconds(1000);
}
 delay(432000); // Short delay of one second

  // 10 let us rotate shaft clockwise
 digitalWrite(DirPin, HIGH);   // defines the direction to clockwise

 // Pulse the step pin
 for(int x = 0; x < SPR/100; x++)
 {
   digitalWrite(StepPin, HIGH);
   delayMicroseconds(1000);
   digitalWrite(StepPin, LOW);
   delayMicroseconds(1000);
}
 delay(432000); // Short delay of one second

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digitalWrite(DirPin, HIGH);   // defines the direction to clockwise

// Pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
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}
delay(432000); // Short delay of one second
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 // 9 let us rotate shaft clockwise
digitalWrite(DirPin, HIGH);   // defines the direction to clockwise

// Pulse the step pin
for(int x = 0; x < SPR/100; x++)
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digitalWrite(DirPin, HIGH);   // defines the direction to clockwise

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  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

    // 1 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second
```

```
// 2 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 3 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 4 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second
```

```
// 5 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 6 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 7 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second
```

```
//  8 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 9 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 10 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second
```

```
    // 1 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 2 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 3 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second
```

```
// 4 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 5 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 6 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second
```

```
// 7 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

//  8 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 9 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second
```

```
// 10 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

    // 1 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 2 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second
```

```
// 3 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 4 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 5 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second
```

```
// 6 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 7 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

//  8 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second
```

```
// 9 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 10 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

    // 1 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second
```

```
// 2 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 3 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 4 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second
```

```
// 5 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 6 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 7 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second
```

```
//  8 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 9 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second

// 10 let us rotate shaft counterclockwise
digitalWrite(DirPin, LOW);

// Again pulse the step pin
for(int x = 0; x < SPR/100; x++)
{
  digitalWrite(StepPin, HIGH);
  delayMicroseconds(1000);
  digitalWrite(StepPin, LOW);
  delayMicroseconds(1000);
}
delay(432000); // Short delay of one second
```

Work Function: There are three parts in solar panel tracker, wherein one part is a light-dependent LDR sensor-operated syringe pump, the second part is the control panel, and the third part is the solar panel. These three parts are interconnected to each other. Stepper motor wire and limit switch connections are made from the control panel to the syringe pump. Also, connections are made for light-dependent sensors from the control panel. This is fixed in the exposed area of the solar panel. Syringe pump will be in working mode when sunlight exceeds set LUX and when the sunlight intensity is below the set LUX then the syringe pump will be in off mode. The direction of the solar panel starting point is set in the morning of sunrise. After that solar panel rotates by 7.2 min./step [per step = 1.8 degree], and after 12 hours it turns 180 degrees, due to

which the reactor panel is exposed to the maximum of sunlight all the day time. After that in the night, the solar panel would reverse rotate and return to the set starting point.

5.2. Optimization of the reaction condition under the fluctuating solar light. A stock solution containing **4a**: **5a**: THF with molar ratio of 1:734:446 was taken in 20 mL BD syringe connected with syringe pump (Table 2). The solution phase was passed through a perfluoroalkoxy (PFA) flat reactor {Outer dimeter (OD 1/16 inches), Inner dimeter (ID 1.0 mm), length 10.2-meter, volume = 8.0 mL}. The solar panel reactor was exposed by sun-light to proceed the reaction. The product mixture was collected at the end of the photo-flow reactor into a flask. As mentioned in Table 1, various reaction parameters (retention time) were regulated to optimize reaction performance.

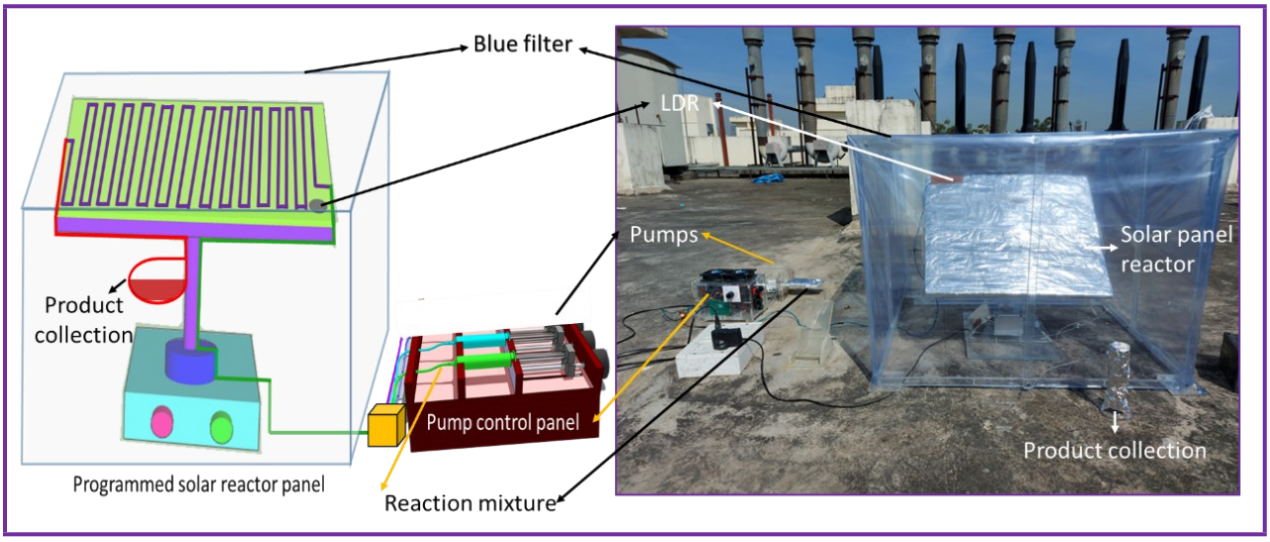
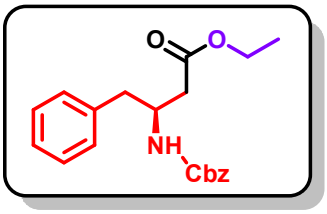


Figure S11. Model and actual photograph of the solar panel tracker reactor for the homologation reaction under the fluctuating light condition.

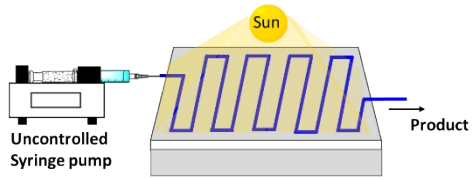
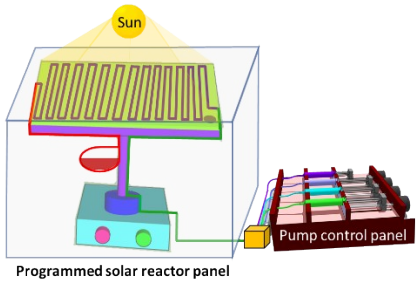
Optimization of the reaction condition under the fluctuating solar light.

Ethyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (6aa):

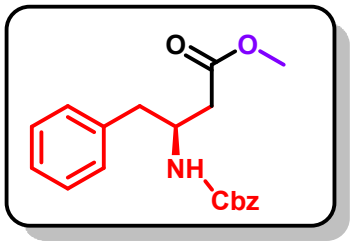


A stock solution containing **4a**: **5a**: THF with molar ratio of 1:734:446 was taken in syringe connected with syringe pump and covered with aluminium foil. The mixture of solutions was introduced to a solar tracker reactor with 100 µL/min. flow rate and then passed through a PFA tubing (id = 1000 µm, l = 10.2 m, vol. = 8 mL); light cut-off 50000 lux; to yield **6aa** product. The product mixture was collected under the stable condition and concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.25 (15% ethyl acetate/hexane); 76% yield of **6aa** as a white solid. **mp:** 64-66 °C; $[\alpha]_D^{25}$ = -12.0 (c 1.00, $CHCl_3$), $[\alpha]_D^{20}$ = -14.0 (c 0.92, $CHCl_3$).[10] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.38 – 7.16 (m, 10H), 5.33 (d, *J* = 8.0 Hz, 1H), 5.07 (s, 2H), 4.26 – 4.19 (m, 1H), 4.16 – 4.07 (m, 2H), 2.98 –2.82 (m, 2H), 2.55 – 2.43 (m, 2H), 1.25 (t, *J* = 7.16 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 171.66, 155.73, 137.63, 136.67, 129.49, 128.70, 128.62, 128.20, 128.14, 126.81, 66.72, 60.79, 49.49, 40.38, 37.67, 14.32. **IR (ν_{max}):** 3367.15, 3033.99, 2973.04, 1728.39, 1528.53, 1451.61 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{20}H_{24}NO_4$** $[M+H]^+$ 342.1705, Found 342.1707. Verified the analytical data with those reported in the literature.[10]

Comparison between light availability in static and solar tracker under the fluctuating (Sun) light condition.

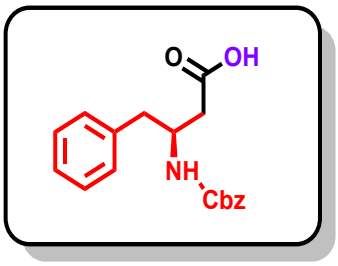
Entry	Static Condition	Sun-flower equivalent reactor condition
		
1	Ordinary pump	Smart LDR controlled pump (light cut-off = 50000 lux)
2	Panel rotation = no	Panel rotation = Programmed 100 pulse clockwise, each pulse = 1.8 degree and 4.2 min delay.
3	Panel angular angle = 180 degree	Panel angular angle = 50 degree.
4	3-day (9 am - 5 pm) outdoor experiment results in yield 38% (57 mg).	3-day outdoor experiment results in yield 76% (114 mg).

Methyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (6ab):



The title compound **(6ab)** was prepared according to the general procedure as described in section **(6aa)** using **4a** and methanol. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.35 (20% ethyl acetate / hexane); 80% yield of **6ab** as a white solid and **mp:** 51-53 °C; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.37 – 7.15 (m, 10H), 5.34 (d, *J* = 8.4 Hz, 1H), 5.06 (s, 2H), 4.23 (d, *J* = 6.6 Hz, 1H), 3.66 (s, 3H), 2.97 – 2.80 (m, 2H), 2.56 – 2.44 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 170.93, 154.58, 136.42, 135.49, 128.30, 127.54, 127.46, 127.04, 126.98, 125.67, 65.56, 50.70, 48.31, 39.19, 36.32. **IR (ν_{max}):** 3322.21, 3031.33, 2953.77, 1713.23, 1525.50, 1444.86 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{19}H_{21}NNaO_4$** $[M+Na]^+$ 350.1368, Found 350.1370. Verified the analytical data with those reported in the literature.[7]

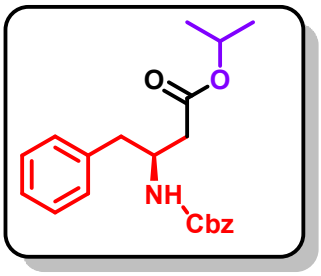
(*S*)-3-(((Benzyloxy)carbonyl) amino)-4-phenylbutanoic acid (6ac):



The title compound of **(6ac)** was prepared according to the general procedure as described in section **(6aa)** using **4a** and water. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.35 (5% methanol/ chloroform); 78% yield of **6ac** as a white solid and **mp:** 105-107 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.36 – 7.14 (m, 10H), 5.32 – 5.22 (m, 1H), 5.07 (d, *J* = 4.0 Hz, 2H), 4.26 – 4.20 (m, 1H), 2.98 – 2.84 (m, 2H), 2.61– 2.50 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 176.66, 155.88, 137.44, 136.49, 129.48, 128.77, 128.67, 128.29, 128.20, 126.92, 66.91, 49.31, 40.28, 37.37. **IR (ν_{max}):** 3315.40, 3031.14, 2926.18, 1707.67, 1519.94, 1450.52 cm^{-1}. **HRMS (ESI):** *m/z* calcd for

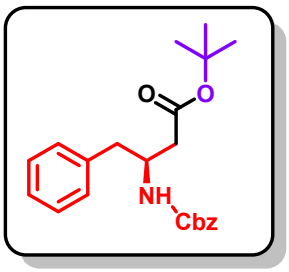
$C_{18}H_{20}NO_4$ $[M+H]^+$ 314.1392, found 314.1399. Verified the analytical data with those reported in the literature.[10]

Isopropyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (6ad):



The title compound of **(6ad)** was prepared according to the general procedure as described in section **(6aa)** using **4a** and isopropanol. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.4 (15% ethyl acetate / hexane); 73% yield of **6ad** as a white solid and **mp:** 55-57 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.37 – 7.16 (m, 10H), 5.33 (d, *J* = 8.3 Hz, 1H), 5.07 (s, 2H), 5.05 – 4.99 (m, 1H), 4.25 – 4.20 (m, 1H), 2.97 – 2.81 (m, 2H), 2.51 – 2.38 (m, 2H), 1.25 – 1.21 (m, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 171.18, 155.71, 137.65, 136.69, 129.50, 128.68, 128.61, 128.18, 128.13, 126.79, 68.28, 66.69, 49.55, 40.38, 37.94, 21.96, 21.87. **IR (ν_{max}):** 3356.97, 3032.59, 2981.32, 2929.04, 1722.48, 1527.14, 1455.01 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{21}H_{26}NO_4$** $[M+H]^+$ 356.1862, found 356.1864.

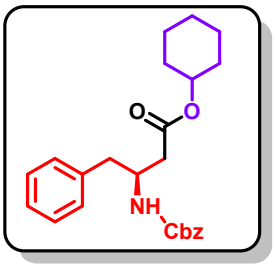
tert-Butyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (6ae):



The title compound of **(6ae)** was prepared according to the general procedure as described in section **(6aa)** using **4a** and *tert*-butanol. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.3 (10% ethyl acetate / hexane); 70% yield of **6ae** as a white solid and **mp:** 57-59 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.37 – 7.16 (m, 10H), 5.35 (d, *J* = 9.0 Hz, 1H), 5.07 (s, 2H), 4.23 – 4.16 (m, 1H), 2.96 – 2.80 (m, 2H), 2.46 – 2.31 (m, 2H), 1.44 (s, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 171.01, 155.72, 137.75, 136.72, 129.51, 128.65, 128.60, 128.15, 128.13, 126.74, 81.30, 66.65, 49.67, 40.41, 38.73, 28.20. **IR**

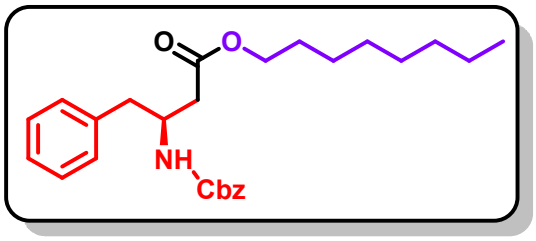
(ν_{max}): 3324.36, 3063.95, 2976.93, 2928.19, 1720.55, 1505.86, 1452.99 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{22}H_{27}NNaO_4$** $[M+Na]^+$ 392.1838, found 392.1838.

Cyclohexyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (6af):



The title compound **(6af)** was prepared according to the general procedure as described in section (**6aa)** using **4a** and cyclohexanol. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.4 (10% ethyl acetate / hexane); 77% yield of **6af** as a white solid and **mp:** 90-92 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.37 – 7.16 (m, 10H), 5.32 (d, *J* = 8.5 Hz, 1H), 5.07 (s, 2H), 4.81 – 4.75 (m, 1H), 4.27 – 4.18 (m, 1H), 2.98 – 2.81 (m, 2H), 2.53 – 2.40 (m, 2H), 1.86 – 1.69 (m, 4H), 1.56 – 1.21 (m, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 171.12, 155.71, 137.65, 136.69, 129.52, 128.68, 128.61, 128.18, 128.12, 126.79, 73.27, 66.68, 49.56, 40.36, 37.92, 31.77, 31.66, 25.41, 23.86. **IR (ν_{max}):** 3337.70, 3066.07, 2938.38, 2860.19, 1723.80, 1528.07, 1451.14 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{24}H_{30}NO_4$** $[M+H]^+$ 396.2175, found 396.2177.

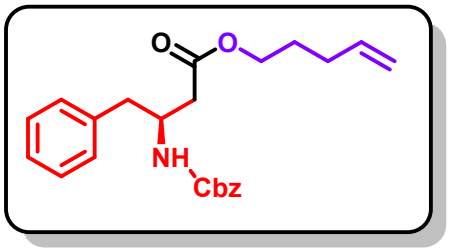
Octyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (6ag):



The title compound of **(6ag)** was prepared according to the general procedure as described in section (**6aa)** using **4a** and octan-1-ol. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.36 (10% ethyl acetate / hexane); 70% yield of **6ag** as a white solid and **mp:** 38-40 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.37 – 7.16 (m, 10H), 5.34 (d, *J* = 8.3 Hz, 1H), 5.07 (s, 2H), 4.25 – 4.18 (m, 1H), 4.10 – 4.02 (m, 2H), 2.98 – 2.82 (m, 2H), 2.55 – 2.43 (m, 2H), 1.64 – 1.58 (m, 2H), 1.30 – 1.27 (m, 10H), 0.88 (t, *J* = 6.7 Hz, 3H).

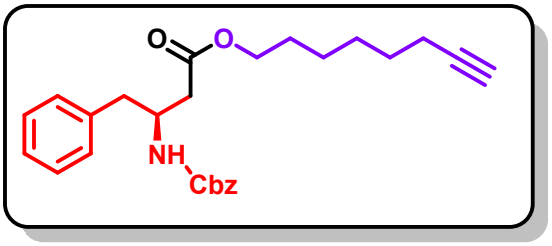
^{13}C NMR (101 MHz, $CDCl_3$) δ 171.79, 155.73, 137.64, 136.64, 129.48, 128.69, 128.62, 128.19, 128.12, 126.80, 66.70, 65.02, 49.46, 40.32, 37.54, 31.90, 29.31, 28.68, 26.03, 22.76, 14.22. **IR (ν_{max}):** 3336.65, 3063.77, 2927.86, 2855.42, 1725.99, 1502.25, 1455.10 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{26}H_{36}NO_4$** $[M+H]^+$ 426.2639, found 426.2649.

Pent-4-en-1-yl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (6ah):



The title compound of **(6ah)** was prepared according to the general procedure as described in section **(6aa)** using **4a** and pent-4-en-1-ol. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.35 (10% ethyl acetate / hexane); 64% yield of **6ah** as a white solid and **mp:** 49-51 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.36 – 7.16 (m, 10H), 5.84 – 5.73 (m, 1H), 5.34 (d, *J* = 8.2 Hz, 1H), 5.07 (s, 2H), 4.98 (d, *J* = 11.3 Hz, 2H), 4.27 – 4.19 (m, 1H), 4.09 (t, *J* = 6.5 Hz, 2H), 2.98 – 2.81 (m, 2H), 2.56 – 2.43 (m, 2H), 2.11 (q, *J* = 7.0 Hz, 2H), 1.75 – 1.68 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 171.68, 155.72, 137.60, 137.42, 136.64, 129.46, 128.69, 128.61, 128.18, 128.12, 126.81, 115.54, 66.71, 64.23, 49.47, 40.34, 37.61, 30.11, 27.83. **IR (ν_{max}):** 3416.50, 3344.18, 3304.23, 2920.77, 1733.43, 1534.19, 1452.92 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{23}H_{28}NO_4$** $[M+H]^+$ 382.2013, found 382.2029.

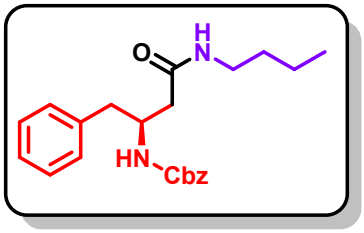
Oct-7-yn-1-yl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (6ai):



The title compound of **(6ai)** was prepared according to the general procedure as described in section **(6aa)** using **4a** and hept-6-yn-1-ol. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.6 (10% ethyl acetate / hexane);

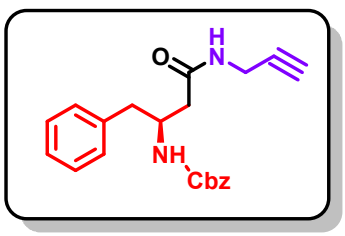
62% yield of **6ai** as a colourless liquid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.37 – 7.16 (m, 10H), 5.33 (d, *J* = 7.3 Hz, 1H), 5.07 (s, 2H), 4.27 – 4.19(m, 1H), 4.07 (t, *J* = 6.2 Hz, 2H), 2.98 – 2.81 (m, 2H), 2.55 – 2.43 (m, 2H), 2.18 (t, *J* = 6.5 Hz, 2H), 1.94 (s, 1H), 1.69 – 1.31 (m, 8H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 171.74, 155.73, 137.61, 136.64, 129.48, 128.70, 128.63, 128.20, 128.13, 126.82, 84.57, 68.46, 66.72, 64.84, 49.47, 40.35, 37.61, 28.55, 28.39, 25.56, 18.43. **IR (ν_{max}):** 3306.93, 2944.59, 1728.19, 1522.16, 1454.90 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{26}H_{32}NO_4$** $[M+H]^+$ 422.2326, found 422.2333

Benzyl (*S*)-(4-(butylamino)-4-oxo-1-phenylbutan-2-yl) carbamate (6aj):



The title compound of **(6aj)** was prepared according to the general procedure as described in section (**6aa)** using **4a** and butylamine. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.4 (50% ethyl acetate / hexane); 74% yield of **6aj** as a white solid. **mp:** 140-142 °C. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.36 – 7.17 (m, 10H), 5.79 (d, *J* = 6.9 Hz, 1H), 5.58 (s, 1H), 5.07 (s, 2H), 4.15 – 4.11 (m, 1H), 3.25 – 3.17 (m, 2H), 3.04 – 2.81 (m, 2H), 2.44 – 2.27 (m, 2H), 1.47 – 1.42 (m, 2H), 1.35 – 1.31 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 170.80, 156.09, 138.15, 136.73, 129.43, 128.71, 128.62, 128.16, 128.04, 126.76, 66.64, 50.32, 40.35, 39.34, 39.01, 31.73, 20.19, 13.86. **IR (ν_{max}):** 3317.85, 2959.55, 2926.32, 1694.68, 1642.60, 1543.32 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{22}H_{29}N_2O_3$** $[M+H]^+$ 369.2173, found 369.2186.

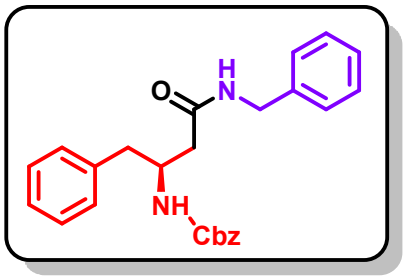
Benzyl (*S*)-(4-oxo-1-phenyl-4-(prop-2-yn-1-ylamino) butan-2-yl) carbamate (6ak):



The title compound of **(6ak)** was prepared according to the general procedure as described in section (**6aa)** using **4a** and prop-2-yn-1-amine. The extracted mixture was concentrated under

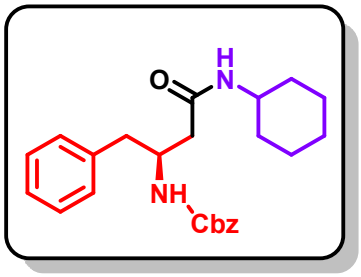
vacuum and the product was purified by flash chromatography **R*f*** = 0.50 (50% ethyl acetate / hexane); 66% yield of **6ak** as a brownish solid. **mp:** 147-148°C. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.34 – 7.26 (m, 10H), 5.82 (s, 1H), 5.66 (d, *J* = 7.4 Hz, 1H), 5.08 (s, 2H), 4.18 – 4.13 (m, 1H), 4.08 – 3.95 (m, 2H), 3.04 – 3.00 (m, 1H), 2.86 – 2.82 (m, 1H), 2.47 – 2.44 (m, 1H), 2.36 – 2.32 (m, 1H), 2.23 (t, *J* = 2.5 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** 170.49, 155.97, 137.79, 136.51, 129.31, 128.67, 128.53, 128.11, 127.98, 126.73, 79.23, 71.71, 66.63, 50.05, 40.14, 38.66, 29.08. **IR (ν_{max}):** 3299.28, 1698.42, 1648.29, 1543.30, 1270.16 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{21}H_{23}N_2O_3$** $[M+H]^+$ 351.1703, found 351.1717.

Benzyl (*S*)-(4-(benzylamino)-4-oxo-1-phenylbutan-2-yl) carbamate (6ai):



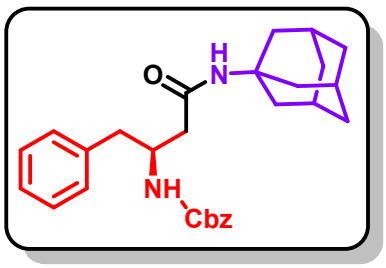
The title compound of **(6ai)** was prepared according to the general procedure as described in section **(6aa)** and using **4a** and phenylmethanamine. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.6 (5% methanol/ chloroform); 69% yield of **6ai** as a white solid. **mp:** 168-170 °C. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.33 – 7.13 (m, 15H), 5.91 (s, 1H), 5.76 (s, 1H), 5.04 (s, 2H), 4.45 – 4.37 (m, 2H), 4.16 (d, *J* = 5.5 Hz, 1H), 3.01 – 2.81 (m, 2H), 2.48 – 2.33 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 170.73, 156.06, 138.12, 136.69, 129.41, 128.89, 128.71, 128.63, 128.18, 128.06, 127.97, 127.76, 126.76, 66.68, 50.33, 43.69, 40.33, 39.02. **IR (ν_{max}):** 3306.09, 1695.95, 1644.69, 1542.38 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{25}H_{27}N_2O_3$** $[M+H]^+$ 403.2016, found 403.2030.

Benzyl (*S*)-(4-(cyclohexylamino)-4-oxo-1-phenylbutan-2-yl) carbamate (6am):



The title compound of **(6am)** was prepared according to the general procedure as described in section (**6aa)** using **4a** and cyclohexanamine. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.4 (50% ethyl acetate / hexane); 61% yield of **6am** as a white solid. **mp:** 150-152 °C. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.36 – 7.18 (m, 10H), 5.81 (s, 1H), 5.47 (s, 1H), 5.07 (s, 2H), 4.16 – 4.09 (m, 1H), 3.79 – 3.72 (m, 1H), 3.04 – 2.80 (m, 2H), 2.42 – 2.24 (m, 2H), 1.92 – 1.83 (m, 2H), 1.68 – 1.59 (m, 2H), 1.38 – 1.32 (m, 2H), 1.18 – 1.00 (m, 4H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 169.74, 155.96, 154.96, 138.03, 136.62, 129.32, 128.58, 128.48, 128.02, 127.93, 126.62, 66.50, 50.21, 48.24, 40.23, 38.98, 33.18, 32.99, 25.46, 24.85. **IR (ν_{max}):** 3305.10, 2928.54, 2853.52, 1694.42, 1635.64, 1542.30 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{24}H_{31}N_2O_3$** $[M+H]^+$ 395.2329, found 395.2344.

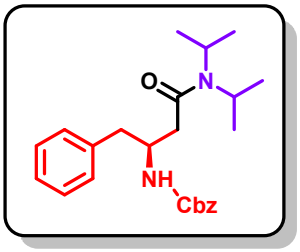
Benzyl((*S*)-4-(((3R,5R,7R)-adamantan-1-yl) amino)-4-oxo-1-phenylbutan-2-yl) carbamate (6an):



The title compound of **(6an)** was prepared according to the general procedure as described in section (**6aa)** using **4a** and adamantan-1-amine. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.35 (30% ethyl acetate / hexane); 63% yield of **6an** as a white solid. **mp:** 168-170 °C. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.34 – 7.21 (m, 10H), 5.96 (d, *J* = 7.6 Hz, 1H), 5.21 (s, 1H), 5.07 (s, 2H), 4.13 – 4.06 (m, 1H), 3.05 – 3.01 (m, 1H), 2.84 – 2.79 (m, 1H), 2.36 – 2.32 (m, 1H), 2.17 – 2.13 (m, 1H), 2.07 (s, 3H), 1.99 – 1.97 (m, 6H), 1.67 (t, *J* = 2.8 Hz, 6H). **^{13}C**

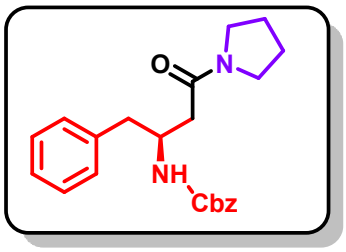
NMR (101 MHz, $CDCl_3$) δ 170.10, 155.92, 138.21, 136.68, 129.34, 128.55, 128.47, 127.99, 127.92, 126.57, 66.45, 52.21, 50.32, 41.60, 40.13, 39.40, 36.32, 29.40. **IR (ν_{max}):** 3341.08, 2909.65, 1703.61, 1648.50, 1545.46 cm^{-1}. **HRMS (ESI):** *m/z* calcd for $C_{28}H_{35}N_2O_3$ $[M+H]^+$ 447.2642, found 447.2648.

Benzyl (*S*)-(4-(diisopropylamino)-4-oxo-1-phenylbutan-2-yl) carbamate (6ao):



The title compound of **(6ao)** was prepared according to the general procedure as described in section (**6aa)** using **4a** and diisopropylamine. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.3 (20% ethyl acetate / hexane); 70% yield of **6ao** as a white solid. **mp:** 83-85 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.36 – 7.17 (m, 10H), 6.15 (d, *J* = 8.2 Hz, 1H), 5.08 (s, 2H), 4.18 – 4.12 (m, 1H), 3.73 – 3.69 (m, 1H), 3.45 – 3.40 (m, 1H), 3.06 – 2.94 (m, 2H), 2.39 – 2.37 (m, 2H), 1.40 (dd, *J* = 19.4, 6.8 Hz, 6H), 1.06 (dd, *J* = 31.3, 6.7 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 169.85, 155.97, 138.75, 136.82, 129.37, 128.50, 128.45, 127.93, 126.45, 66.36, 50.18, 48.46, 45.77, 40.00, 36.21, 20.78, 20.69, 20.64. **IR (ν_{max}):** 3414.36, 3305.56, 2968.45, 1719.97, 1630.31, 1499.48 cm^{-1}. **HRMS** (ESI): *m/z* calcd for $C_{24}H_{33}N_2O_3$ $[M+H]^+$ 397.2486, found 397.2492.

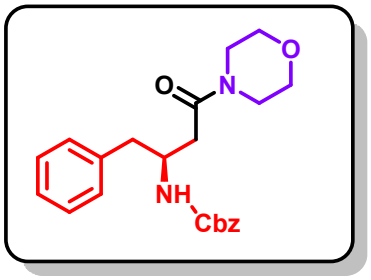
Benzyl (*S*)-(4-oxo-1-phenyl-4-(pyrrolidin-1-yl) butan-2-yl) carbamate (6ap):



The title compound of **(6ap)** was prepared according to the general procedure as described in section (**6aa)** using **4a** and pyrrolidine. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.2 (40% ethyl acetate / hexane); 65% yield of **6ap** as a white solid. **mp:** 84 – 86 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.34 – 7.17 (m, 10H), 6.21 (d, *J* = 8.5 Hz, 1H), 5.07 (s, 2H), 4.22 – 4.16 (m, 1H), 3.51 – 3.40 (m, 2H), 3.24 – 3.18 (m, 1H), 3.12 – 3.06 (m, 2H), 2.91 (dd, *J* = 13.4, 8.5 Hz, 1H), 2.40

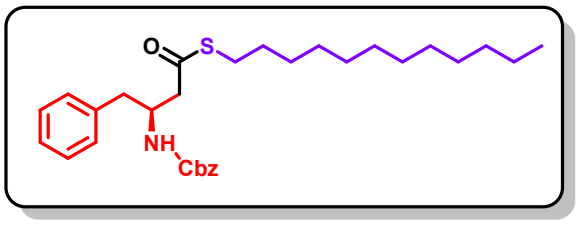
(d, *J* = 4.7 Hz, 2H), 1.96 – 1.73 (m, 4H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 169.75, 156.03, 138.73, 136.91, 129.36, 128.60, 128.55, 128.01, 126.57, 66.45, 49.96, 46.68, 45.64, 40.15, 36.24, 26.10, 24.45. **IR (ν_{max}):** 3317.29, 2975.80, 1718.64, 1627.86, 1500.42 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{22}H_{27}N_2O_3$** $[M+H]^+$ 367.2016, found 367.2023.

Benzyl (*S*)-(4-morpholino-4-oxo-1-phenylbutan-2-yl) carbamate (6aq):



The title compound of **(6aq)** was prepared according to the general procedure as described in section (**6aa)** using **4a** and morpholine. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.25 (50% ethylacetate / hexane); 65% yield of **6aq** as a white solid. **mp:** 80-82 °C. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.36 – 7.17 (m, 10H), 5.83 (d, *J* = 7.2 Hz, 1H), 5.07 (s, 2H), 4.20 – 4.15 (m, 1H), 3.65 – 3.54 (m, 6H), 3.29 (s, 2H), 3.07 – 2.91 (m, 2H), 2.55 – 2.41 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 169.66, 155.99, 138.37, 136.72, 129.37, 128.71, 128.59, 128.15, 128.08, 126.74, 66.94, 66.59, 50.06, 46.05, 41.90, 40.08, 35.08. **IR (ν_{max}):** 3295.44, 2920.89, 2854.92, 1718.51, 1641.69, 1502.81 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{22}H_{27}N_2O_4$** $[M+H]^+$ 383.1965, found 383.1979.

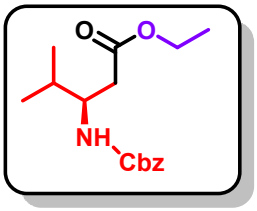
S-Dodecyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanethioate (6ar):



The title compound of **(6ar)** was prepared according to the general procedure as described in section (**6aa)** using **4a** and dodecane-1-thiol. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.35 (5% ethyl acetate / hexane); 80% yield of **6ar** as a white solid. **mp:** 57-59 °C. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.34 – 7.17 (m, 10H), 5.26 (d, *J* = 8.0 Hz, 1H), 5.07 (s, 2H), 4.25 – 4.21 (m, 1H), 2.95 – 2.82 (m, 4H), 2.74 (d, *J*

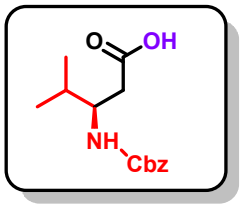
= 5.4 Hz, 2H), 1.58 – 1.53 (m, 2H), 1.36 – 1.31 (m, 2H), 1.25 (s, 16H), 0.88 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 198.12, 155.66, 137.53, 136.63, 129.51, 128.68, 128.60, 128.17, 128.11, 126.82, 66.71, 50.30, 46.52, 40.19, 32.03, 29.75, 29.70, 29.61, 29.56, 29.46, 29.22, 29.20, 28.95, 22.80, 14.24. **IR (ν_{max}):** 3323.23, 3033.84, 2924.10, 2853.77, 1723.68, 1523.97, 1459.62 cm^{-1}. **HRMS** (ESI): *m/z* calcd for **$C_{30}H_{44}NO_3S$** $[M+H]^+$ 498.3036, Found 498.3050.

Ethyl (*R*)-3-(((benzyloxy)carbonyl) amino)-4-methylpentanoate (6ba):



The title compound of **(6ba)** was prepared according to the general procedure as described in section (**6aa)** using **4b** and ethanol. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.25 (10% Ethyl acetate / Hexane); 75% yield of **6ba** as a Colourless liquid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.36 – 7.27 (m, 5H), 5.18 (d, *J* = 9.0 Hz, 1H), 5.08 (s, 2H), 4.11 (q, *J* = 7.1 Hz, 2H), 3.87 – 3.80 (m, 1H), 2.55 – 2.44 (m, 2H), 1.88 – 1.79 (m, 1H), 1.22 (t, *J* = 7.1 Hz, 3H), 0.92 (d, *J* = 6.8 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 171.83, 156.11, 136.71, 128.55, 128.10, 66.67, 60.68, 53.72, 37.21, 31.86, 19.34, 18.57, 14.21. **IR (ν_{max}):** 3345.58, 2966.26, 1733.84, 1536.13, 1464.09 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{16}H_{24}NO_4$** $[M+H]^+$ 294.1700, Found 294.1701.

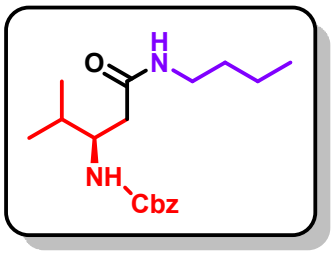
(*R*)-3-(((Benzyloxy)carbonyl) amino)-4-methylpentanoic acid (6bc):



The title compound of **(6bc)** was prepared according to the general procedure as described in section (**6aa)** using **4b** and water. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.4 (5% methanol/ chloroform); 72% yield of **6bc** as a white solid. **mp:** 80-82 °C. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.36 – 7.28 (m, 5H), 5.29 (d, *J* = 9.4 Hz, 1H), 5.15 – 5.06 (m, 2H), 3.86 – 3.80 (m, 1H), 2.60 – 2.52 (m, 2H), 1.91 – 1.84 (m, 1H), 0.92 (dd, *J* =

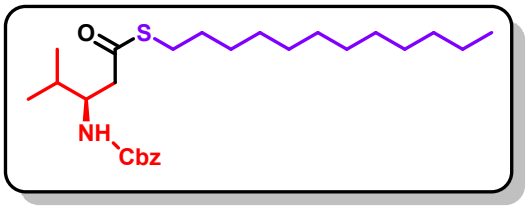
6.5, 3.3 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 176.48, 156.35, 136.56, 128.59, 128.17, 128.12, 66.87, 53.59, 36.87, 31.73, 19.40, 18.63. **IR (ν_{max}):** 3347.48, 3303.23, 2965.27, 1715.19, 1528.82, 1458.16 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{14}H_{20}NO_4$** $[M+H]^+$ 266.1387, Found 266.1397. Verified the analytical data with those reported in the literature.[10]

Benzyl (*R*)-(1-(butylamino)-4-methyl-1-oxopentan-3-yl) carbamate (6bj):



The title compound of **(6bj)** was prepared according to the general procedure as described in section **(6aa)** using **4b** and butylamine. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.25 (40% ethyl acetate / hexane); 77% yield of **6bj** as a white solid. **mp:** 140-142 °C.**^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.35 – 7.28 (m, 5H), 6.08 (s, 1H), 5.54 (d, *J* = 8.8 Hz, 1H), 5.07 (s, 2H), 3.72 – 3.67 (m, 1H), 3.21 – 3.13 (m, 2H), 2.46 – 2.35 (m, 2H), 1.89 – 1.82 (m, 1H), 1.45 – 1.39 (m, 2H), 1.34 – 1.26 (m, 2H), 0.90 (dd, *J* = 13.8, 6.5 Hz, 9H) **^{13}C NMR (101 MHz, $CDCl_3$)** δ 171.07, 156.63, 136.68, 128.58, 128.14, 127.98, 66.73, 54.36, 39.35, 39.16, 32.03, 31.61, 20.14, 19.55, 18.65, 13.83. **IR (ν_{max}):** 3310.02, 2958.65, 2928.64, 1691.88, 1642.85, 1547.38 cm^{-1}. **HRMS** (ESI): *m/z* calcd for **$C_{18}H_{29}N_2O_3$** $[M+H]^+$ 321.2173, Found 321.2182.

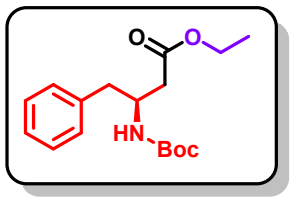
S-Dodecyl (*R*)-3-(((benzyloxy)carbonyl) amino)-4-methylpentanethioate (6br):



The title compound of **(6br)** was prepared according to the general procedure as described in section **(6aa)** using **4b** and dodecane-1-thiol. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.30 (5% ethyl acetate / hexane); 82% yield of **6br** as a colourless liquid. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.35 – 7.27 (m, 5H), 5.16 (d, *J* = 9.5 Hz, 1H), 5.09 (s, 2H), 3.89 – 3.82 (m, 1H), 2.85 (t, *J* = 7.3 Hz, 2H), 2.80 – 2.70 (m, 2H), 1.87 – 1.82 (m, 1H), 1.57 – 1.50 (m, 2H), 1.35 –

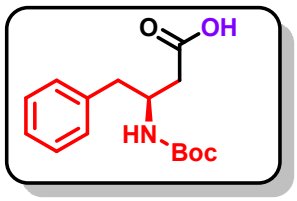
1.25 (m, 18H), 0.94 – 0.82 (m, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 197.99, 156.06, 136.72, 128.55, 128.07, 66.70, 54.46, 46.04, 32.00, 31.75, 29.72, 29.67, 29.57, 29.52, 29.43, 29.19, 29.15, 28.91, 22.77, 19.46, 18.54, 14.21. **IR (ν_{max}):** 3351.20, 2925.53, 2854.13, 1700.27, 1531.85, 1463.38 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{26}H_{44}NO_3S$** $[M+H]^+$ 450.3036, found 450.3041.

Ethyl (*S*)-3-((tert-butoxycarbonyl) amino)-4-phenylbutanoate (6ca):



The title compound of **(6ca)** was prepared according to the general procedure as described in section **(6aa)** using **4c** and with ethanol. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.40 (10% ethyl acetate / hexane); 65% yield of **6ca** as a colourless liquid. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.23 – 7.11 (m, 5H), 4.99 (d, *J* = 6.5 Hz, 1H), 4.09 – 4.05 (m, 3H), 2.89 – 2.72 (m, 2H), 2.44 – 2.33 (m, 2H), 1.33 (s, 9H), 1.19 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 171.71, 155.16, 137.78, 129.41, 128.50, 126.57, 79.33, 60.60, 48.84, 40.42, 37.76, 28.36, 14.21. **IR (ν_{max}):** 3398.07, 2975.02, 1712.15, 1507.57, 1455.75 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{17}H_{26}NO_4$** $[M+H]^+$ 308.1856, found 308.1863.

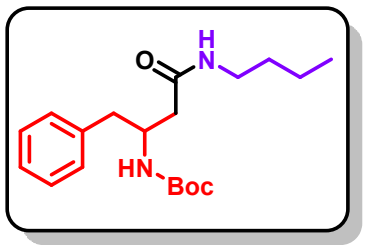
(S)-3-((Tert-butoxycarbonyl) amino)-4-phenylbutanoic acid (6cc):



The title compound of **(6cc)** was prepared according to the general procedure as described in section **(6aa)** using **4c** and with water. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.40 (5% methanol / chloroform); 69% yield of **6cc** as an off-white solid. **mp:** 100 - 102 °C. **^{1}H NMR (400 MHz, $CDCl_3$ mixture of rotamers)** δ 7.31 – 7.19 (m, 5H), 5.11 (s, 1H), 4.19 – 4.05 (m, 1H), 2.93 – 2.83 (m, 2H), 2.57 – 2.46 (m, 2H), 1.40 (s, 9H). **^{13}C NMR (101 MHz, $CDCl_3$ mixture of rotamers)** δ 176.00, 155.41, 137.86, 129.52, 128.64, 126.71, 79.67, 48.80, 40.39, 37.51, 28.45. **IR (ν_{max}):** 3316.58, 3149.68, 2978.62,

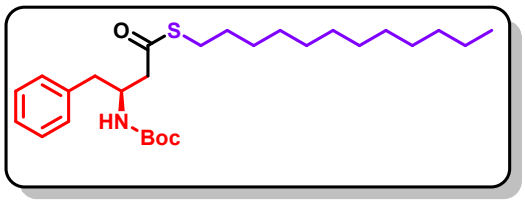
1711.50, 1503.00, 1453.29 cm^{-1}. **HRMS (ESI):** *m/z* calcd for $C_{15}H_{22}NO_4$ $[M+H]^+$ 280.1543, found 280.1553. Verified the analytical data with those reported in the literature.[10]

***tert*-butyl (4-(butylamino)-4-oxo-1-phenylbutan-2-yl) carbamate (6cj):**



The title compound of **(6cj)** was prepared according to the general procedure as described in section (**6aa)** using **4c** and butyl amine. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.40 (40% ethyl acetate / hexane); 73% yield of **6cj** as a white solid. **mp:**148-150 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.31 – 7.19 (m, 5H), 5.75 (s, 1H), 5.43 (d, *J* = 4.3 Hz, 1H), 4.10 – 4.02 (m, 1H), 3.31 – 3.17 (m, 2H), 3.01 – 2.96 (m, 1H), 2.82 – 2.77 (m, 1H), 2.44 – 2.24 (m, 2H), 1.52 – 1.44 (m, 2H), 1.40 (s, 9H), 1.38 – 1.30 (m, 2H), 0.93 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 170.95, 155.75, 138.29, 129.45, 128.64, 126.65, 79.49, 49.72, 40.63, 39.50, 39.35, 31.74, 28.49, 20.20, 13.86. **IR (ν_{max}):** 3344.10, 2978.51, 2928.19, 1690.75, 1647.30, 1533.41 cm^{-1}. **HRMS (ESI):** *m/z* calcd for $C_{19}H_{31}N_2O_3$ $[M+H]^+$ 335.2329, found 335.2338.

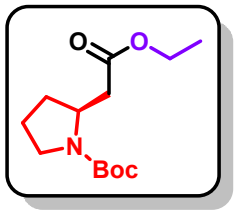
S-dodecyl (S)-3-((*tert*-butoxycarbonyl) amino)-4-phenylbutanethioate (6cr):



The title compound of **(6cr)** was prepared according to the general procedure as described in section (**6aa)** using **4c** and dodecane-1-thiol. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.30 (5% ethyl acetate / hexane); 70% yield of **6cr** as a brownish solid. **mp:** 62-64 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.31 – 7.19 (m, 5H), 4.98 (s, 1H), 4.15 (d, *J* = 7.1 Hz, 1H), 2.90 – 2.79 (m, 3H), 2.72 (d, *J* = 5.5 Hz, 2H), 1.60 – 1.53 (m, 3H), 1.41 (s, 9H), 1.26 (s, 18H), 0.88 (t, *J* = 6.8 Hz, 3H).**^{13}C NMR (101 MHz, $CDCl_3$)** δ 198.24, 155.07, 137.69, 129.46, 128.52, 126.61, 79.39, 49.76, 46.55, 40.18, 31.93, 29.64, 29.60, 29.51, 29.36, 29.13, 29.08, 28.86, 28.37, 22.70, 14.14.

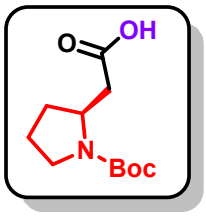
IR (ν_{max}): 3378.54, 3022.69, 2928.34, 2854.03, 1713.50, 1498.78 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{27}H_{46}NO_3S$** $[M+H]^+$ 464.3193, Found 464.3202.

***tert*-butyl (S)-2-(2-ethoxy-2-oxoethyl) pyrrolidine-1-carboxylate (6da):**



The title compound of **(6da)** was prepared according to the general procedure as described in section (**6aa)** using **4d** and ethanol. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.30 (10% ethyl acetate / hexane); 68% yield of **6da** as a colourless liquid. **^{1}H NMR (500 MHz, $CDCl_3$, mixture of rotamers)** δ 4.14– 4.05 (m, 3H), 3.39– 3.29 (m, 2H), 2.92 – 2.73 (m, 1H), 2.28 – 2.23 (m, 1H), 2.05 – 1.97 (m, 1H), 1.86 – 1.73 (m, 3H), 1.42 (s, 9H), 1.21 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$, mixture of rotamers)** δ 171.69, 154.41, 79.67, 79.37, 60.44, 54.18, 46.69, 46.31, 39.49, 38.69, 31.36, 30.61, 23.64, 22.92, 14.33. **IR (ν_{max}):** 2980.19, 2887.22, 1737.98, 1699.44, 1463.34, 1399.88, 1304.59, 1253.12, 1174.13, 1115.81, 1038.30, 906.32, 773.93, 631.97 cm^{-1}. **HRMS (ESI):** *m/z* calcd for **$C_{13}H_{24}NO_4$** $[M+H]^+$ 258.1700, Found 258.1699.

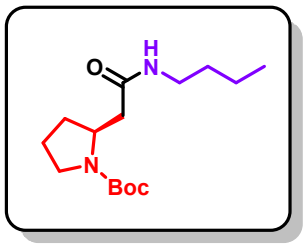
(S)-2-(1-(*tert*-butoxycarbonyl) pyrrolidin-2-yl) acetic acid (6dc):



The title compound of **(6dc)** was prepared according to the general procedure as described in section (**6aa)** using **4d** and water. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.40 (5% methanol/ chloroform); 75% yield of **6dc** as a viscous liquid. **^{1}H NMR (500 MHz, $CDCl_3$ mixture of rotamers)** δ 4.32 – 4.13 (m, 1H), 3.34 (s, 2H), 2.96– 2.83 (m, 1H), 2.35 – 2.30 (m, 1H), 2.11 – 2.03 (m, 1H), 1.85 – 1.76 (m, 3H), 1.44 (s, 9H). **^{13}C NMR (101 MHz, $CDCl_3$, mixture of rotamers)** δ 176.33, 154.53, 79.94, 53.98, 46.64, 39.16, 31.41, 28.51, 23.53, 22.83. **IR (ν_{max}):** 3319.96, 3149.77, 2979.04, 2893.60,

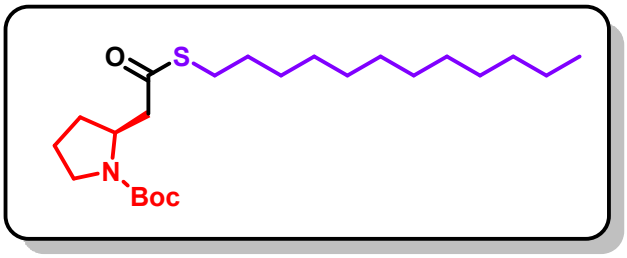
1736.65 cm^{-1}. **HRMS (ESI):** *m/z* calcd for $\mathbf{C_{11}H_{20}NO_4}$ $[M+H]^+$ 230.1387, Found 230.1387. Verified the analytical data with those reported in the literature.[10]

***tert*-Butyl (*S*)-2-(2-(butylamino)-2-oxoethyl) pyrrolidine-1-carboxylate (6dj):**



The title compound of **(6dj)** was prepared according to the general procedure as described in section (**6aa)** using **4d** and butylamine. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.50 (50% ethyl acetate / hexane); 70% yield of **6dj** as a yellow viscous liquid. **^{1}H NMR (400 MHz, $CDCl_3$, mixture of rotamers)** δ 4.03 (s, 1H), 3.30 – 3.11(m, 4H), 2.62 (dd, *J* = 13.8, 3.7 Hz, 1H), 2.26 – 2.10 (m, 2H), 1.97 – 1.75 (m, 4H), 1.50 – 1.46 (m, 2H), 1.44 (s, 9H), 1.34 – 1.27 (m, 2H), 0.89 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$, mixture of rotamers)** δ 170.99, 155.09, 79.63, 59.19, 58.81, 55.33, 46.64, 46.40, 41.61, 39.26, 31.74, 30.95, 28.61, 28.38, 23.77, 23.58, 20.15, 13.82. **IR (ν_{max}):** 3334.69, 2966.65, 2874.68, 1745.87, 1689.95, 1551.11 cm^{-1}. **HRMS (ESI):** *m/z* calcd for $\mathbf{C_{15}H_{29}N_2O_3}$ $[M+H]^+$ 285.2173, Found 285.2172.

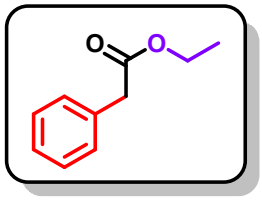
***tert*-Butyl (*S*)-2-(2-(dodecylthio)-2-oxoethyl) pyrrolidine-1-carboxylate (6dr):**



The title compound of **(6dr)** was prepared according to the general procedure as described in section (**6aa)** using **4d** and dodecane-1-thiol. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.45 (5% ethyl acetate / hexane);73% yield of **6dr** as a yellow liquid. **^{1}H NMR (500 MHz, $CDCl_3$, mixture of rotamers)** δ 4.15 (d, *J* = 39.1 Hz, 1H), 3.33 (d, *J* = 23.8 Hz, 2H), 3.19 – 2.99 (m, 1H), 2.86 (t, *J* = 6.7 Hz, 2H), 2.60 – 2.51 (m, 1H), 2.03 – 1.96 (m, 1H), 1.85 – 1.82 (m, 3H), 1.57 – 1.54 (m, 2H), 1.47 (d, *J* = 11.3 Hz, 9H), 1.34 – 1.28 (m, 4H), 1.26 (s, 14H), 0.88 (t, *J* = 6.9 Hz, 3H). **^{13}C**

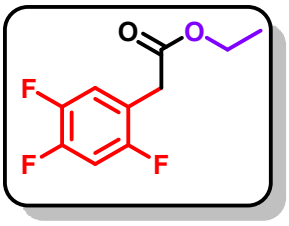
NMR (101 MHz, $CDCl_3$, mixture of rotamers) δ 197.35, 154.16, 79.70, 79.28, 54.64, 48.60, 47.59, 46.57, 46.12, 31.91, 30.78, 30.06, 29.62, 29.56, 29.50, 29.34, 29.10, 28.90, 28.81, 28.52, 23.53, 22.68, 14.11. **IR (ν_{max}):** 3086.93, 2925.77, 2854.94, 1693.69,1458.84, 1393.07 cm^{-1}. **HRMS (ESI):** *m/z* calcd for $C_{23}H_{44}NO_3S$ $[M+H]^+$ 414.3036, found 414.3034.

Ethyl 2-phenylacetate (6ea):



The title compound of **(6ea)** was prepared according to the general procedure as described in section **(6aa)** using **4e** and ethanol. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.5 (15% ethyl acetate / hexane); 60% yield of **6ea** as a colourless liquid. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.34 – 7.25 (m, 5H), 4.15 (q, *J* = 7.1 Hz, 2H), 3.61 (s, 2H), 1.25 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 171.78, 134.30, 129.38, 128.68, 127.17, 61.00 ,41.59, 14.31. **IR (ν_{max}):** 3068.28, 2975.96, 1737.59, 1494.63 cm^{-1}. Verified the analytical data with those reported in the literature.[11]

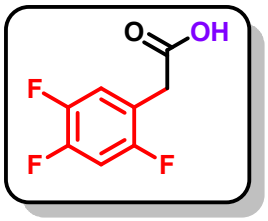
Ethyl 2-(2,4,5-trifluorophenyl) acetate (6fa):



The title compound of **(6fa)** was prepared according to the general procedure as described in section **(6aa)** using **4f** and ethanol. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.60 (10% ethyl acetate / hexane); 61% yield of **6fa** as a colourless liquid. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.15 – 7.10 (m, 1H), 7.04 – 6.91 (m, 1H), 4.20 – 4.16 (m, 2H), 3.64 – 3.60 (m, 2H), 1.26 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 169.08, 168.90, 158.63, 157.26, 156.21, 154.85, 153.83, 153.76, 153.74, 149.68, 149.55, 149.42, 147.19, 147.05, 146.92, 146.87, 146.78, 146.75, 144.48, 144.44, 144.35, 144.32, 118.16, 118.11, 117.96, 117.91, 116.95, 116.91, 116.77, 116.71, 116.66, 116.59,

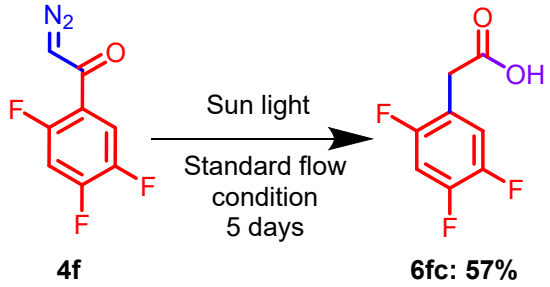
115.42, 115.33, 115.17, 115.08, 114.46, 114.38, 114.22, 114.14, 104.68, 104.47, 104.40, 104.19, 60.32, 33.39, 32.72, 13.09. **IR (ν_{max}):** 2983.21, 2850.28, 1741.27, 1524.18, 1329.92 cm^{-1}. Verified the analytical data with those reported in the literature.[12]

2-(2,4,5-Trifluorophenyl) acetic acid (6fc):



The title compound of **(6fc)** was prepared according to the general procedure as described in section (**6aa)** using **4f** and water. The extracted mixture was concentrated under vacuum and the product was purified by flash chromatography **R*f*** = 0.40 (20% ethyl acetate / hexane); 58% yield of **6fc** as a white solid. **mp:** 122-124 °C. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.14 – 7.09 (m, 1H), 6.97 – 6.92 (m, 1H), 3.66 (s, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 175.57, 157.50, 157.41, 155.04, 154.94, 151.08, 150.95, 150.82, 148.58, 148.45, 148.32, 148.12, 148.09, 147.99, 147.96, 145.69, 145.65, 145.56, 119.39, 119.34, 119.20, 119.15, 117.09, 117.05, 117.00, 116.91, 116.86, 105.99, 105.78, 105.71, 105.50, 33.53. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -118.39 (dd, *J* = 15.2, 3.8 Hz), -134.12 (dd, *J* = 20.9, 3.6 Hz), -142.53 (dd, *J* = 21.7, 15.1 Hz). **IR (ν_{max}):** 3517.90, 3338.84, 2932.40, 1694.76, 1521.21, 1334.80 cm^{-1}.

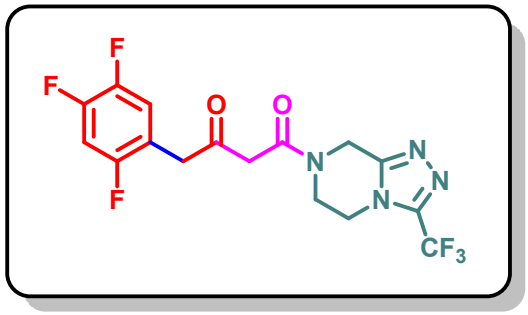
Gram scale synthesis of 6fc:



4f **6fc: 57%**

A stock solution (250 mL) containing **4f**: **5c**: THF with molar ratio of 1:367:223 was manually filled in BD syringe and connected with newly designed syringe pump. The solution phase was passed through a perfluoroalkoxy (PFA) flat reactor {Outer dimeter (OD 1/16 inches), Inner dimeter (ID 1.0 mm), length 10.2-meter, volume = 8.0 mL}. The solar panel reactor was exposed to sun-light (8.30 am to 4:30 pm) for 5 days. The product mixture 230 mL was collected at the end of the photo-flow reactor into a flask and was later purified through the regular methods to obtain product (1.01 g, 57%) as a white solid.

1-(3-(Trifluoromethyl)-5,6-dihydro-[1,2,4]triazolo[4,3-a]pyrazin-7(8H)-yl)-4-(2,4,5-trifluorophenyl)butane-1,3-dione: 9



The corresponding compound has been synthesized by following reported protocol.[13] 2,4,5-Trifluorophenylacetic acid **6fc** (3g, 16 mmol), meldrum's acid (2.5 g, 17 mmol), DMAP (0.15 g, 1.3 mmol), and acetonitrile (15 mL) were charged into a 100 mL three-neck flask. *N*,*N*-diisopropylamine (5.8 mL, 34 mmol) was added in portions at room temperature while maintaining the internal temperature below 50 °C. Pivaloyl chloride (2.1 mL, 17 mmol) was added dropwise over 30 min. while maintaining the temperature below 55 °C. The reaction was stirred at 45– 50 °C for 2-3 h. Triazole hydrochloride (3.6 g, 16 mmol) was added in one portion at 40 – 50°C. Trifluoroacetic acid (36 mL, 0.47 mmol) was added dropwise, and the reaction solution was stirred at 50 – 55 °C for 6 h. Then, 5% aqueous $NaHCO_3$ solution (20 mL) was added between 20-45 °C dropwise. Seeds (5%) were added and the slurry was aged at 20-30 °C for 1 h. Additional 5% aqueous $NaHCO_3$ solution (15 mL) was added dropwise over 2-3 h. After aging for several hours at ambient temperature, the slurry was further cooled to 0-5 °C and stirred for 1h before filtration. The wet cake was washed with 20% acetonitrile in water (10 ml) followed by two slurry washes with 20% acetonitrile in water (10 ml × 2) and finally washed with water (10 mL). The cake was suction dried at ambient temperature. 84% isolated yield of **9 mp**: 90 – 92 °C. **^{1}H NMR (500 MHz, CD_3CN)** δ 7.22 – 7.09 (m, 2H), 4.91 (s, 1H), 4.81 (s, 1H), 4.16 (t, J = 5.5 Hz, 1H), 4.11 (t, J = 5.0 Hz, 1H), 4.00 (t, J = 5.5 Hz, 1H), 3.92 (s, 1H), 3.91 (s, 1H), 3.83 (t, J = 5.5 Hz, 1H), 3.80 (s, 1H), 3.79 (s, 1H). **^{13}C NMR (101 MHz, CD_3CN)** δ 201.40, 167.37, 167.26, 151.83, 151.57, 151.48, 151.43, 151.26, 148.93, 148.80,

148.67, 146.29, 146.17, 120.75, 120.69, 120.55, 120.49, 106.52, 106.31, 106.23, 106.02, 48.78, 48.72, 44.57, 44.12, 43.79, 43.00, 42.93, 39.74, 38.75. **IR (ν_{max}):** 2952.93, 1723.49, 1653.74, 1515.92, 1331.12, 1145.77 cm^{-1}. Verified the analytical data with those reported in the literature. **HRMS (ESI):** *m/z* calcd for $\mathbf{C_{16}H_{12}F_6N_4O_2}$ $[M+H]^+$ 407.0937, Found 407.0938. Verified the analytical data with those reported in the literature.[13]

6. Spectra.

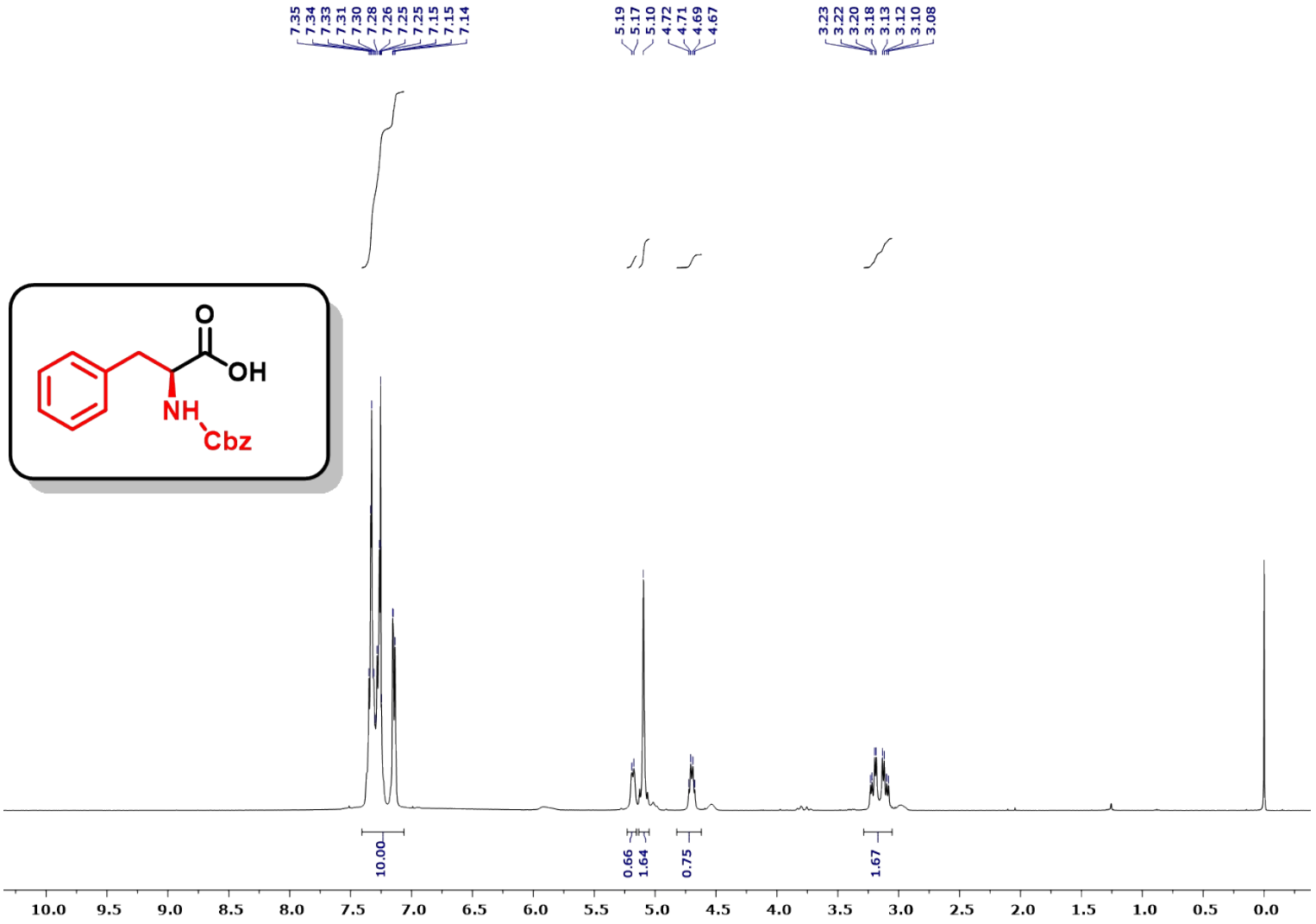


Fig. S12. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of ((benzyloxy)carbonyl)-L-phenylalanine (**2a**).

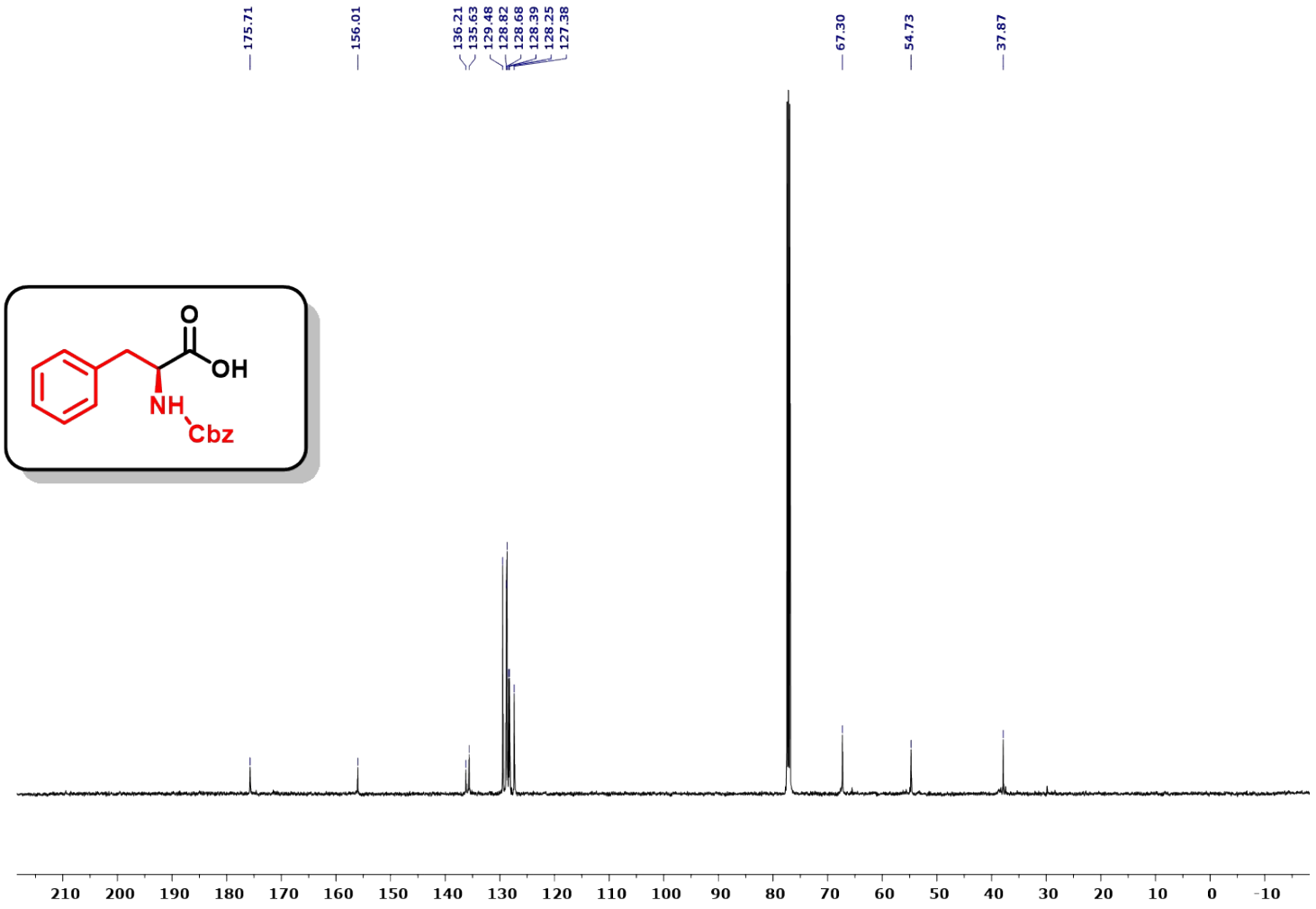


Fig. S13. ^{13}C NMR (125 MHz, $CDCl_3$) spectra of ((benzyloxy)carbonyl)-L-phenylalanine (**2a**).

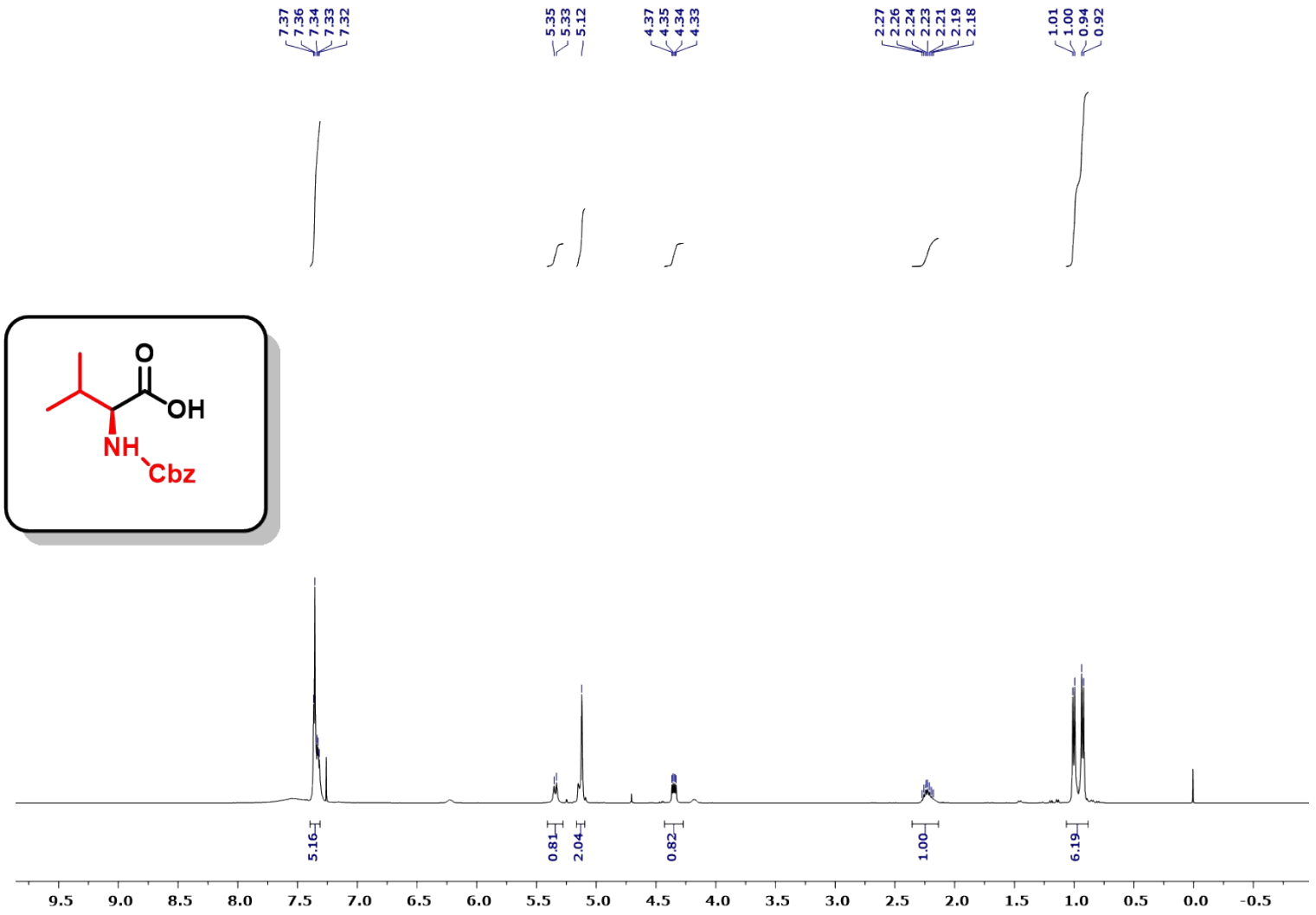


Fig. S14. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of ((benzyloxy)carbonyl)-L-valine (**2b**).

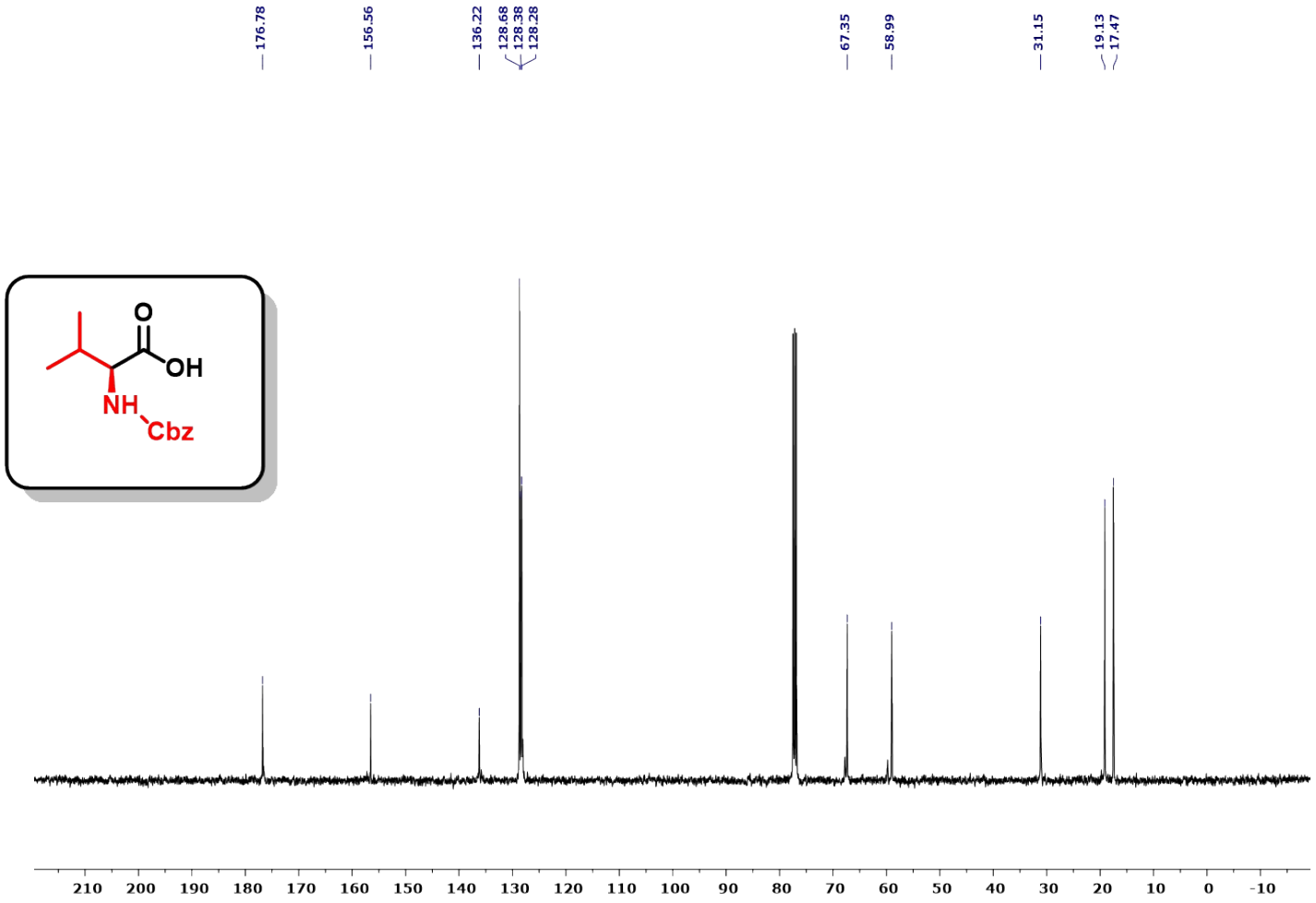


Fig. S15. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of ((benzyloxy)carbonyl)-L-valine (**2b**).

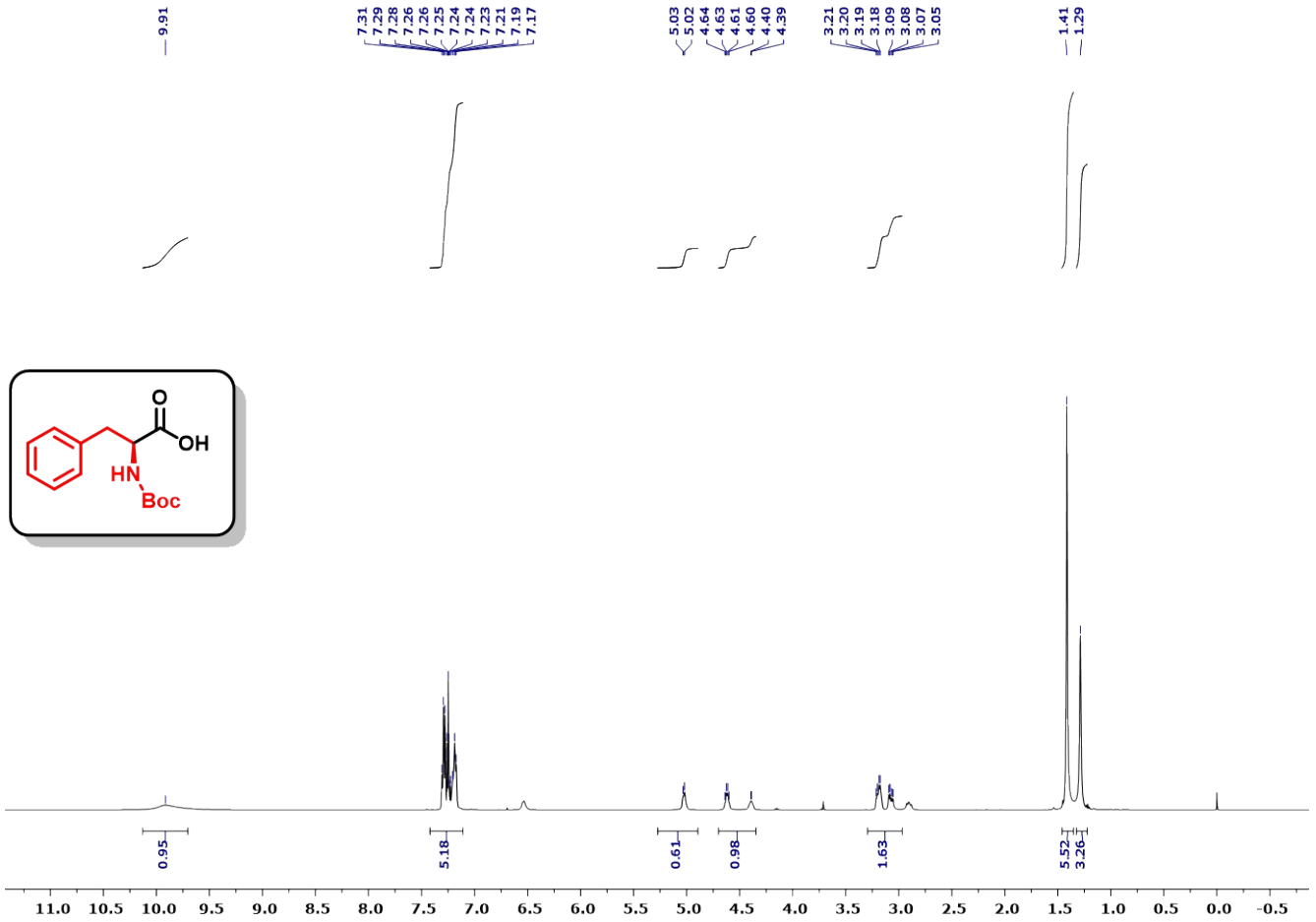


Fig. S16. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of (*tert*-butoxy carbonyl)-L-phenylalanine (**2c**).

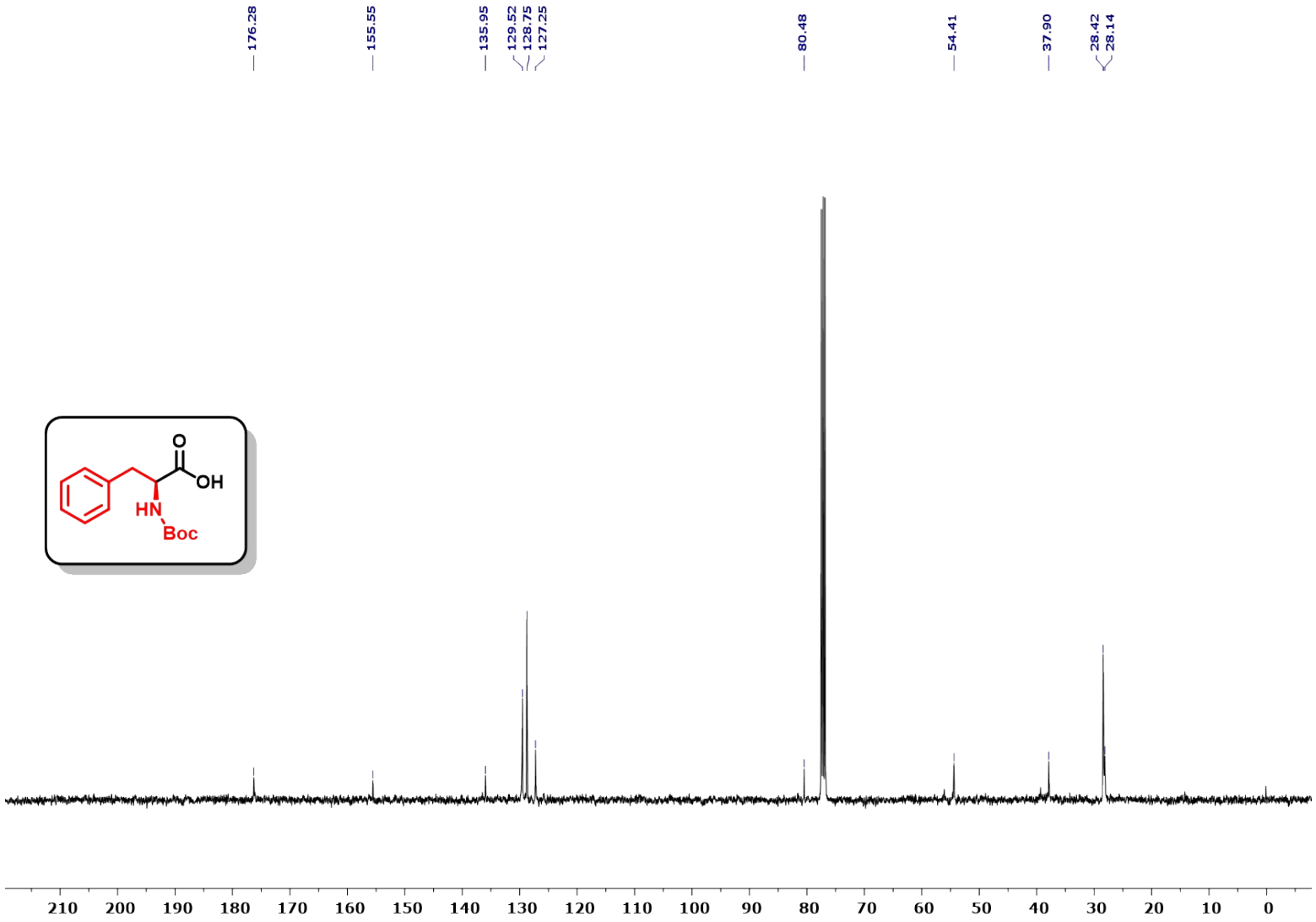


Fig. S17. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of (*tert*-butoxy carbonyl)-L-phenylalanine (**2c**).

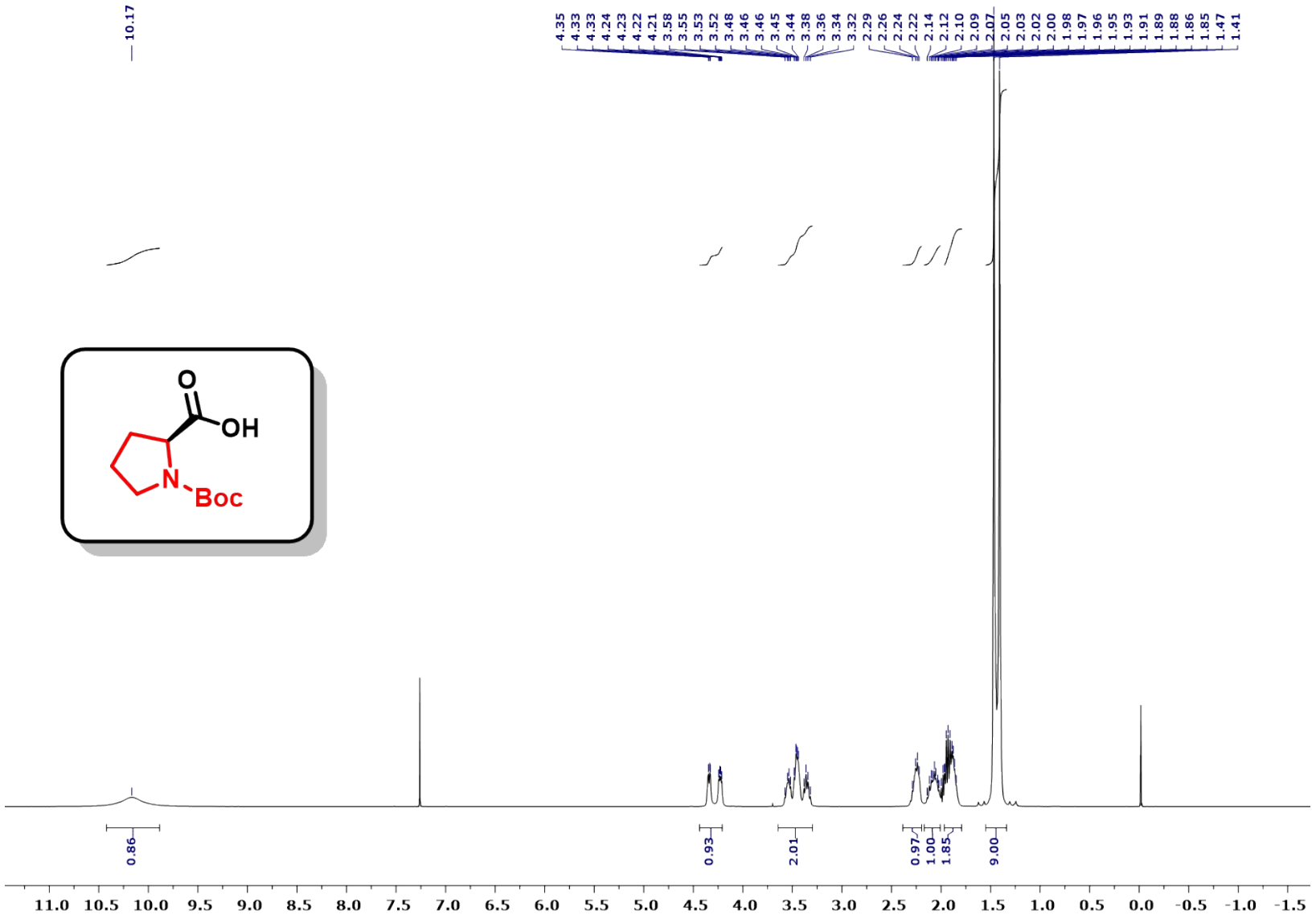


Fig. S18. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of (*tert*-butoxy carbonyl)-L-proline (**2d**).

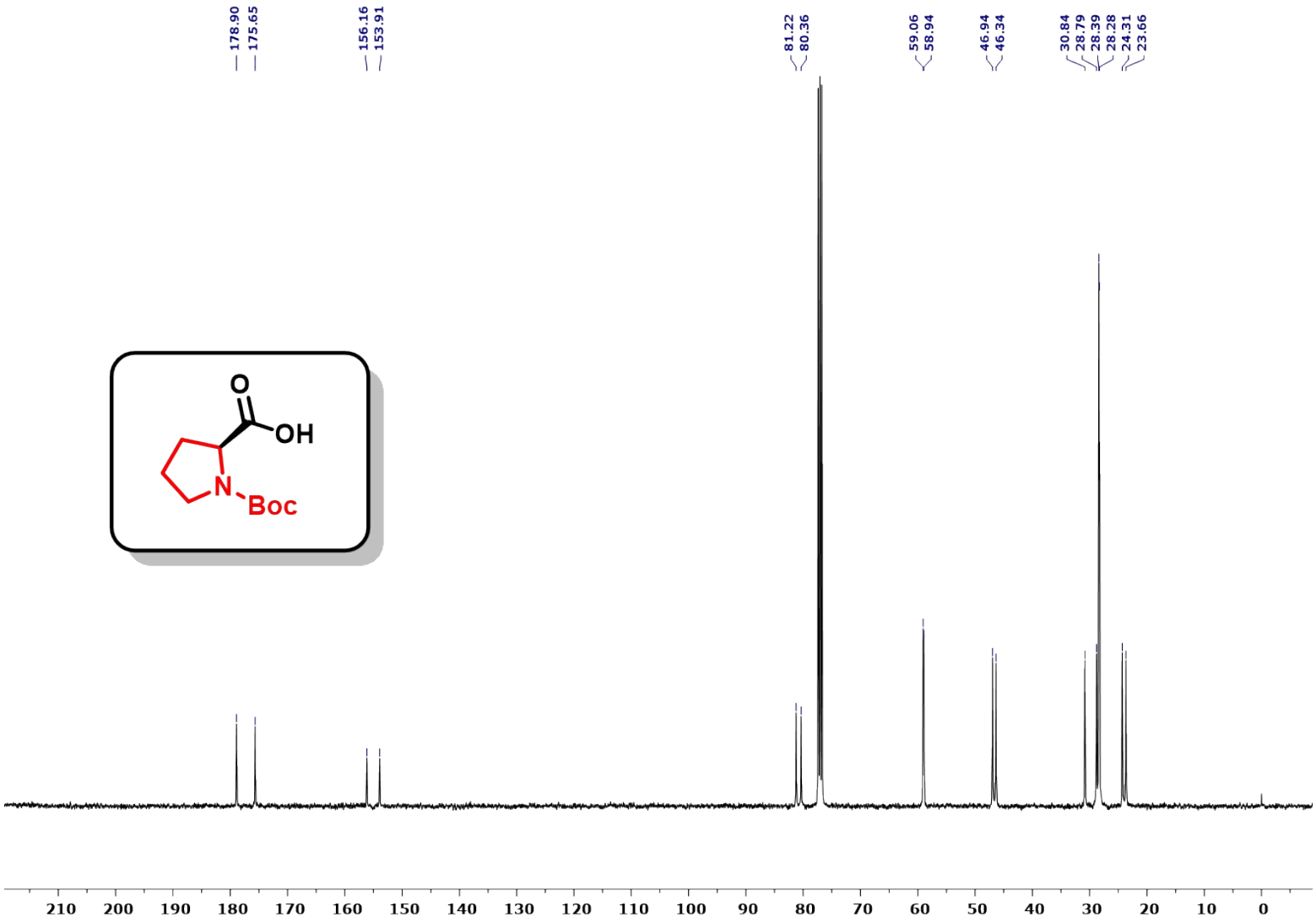


Fig. S19. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of (*tert*-butoxy carbonyl)-L-proline (**2d**).

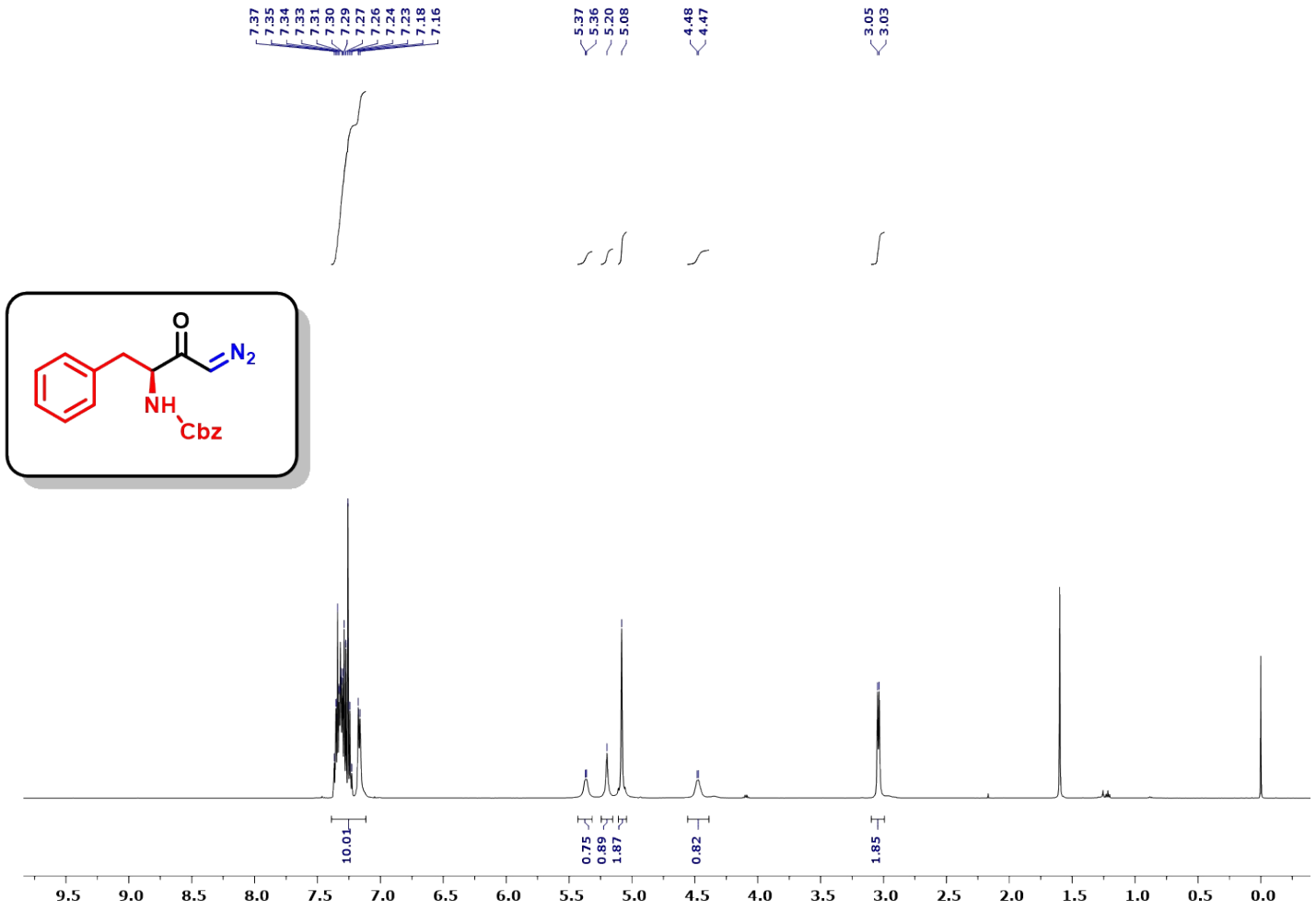


Fig. S20. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of benzyl (*S*)-(4-diazo-3-oxo-1-phenylbutan-2-yl) carbamate (**4a**).

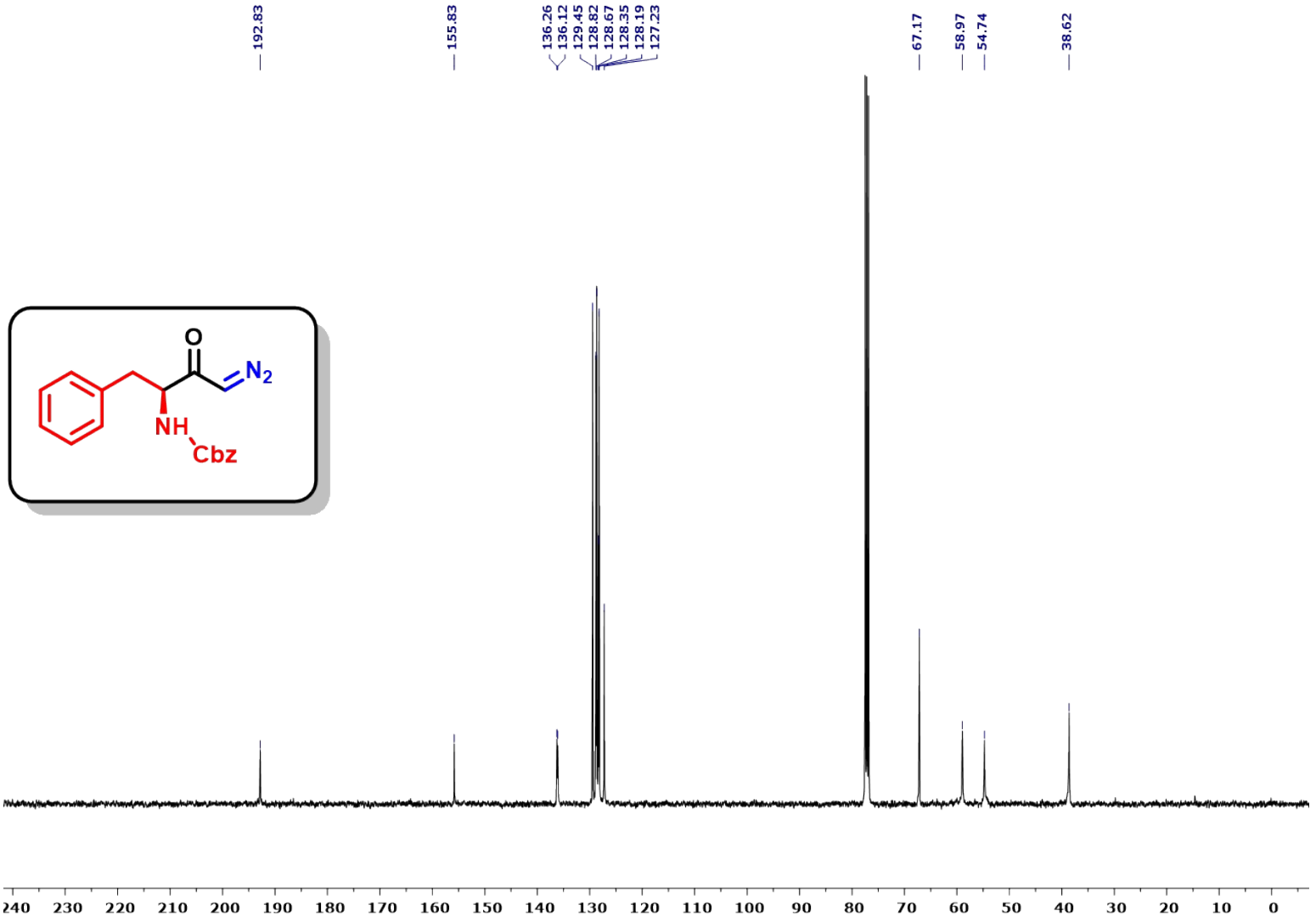


Fig. S21. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of benzyl (*S*)-(4-diazo-3-oxo-1-phenylbutan-2-yl) carbamate (**4a**).

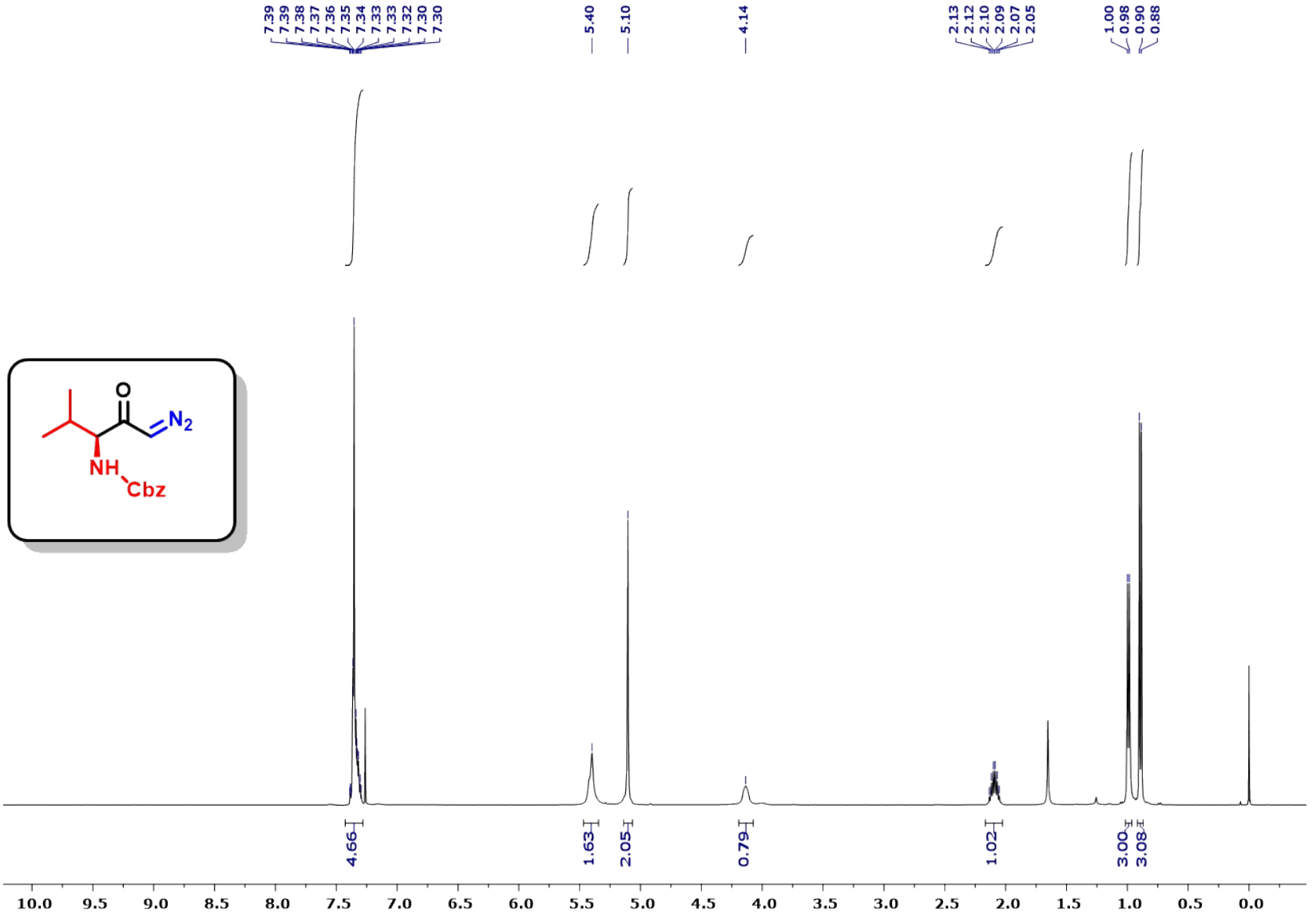


Fig. S22. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of benzyl (*S*)-(1-diazo-4-methyl-2-oxopentan-3-yl) carbamate (**4b**).

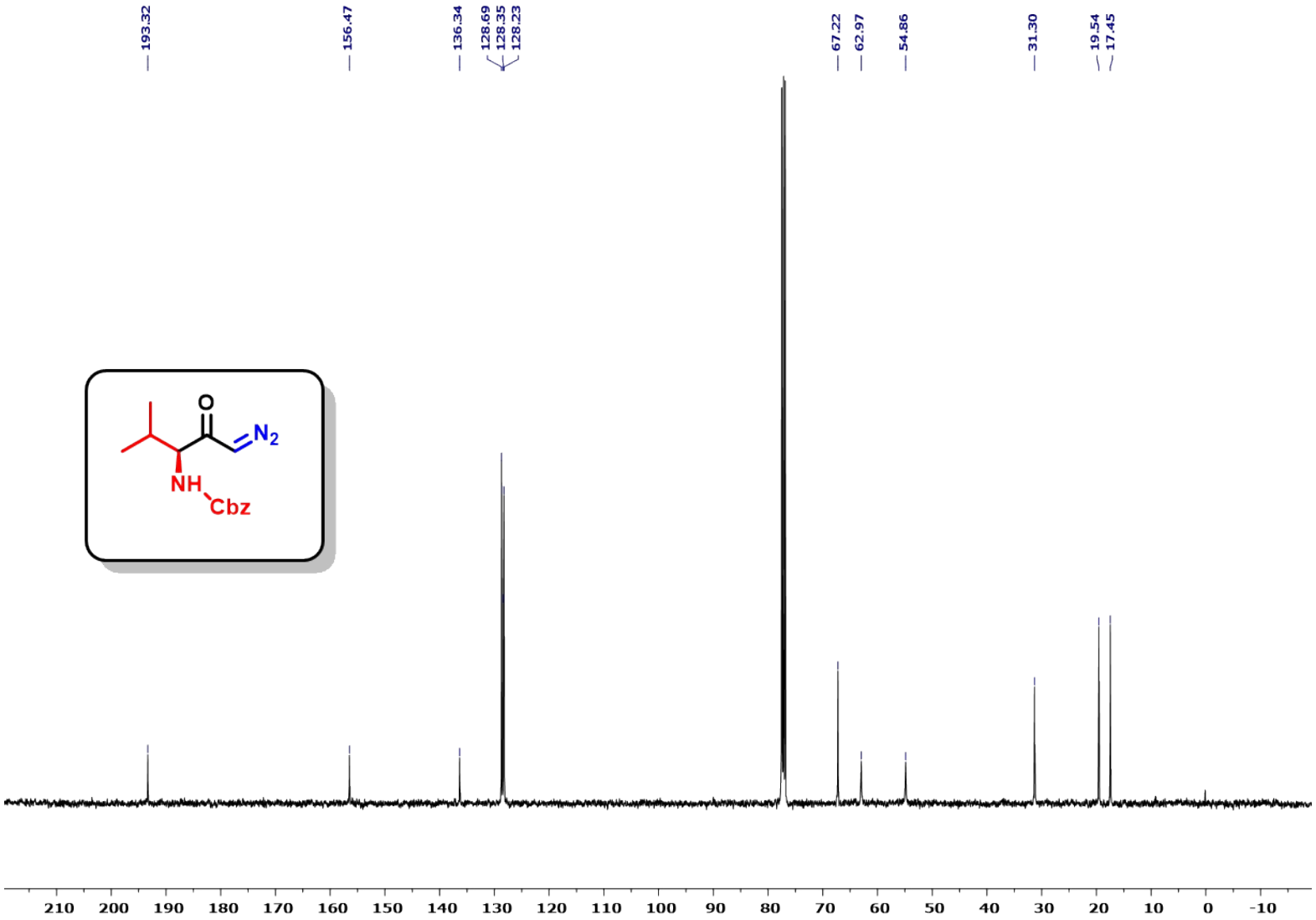


Fig. S23. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of benzyl (*S*)-(1-diazo-4-methyl-2-oxopentan-3-yl) carbamate (**4b**).

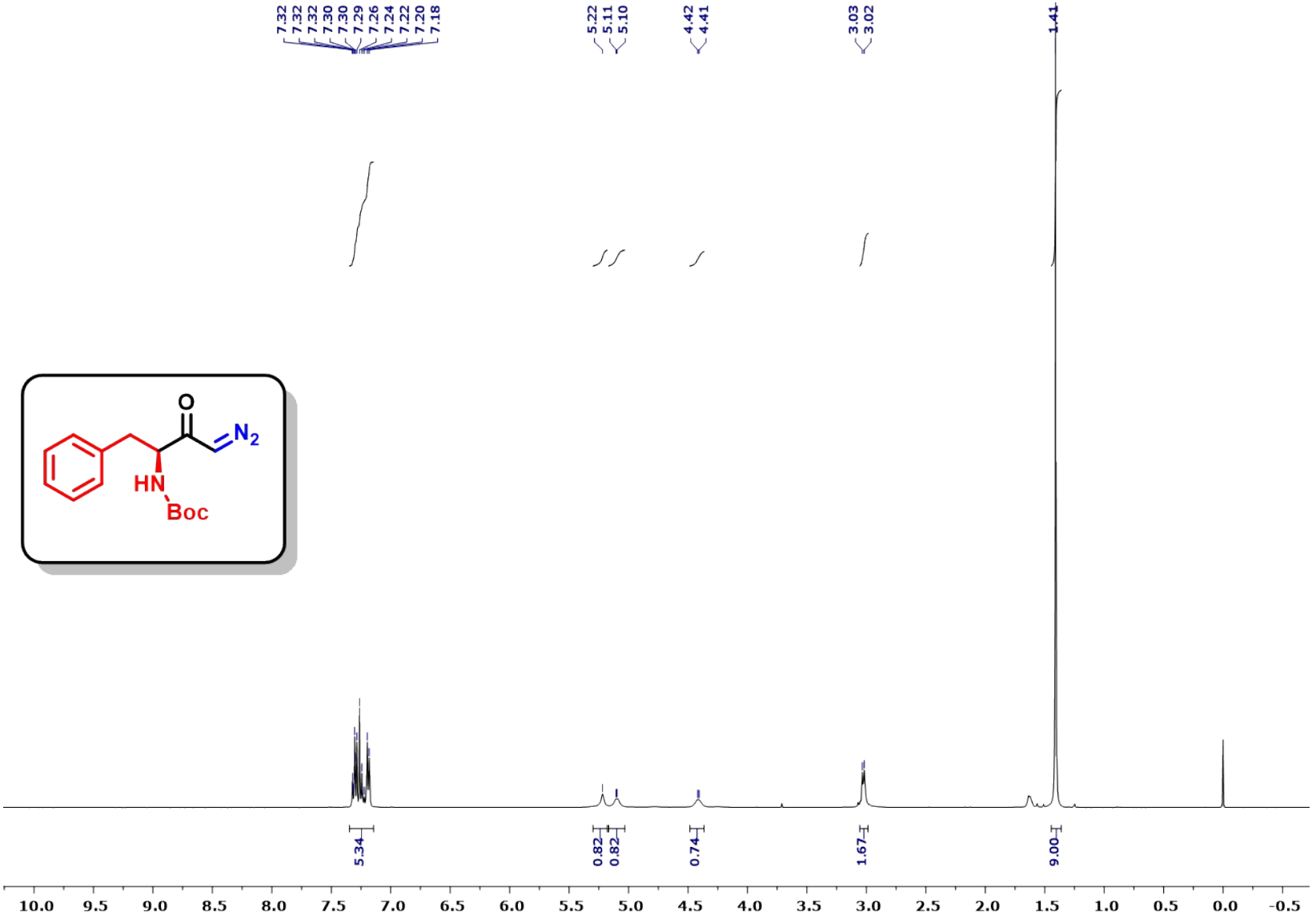


Fig. S24. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of *tert*-butyl (*S*)-(4-diazo-3-oxo-1-phenylbutan-2-yl) carbamate (**4c**).

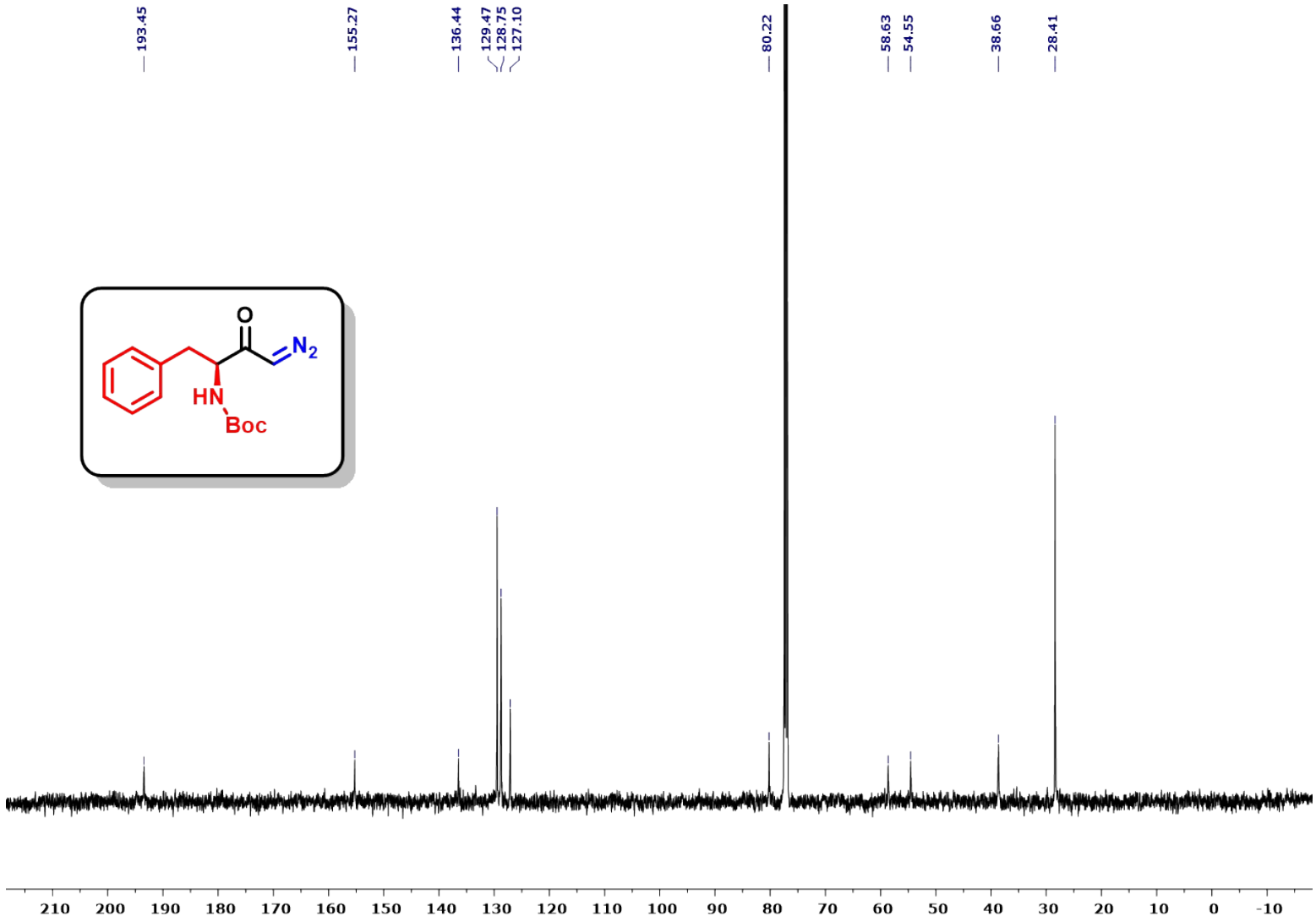


Fig. S25. ^{13}C NMR (125 MHz, $CDCl_3$) spectra of *tert*-butyl (*S*)-(4-diazo-3-oxo-1-phenylbutan-2-yl) carbamate (**4c**).

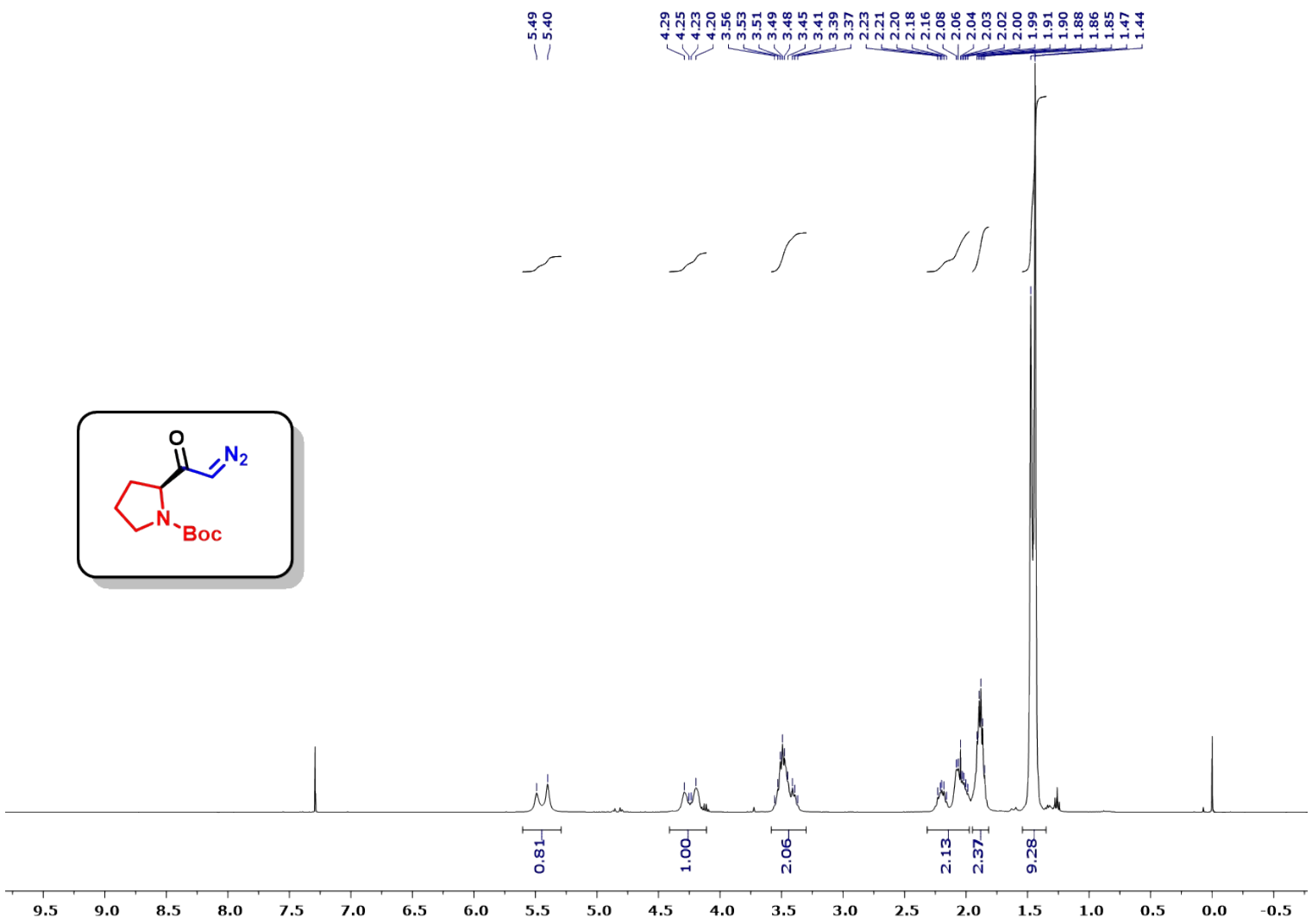


Fig. S26. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of *tert*-butyl (*S*)-2-(2-diazoacetyl) pyrrolidine-1-carboxylate (**4d**).

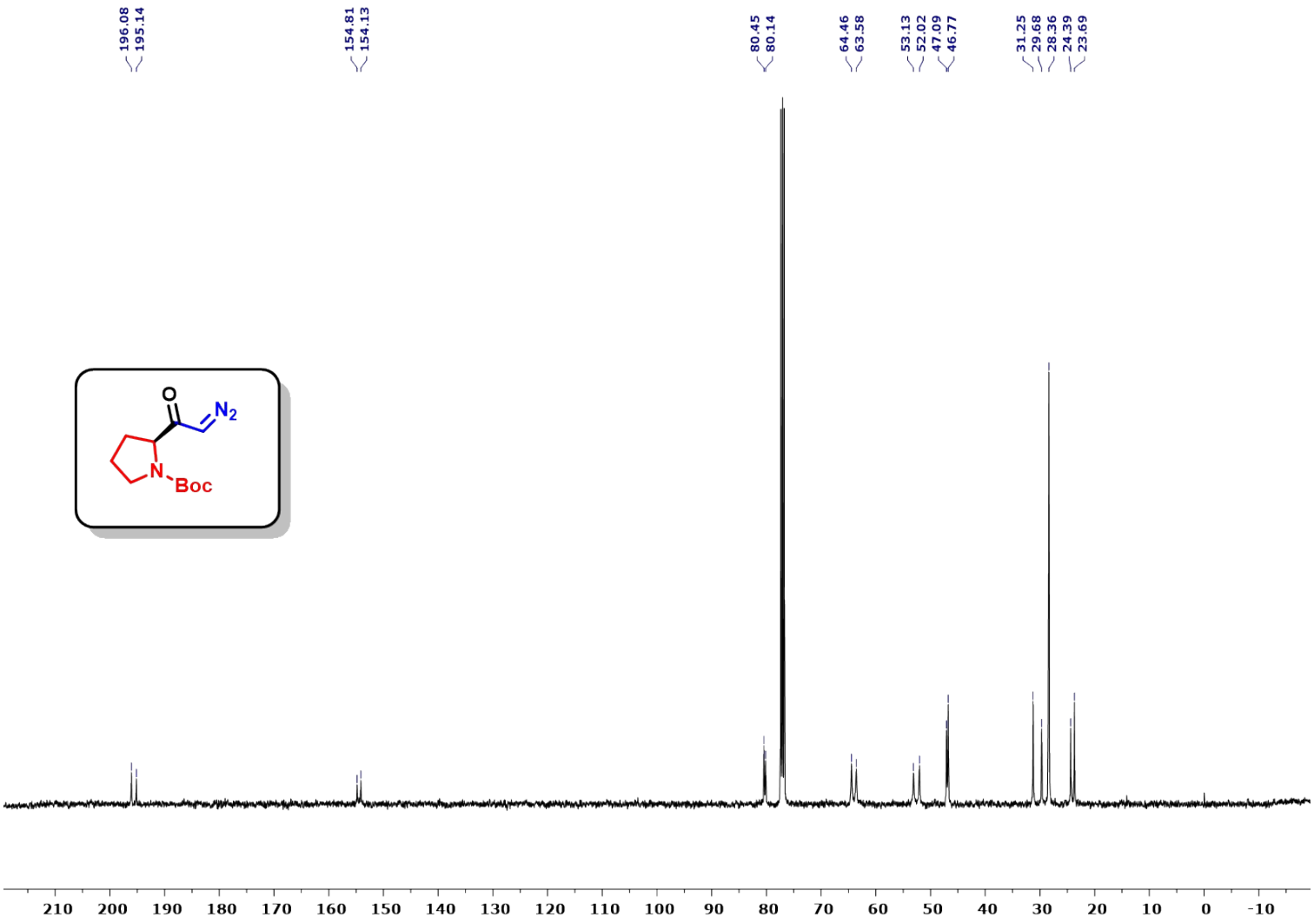


Fig. S27. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of *tert*-butyl (S)-2-(2-diazoacetyl) pyrrolidine-1-carboxylate (**4d**).

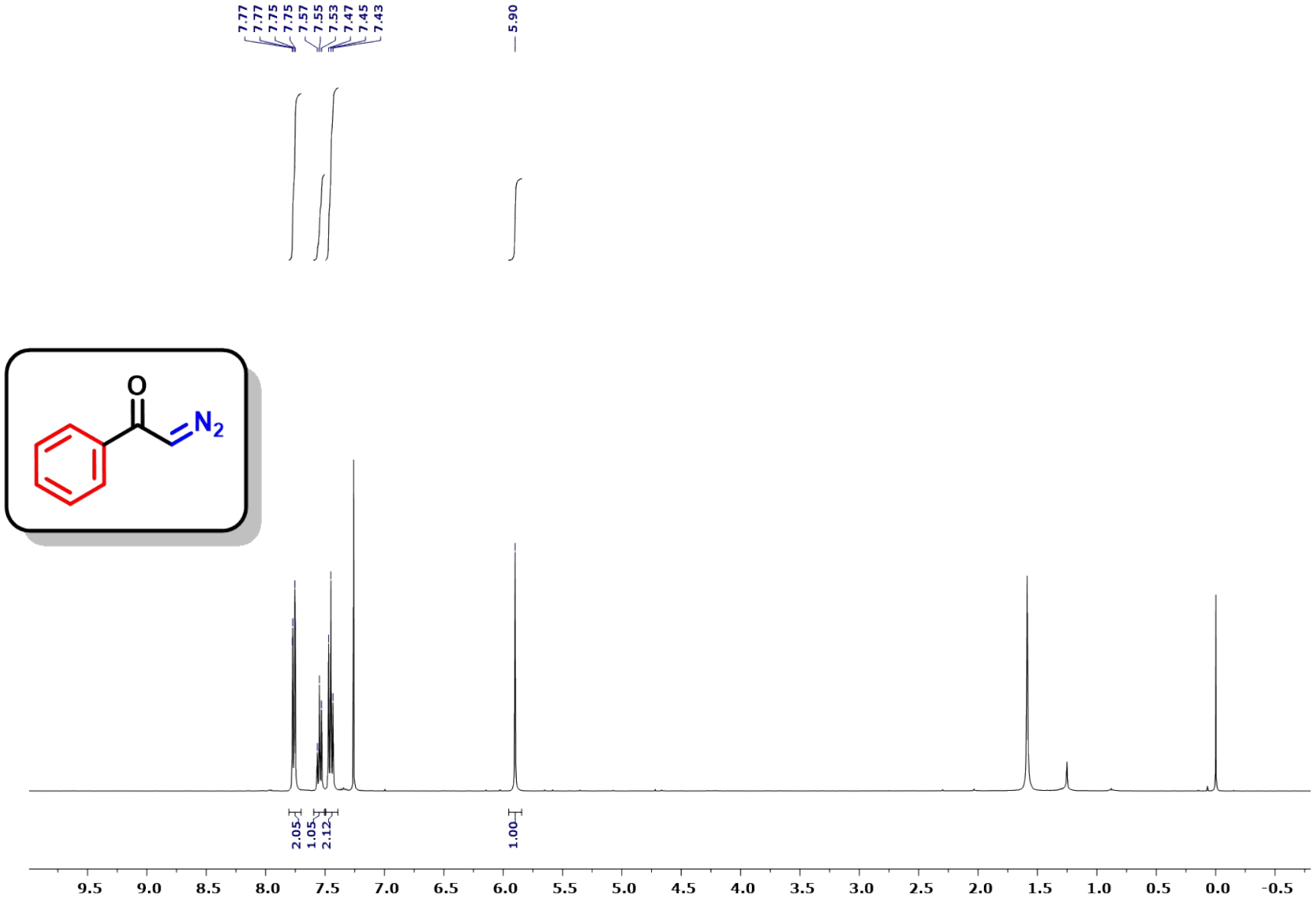


Fig. S28. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of 2-diazo-1-phenylethan-1-one (**4e**).

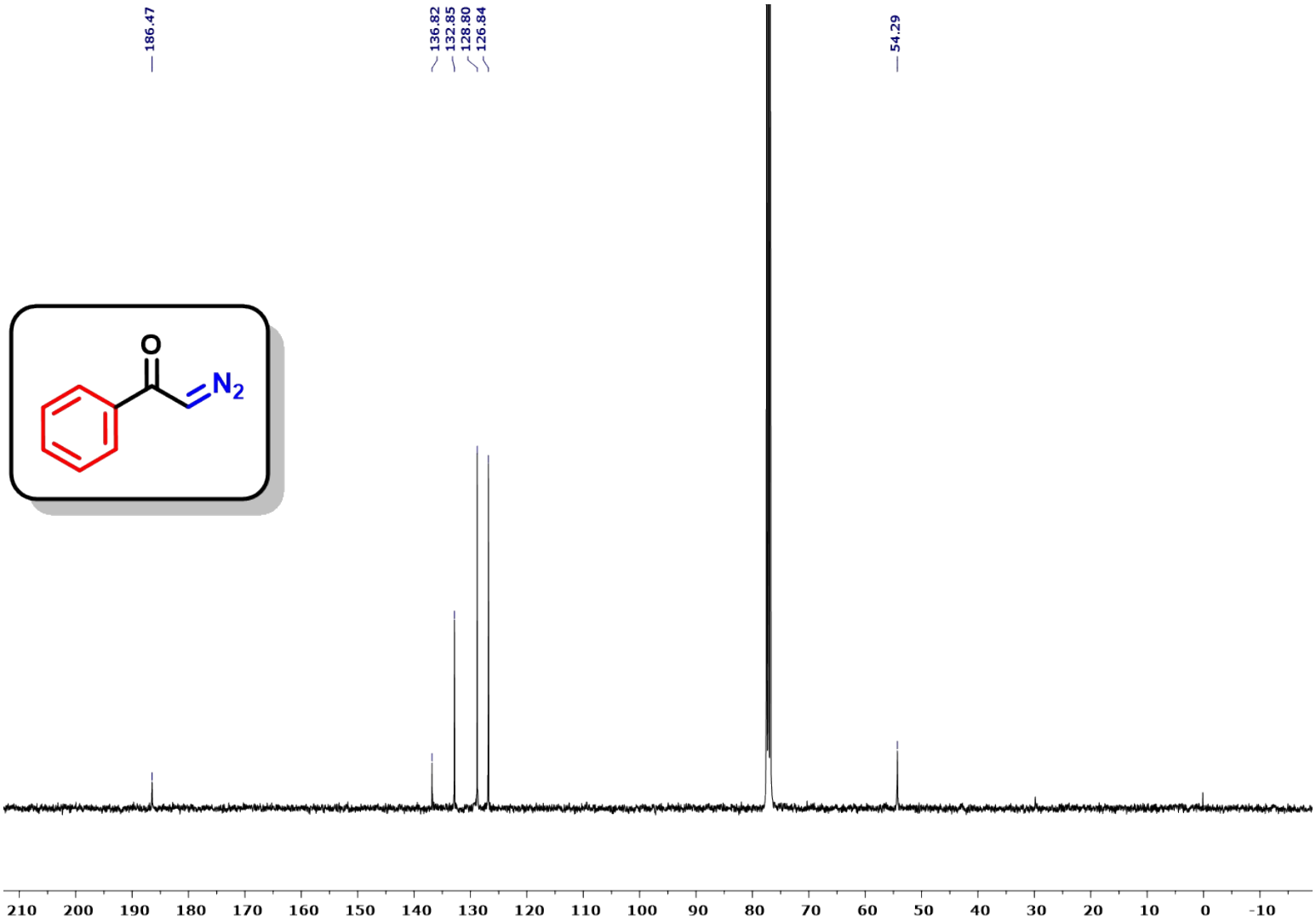


Fig. S29. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of 2-diazo-1-phenylethan-1-one (**4e**).

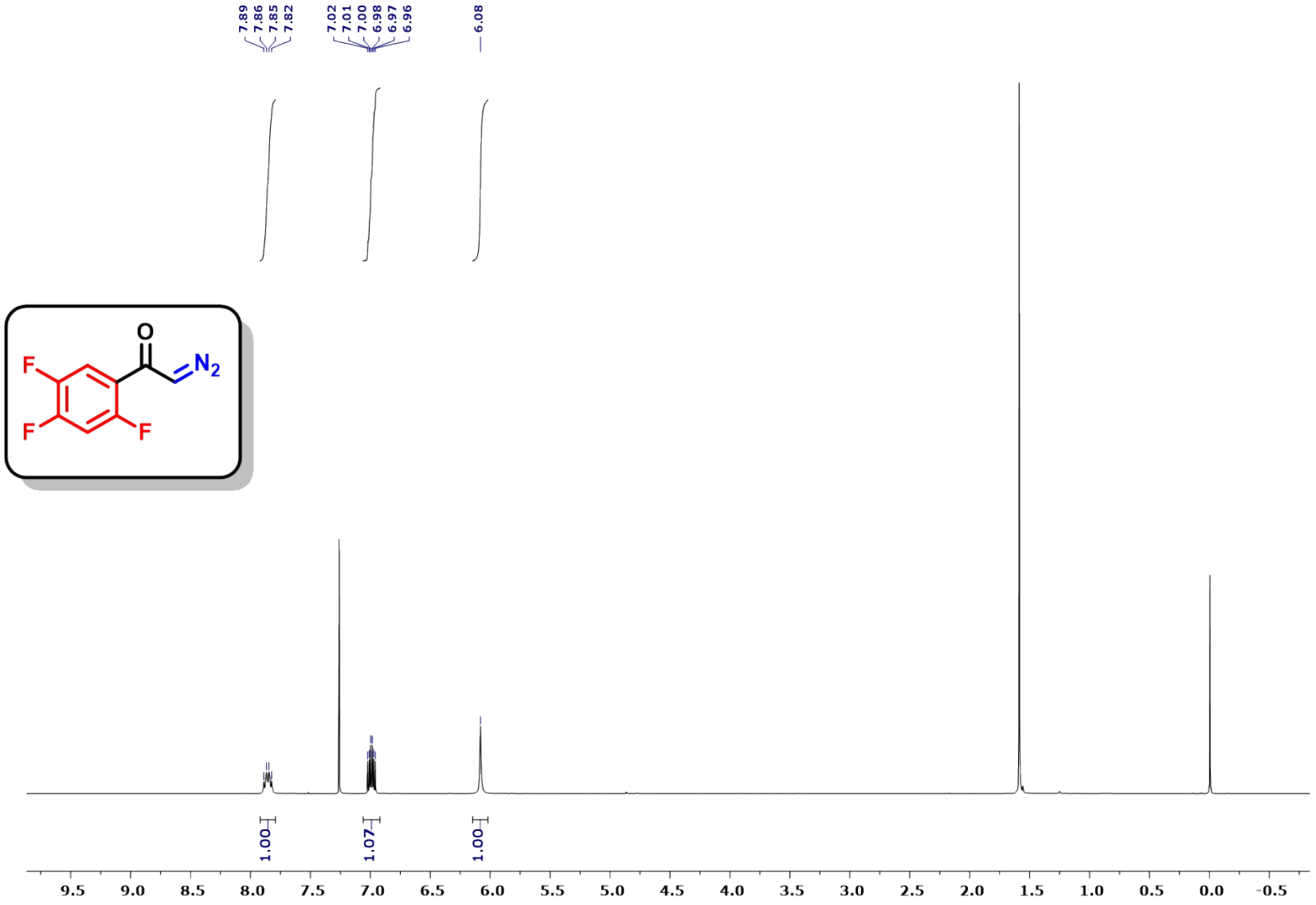


Fig. S30. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of 2-diazo-1-(2,4,5-trifluorophenyl) ethan-1-one (**4f**).

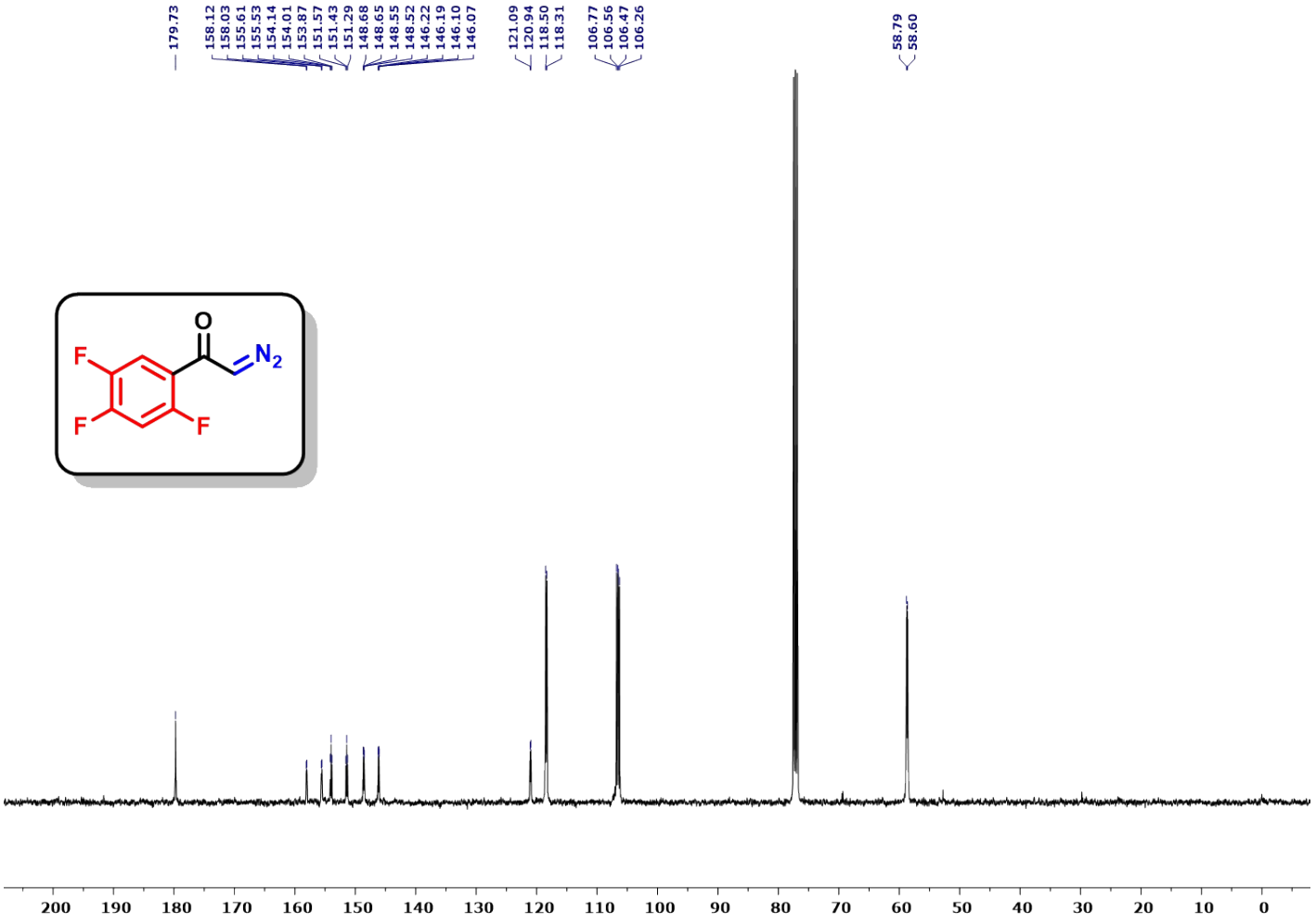


Fig. S31. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of 2-diazo-1-(2,4,5-trifluorophenyl) ethan-1-one (**4f**).

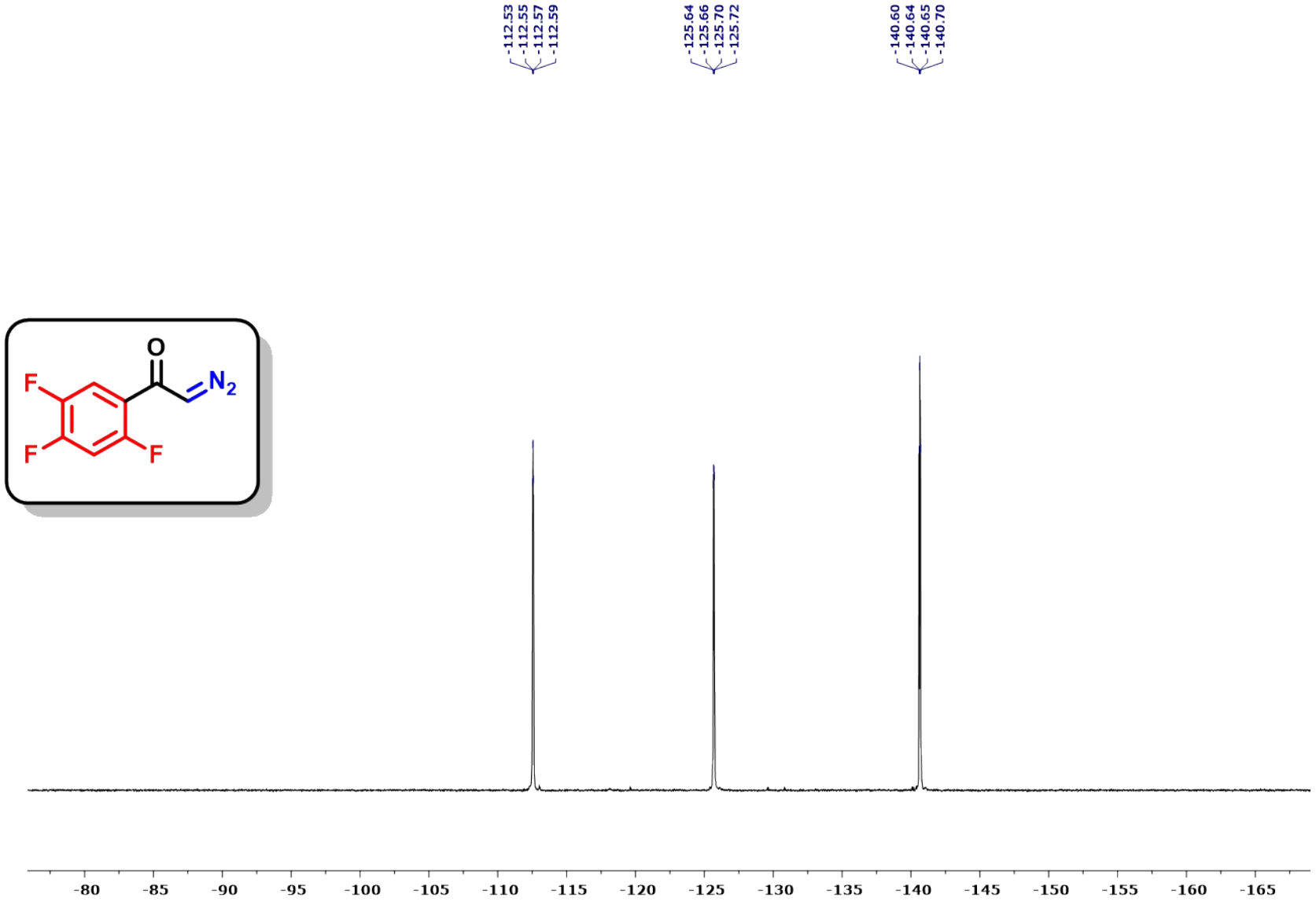


Fig. S32. ^{19}F NMR (377 MHz, $CDCl_3$) spectra of 2-Diazo-1-(2,4,5-trifluorophenyl) ethan-1-one (**4f**).

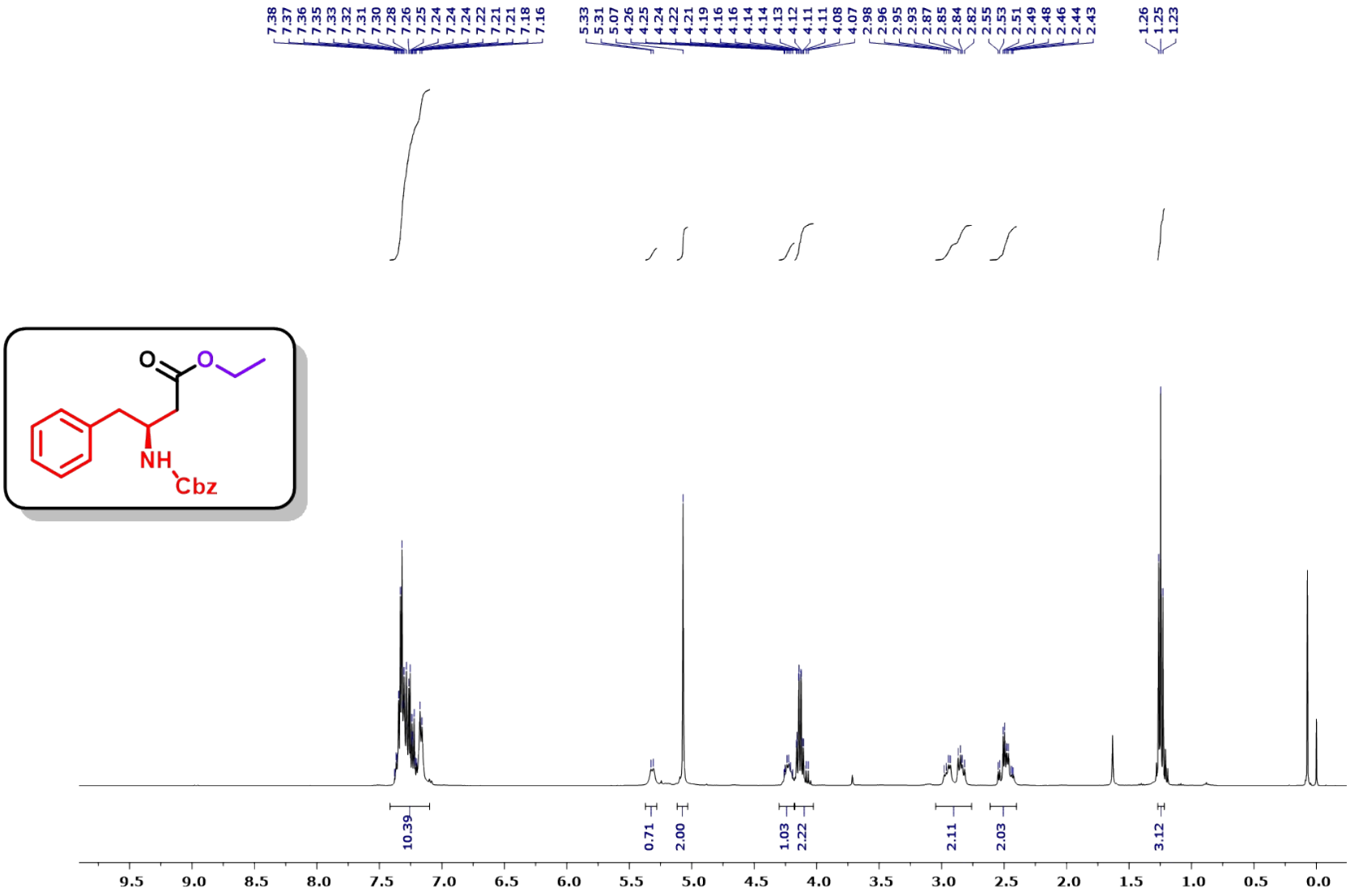


Fig. S33. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of ethyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (**6aa**).

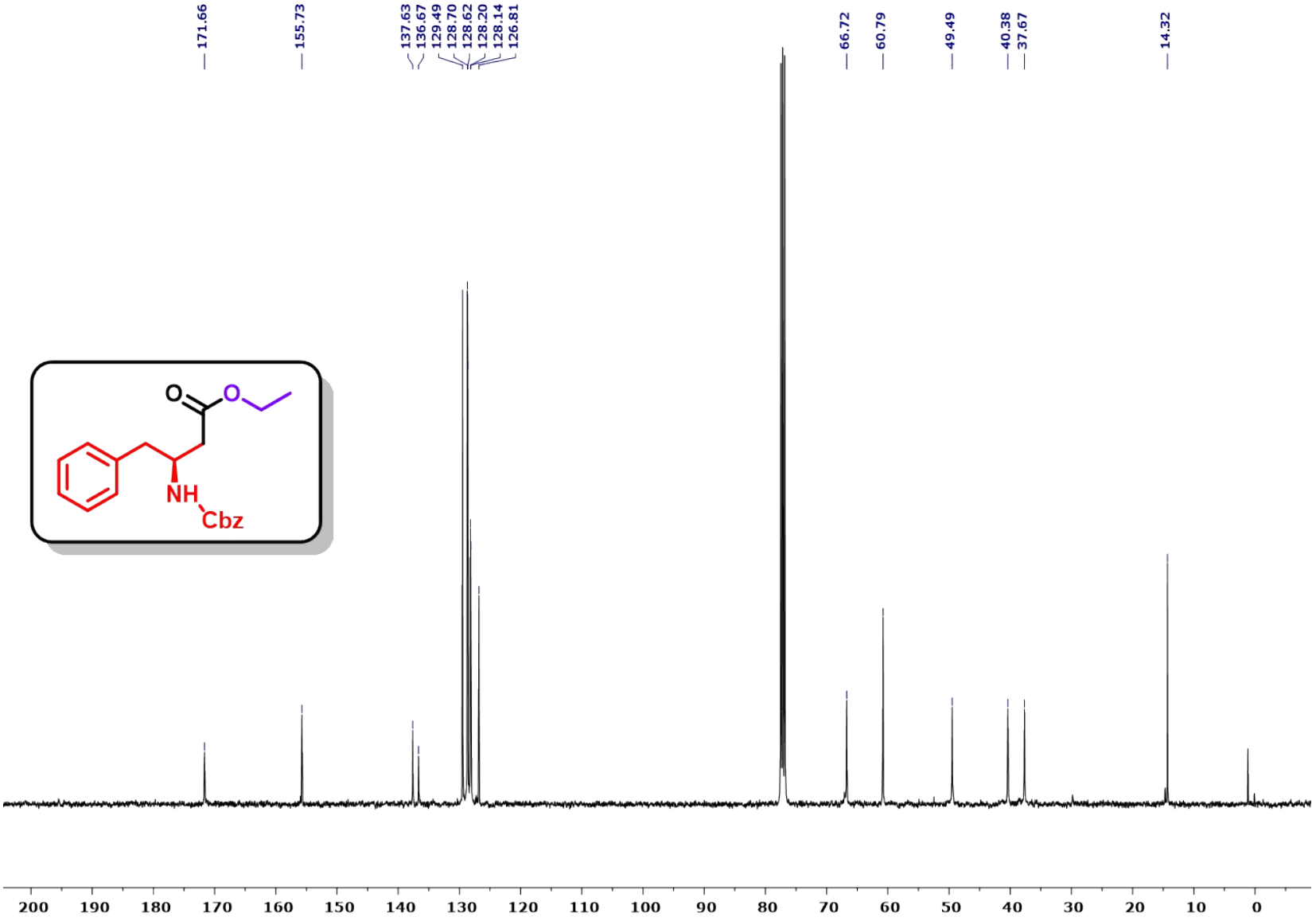


Fig. S34. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of ethyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (**6aa**).

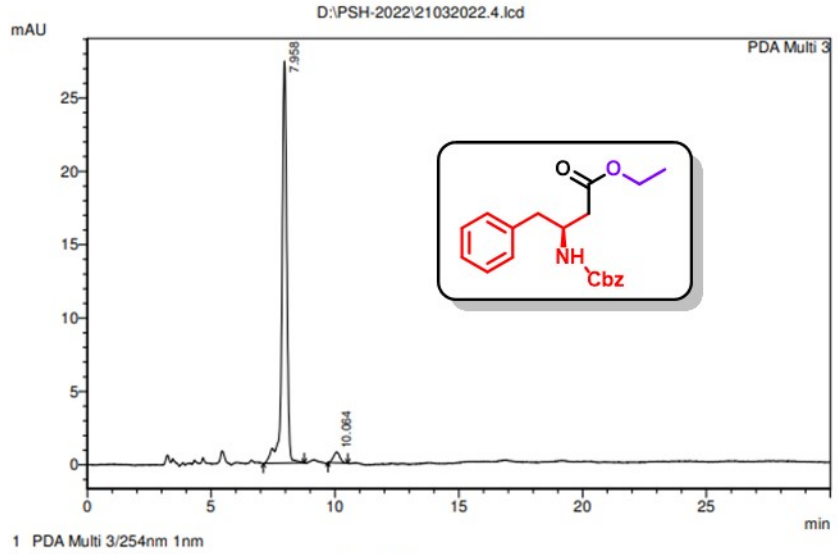
==== Shimadzu LCsolution Analysis Report ====

D:\PSH-2022\21032022.4.lcd

Acquired by	: Manjula
Sample Name	: PSH-AB-341
Sample ID	: PSH-AB-341
Tray#	: 1
Vail #	: 74
Injection Volume	: 10 uL
Data File Name	: 21032022.4.lcd
Method File Name	: 21032022.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 3/21/2022 4:15:19 PM
Data Processed	: 3/21/2022 4:45:21 PM

COLUMN: CHIRALPAK IC 250 X 4.6mm 5u
MOBILE PHASE: 20% IPA IN HEXANE
FLOW RATE : 1.0 ml/min

<Chromatogram>



1 PDA Multi 3/254nm 1nm

PeakTable

PDA Ch3 254nm 1nm

Peak#	Ret. Time	Area	Height	Area %	Height %
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2	10.064	12733	743	3.299	2.642
Total		386022	28133	100.000	100.000

Fig. S35. HPLC data of ethyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate **(6aa).**

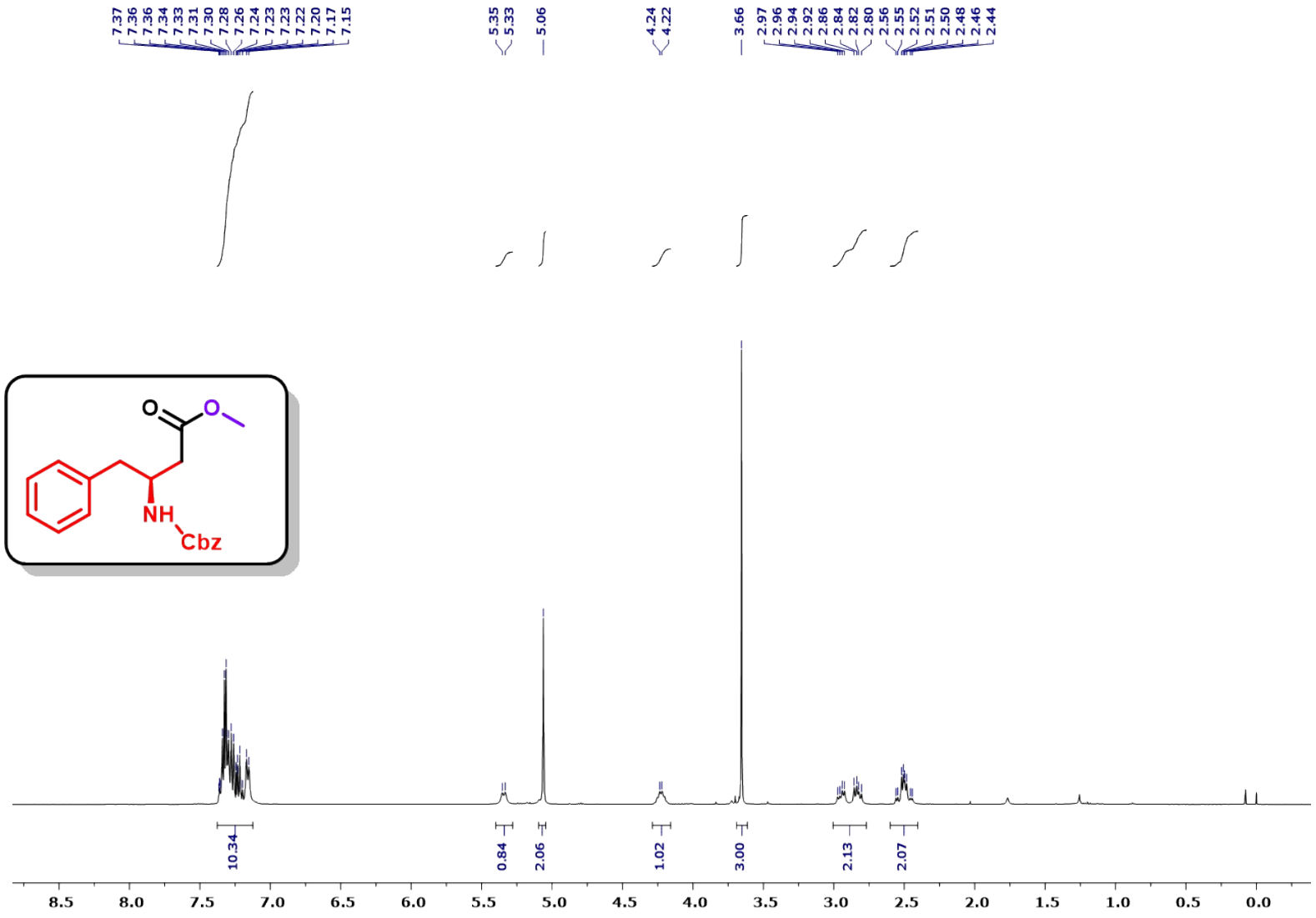

Fig. S36. ^{1}H NMR (400 MHz, $CDCl_3$) of methyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (**6ab**).

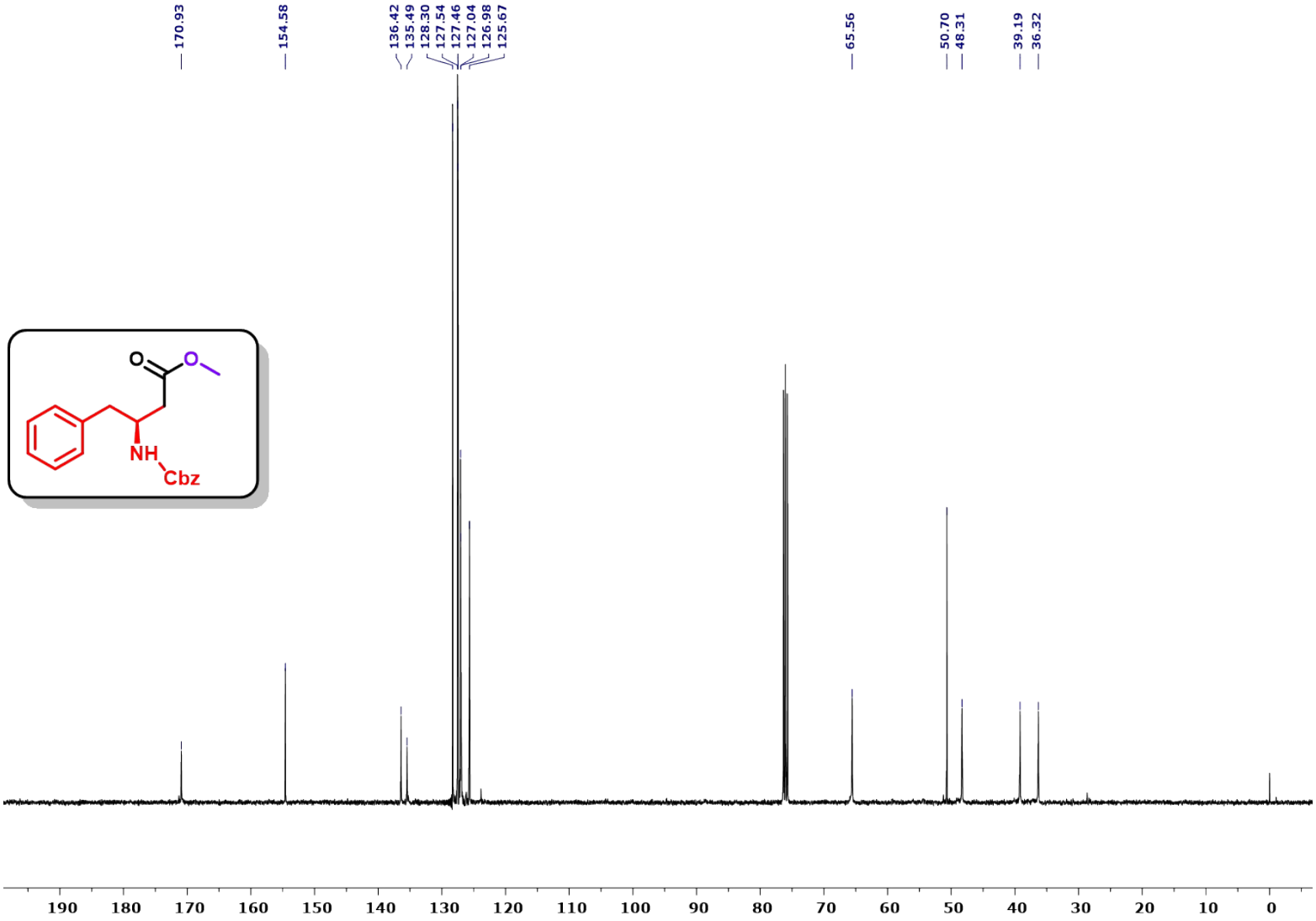

Fig. S37. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of methyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (**6ab**).

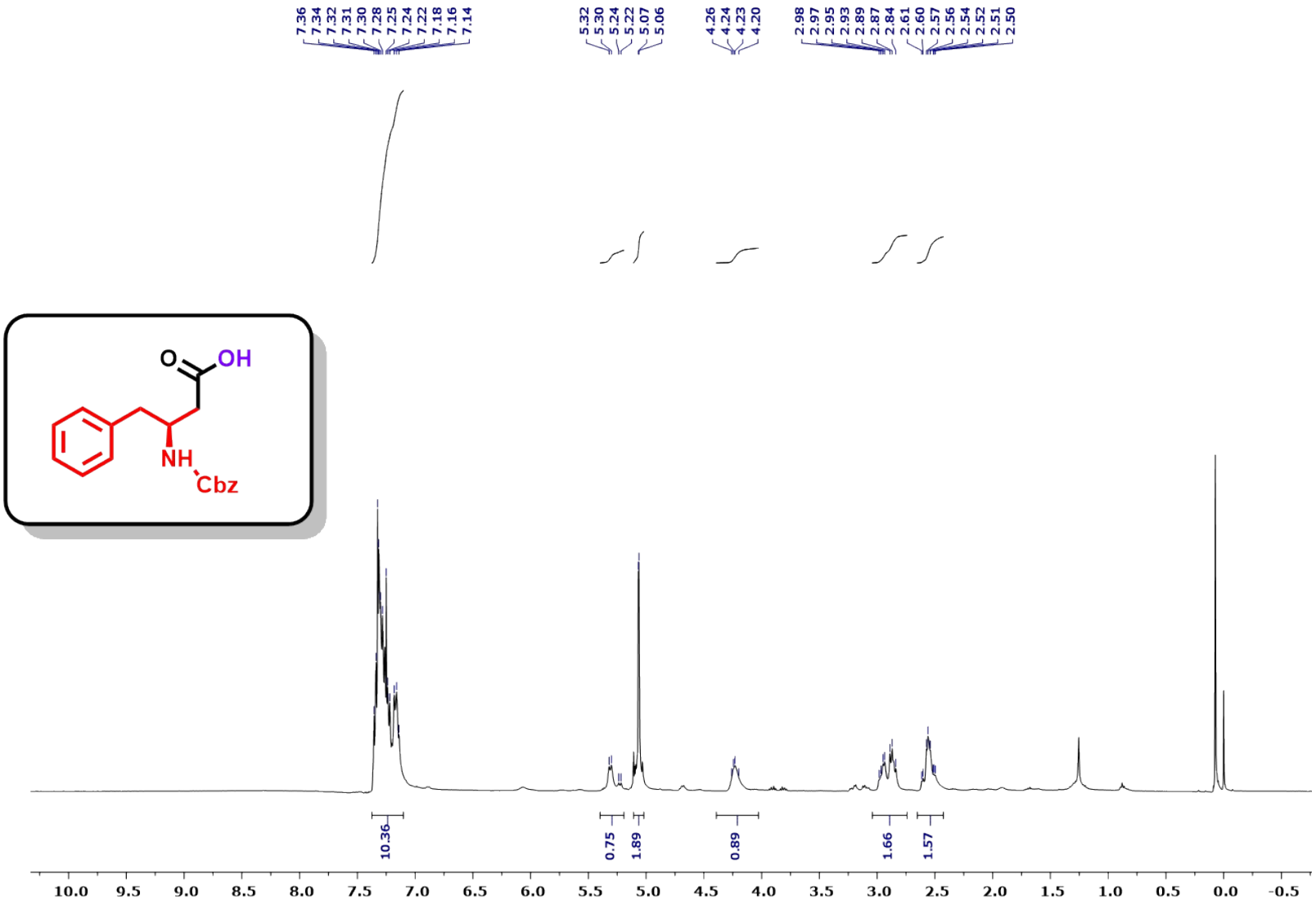

Fig. S38. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoic acid (**6ac**).

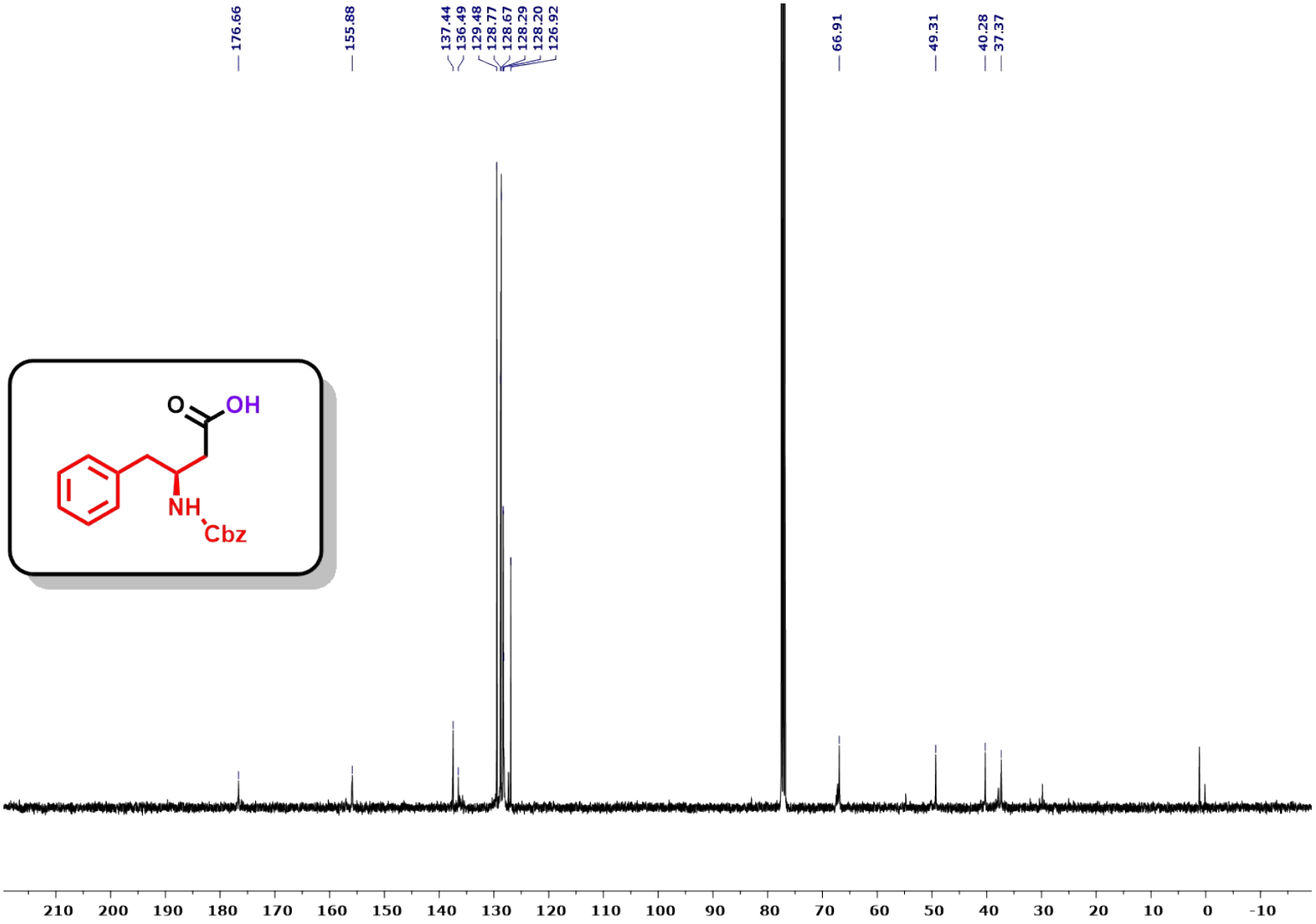

Fig. S39. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoic acid (**6ac**).

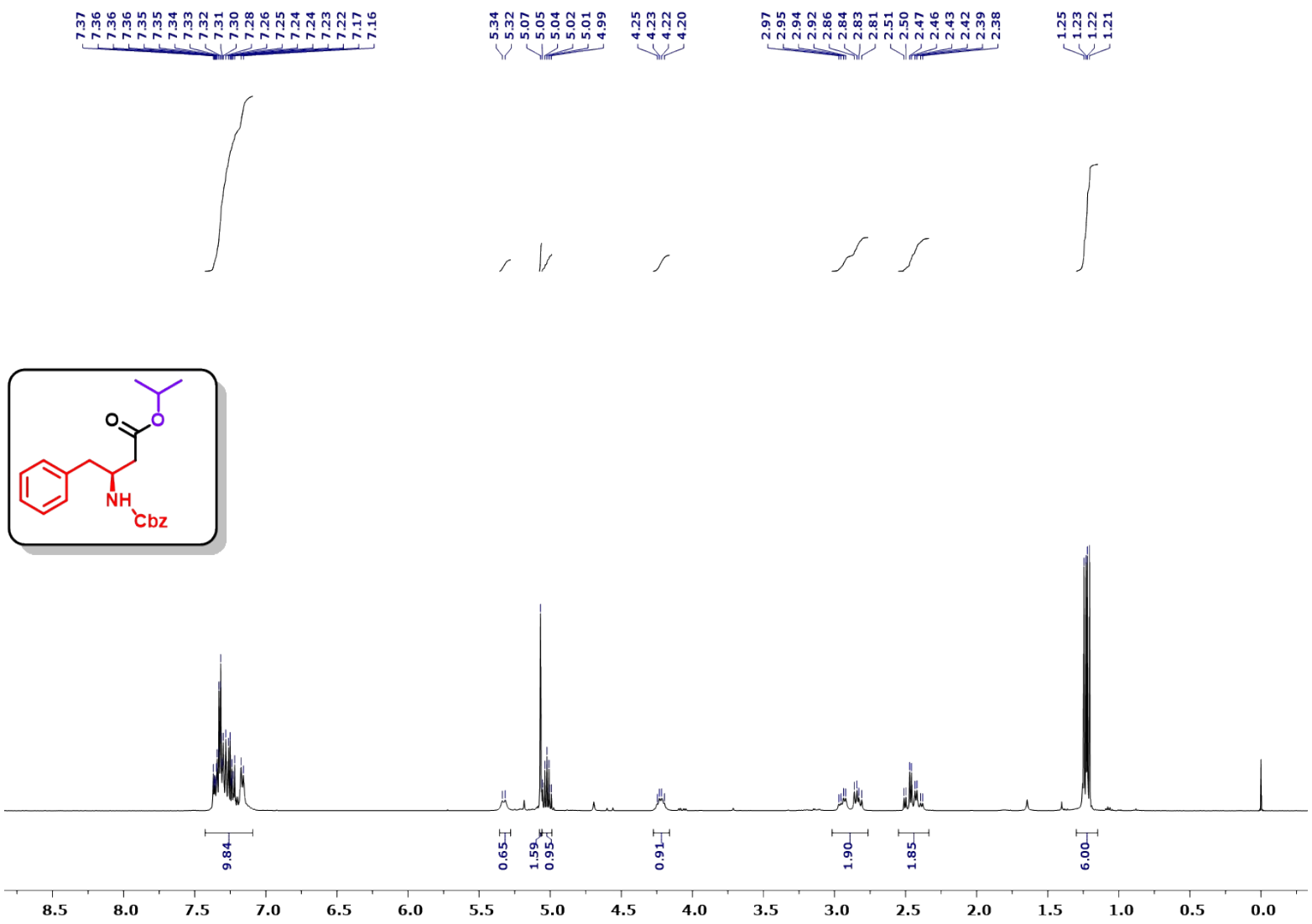

Fig. S40. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of isopropyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (**6ad**).

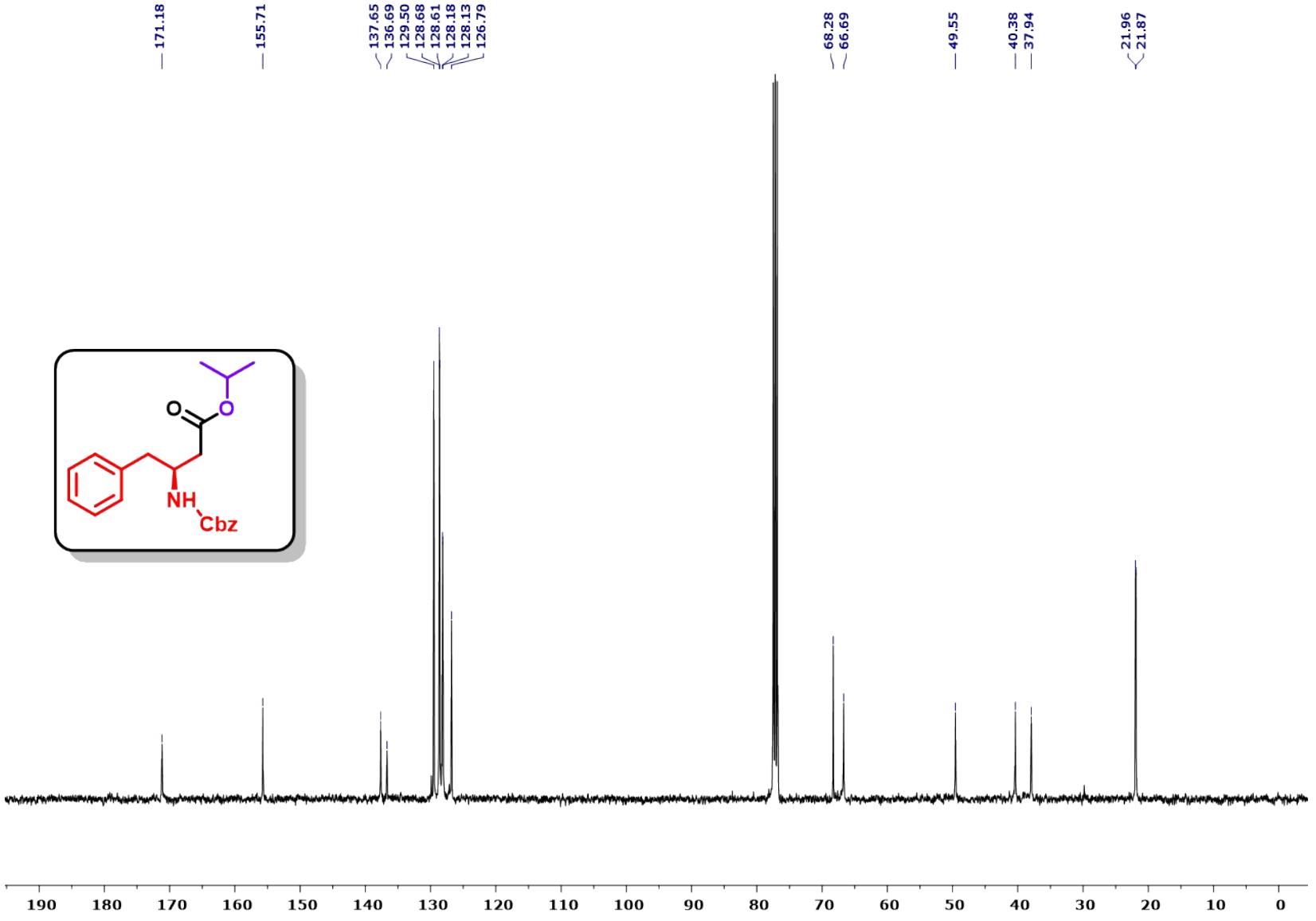

Fig. S41. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of Isopropyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (**6ad**).

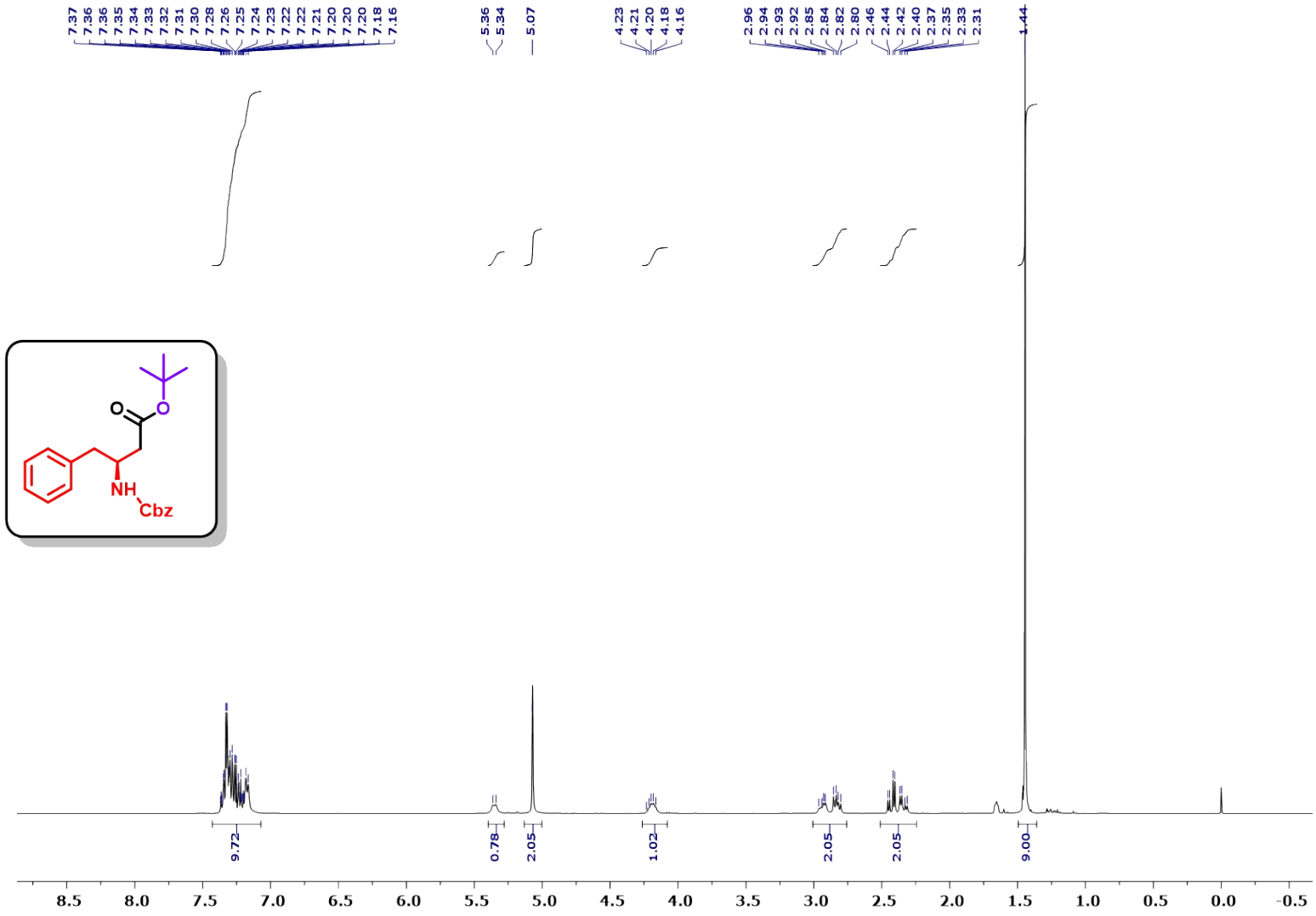

Fig. S42. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of *tert*-butyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (**6ae**).

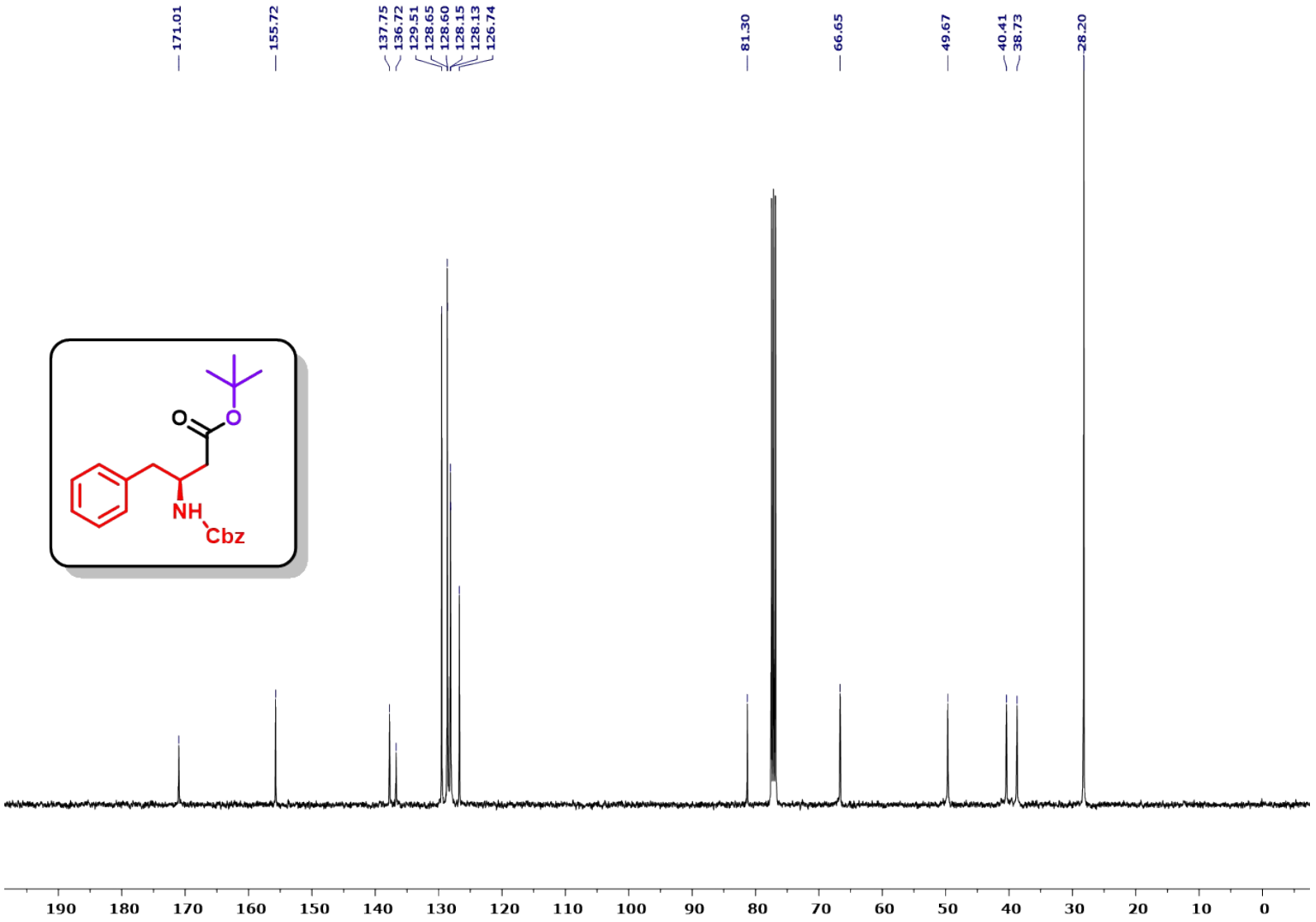

Fig. S43. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of *tert*-butyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (**6ae**).

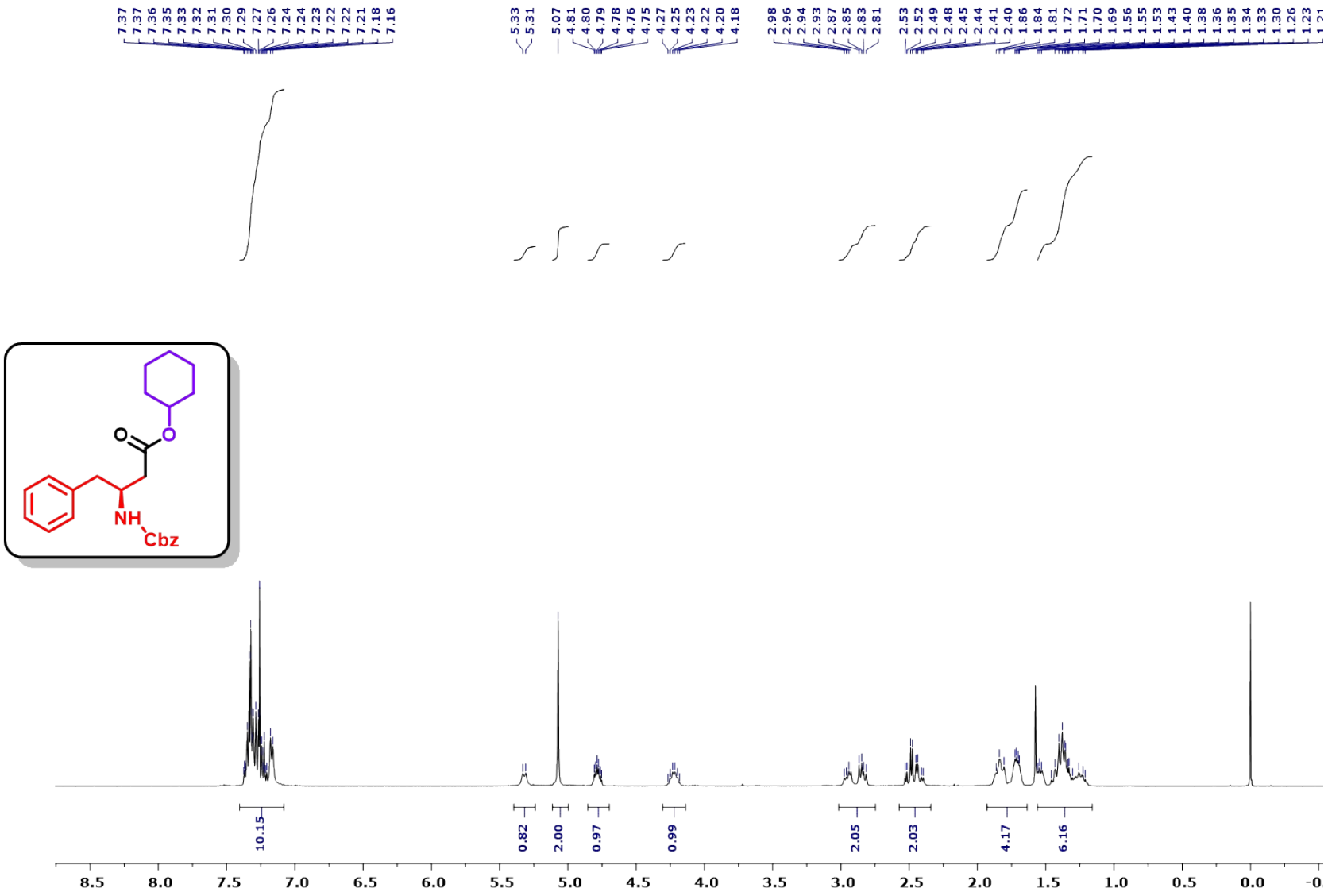

Fig. S44. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of cyclohexyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (**6af**).

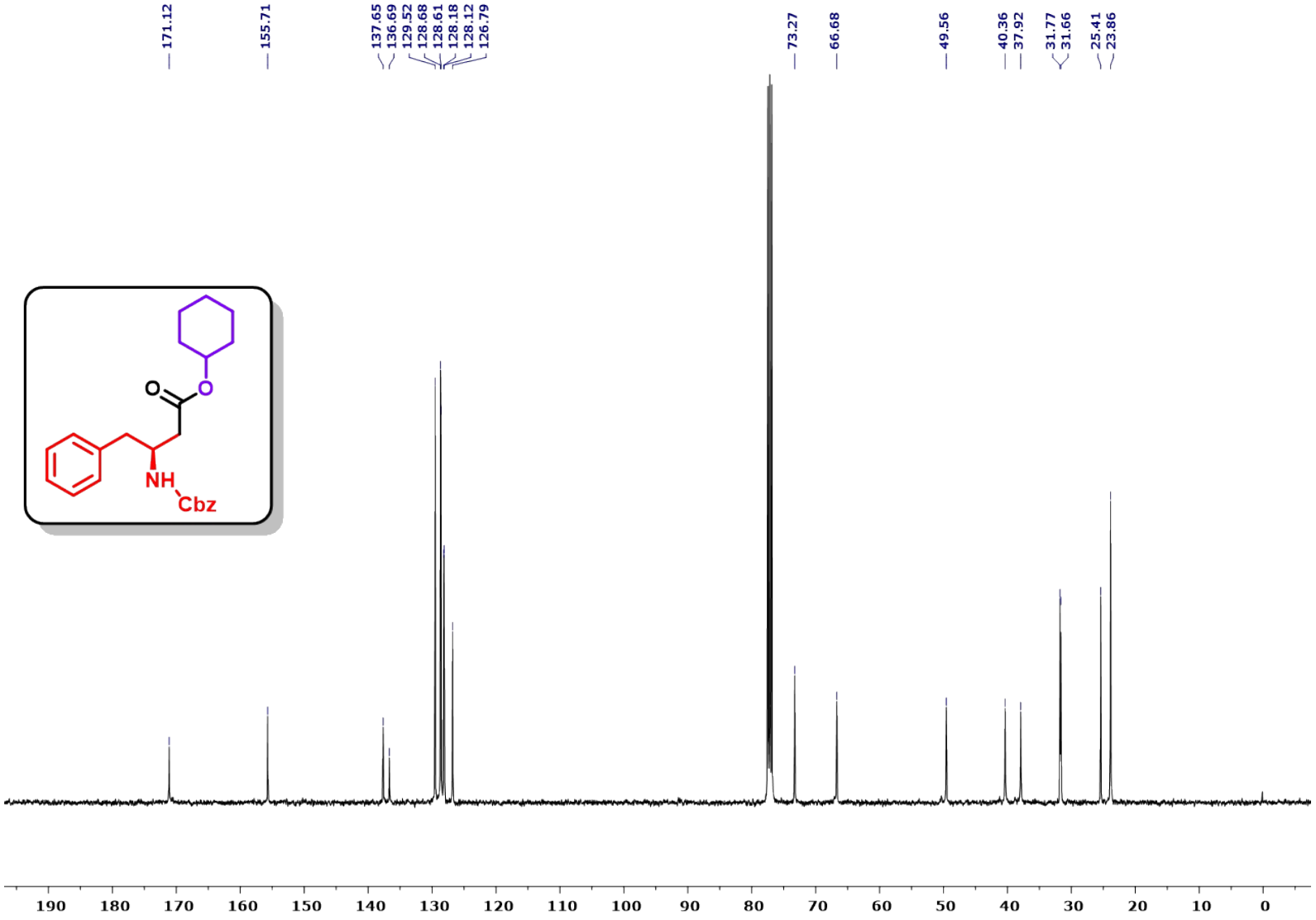

Fig. S45. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of cyclohexyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (**6af**).

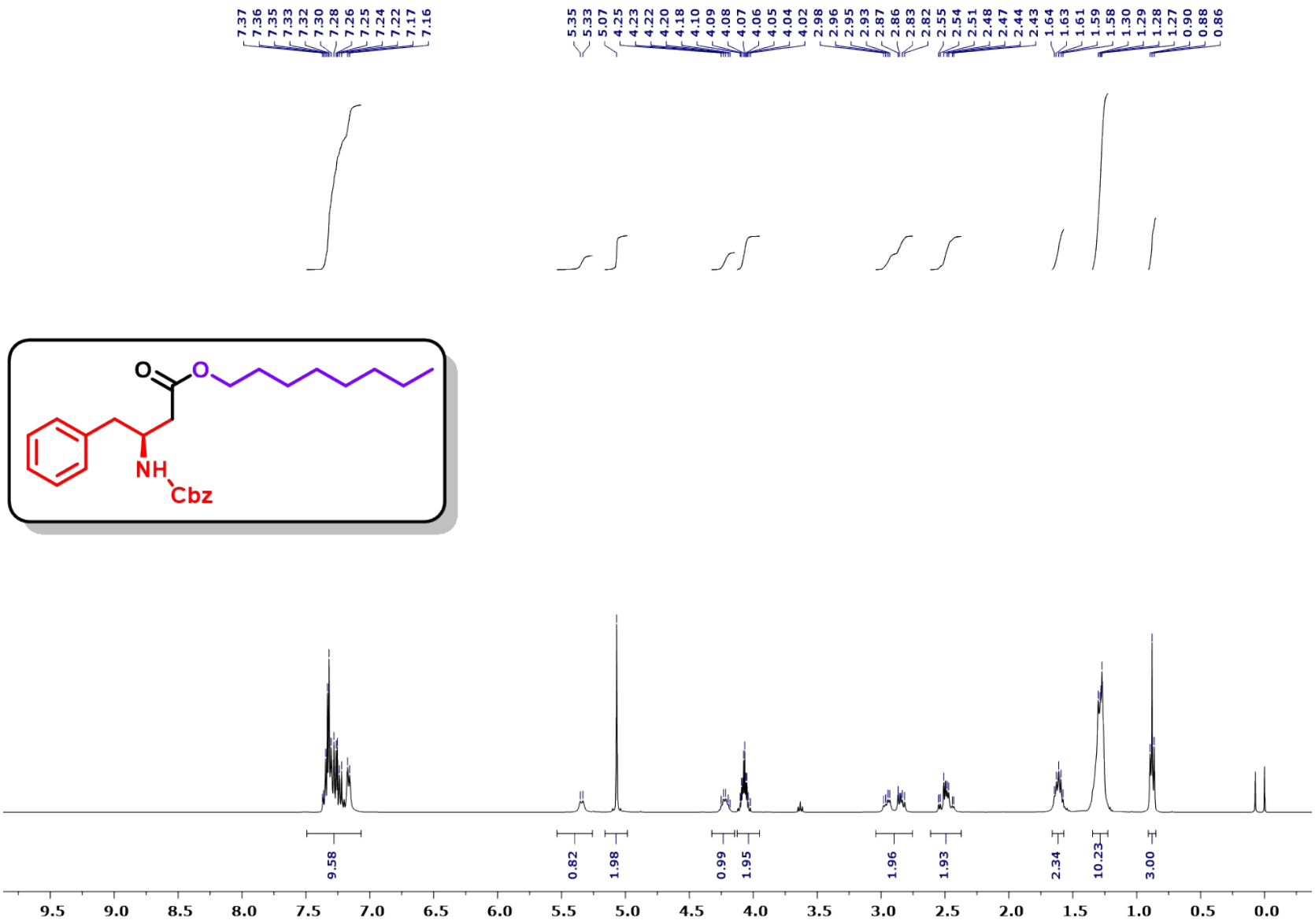

Fig. S46. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of octyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (**6ag**).

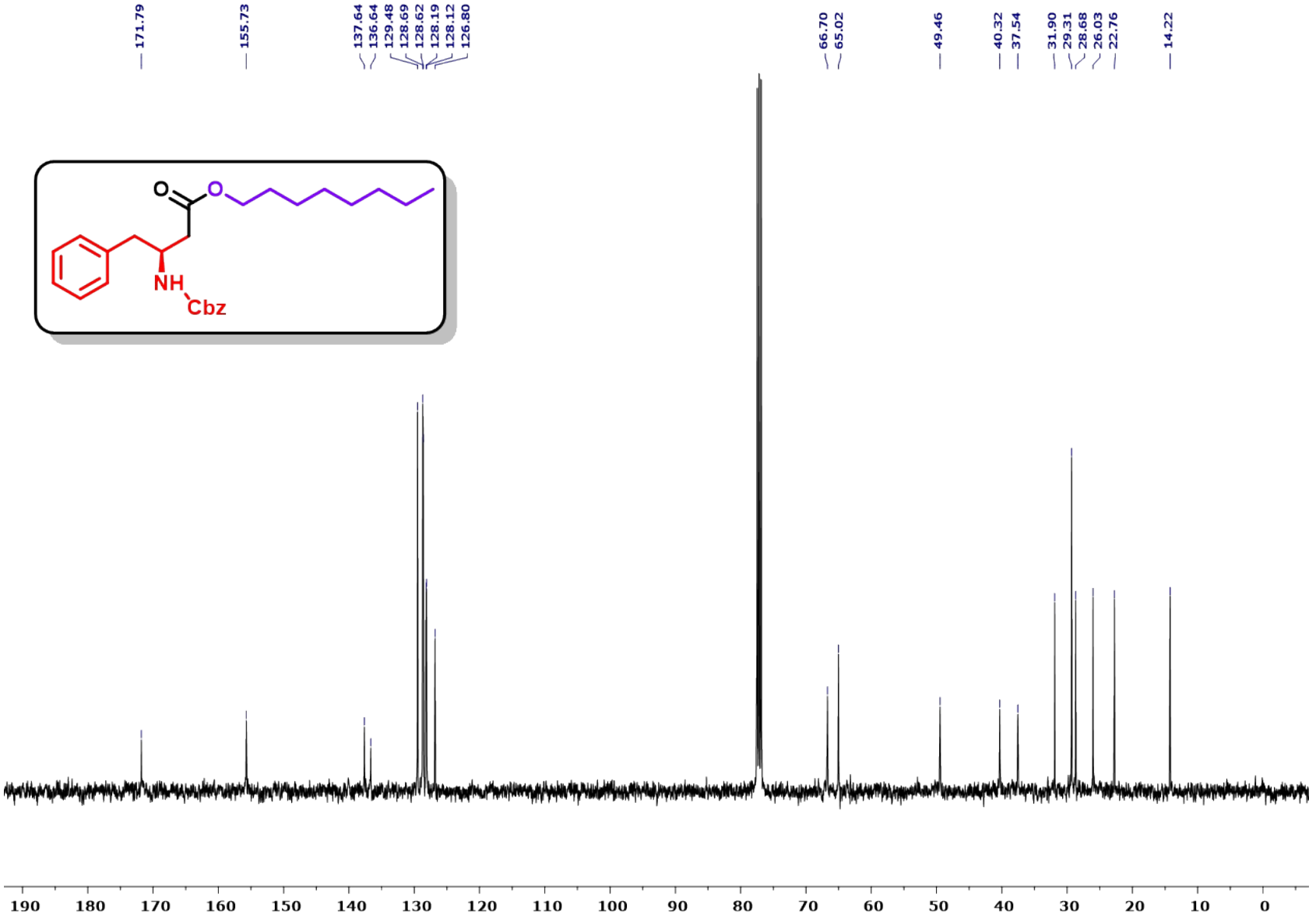

Fig. S47. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of octyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (**6ag**).

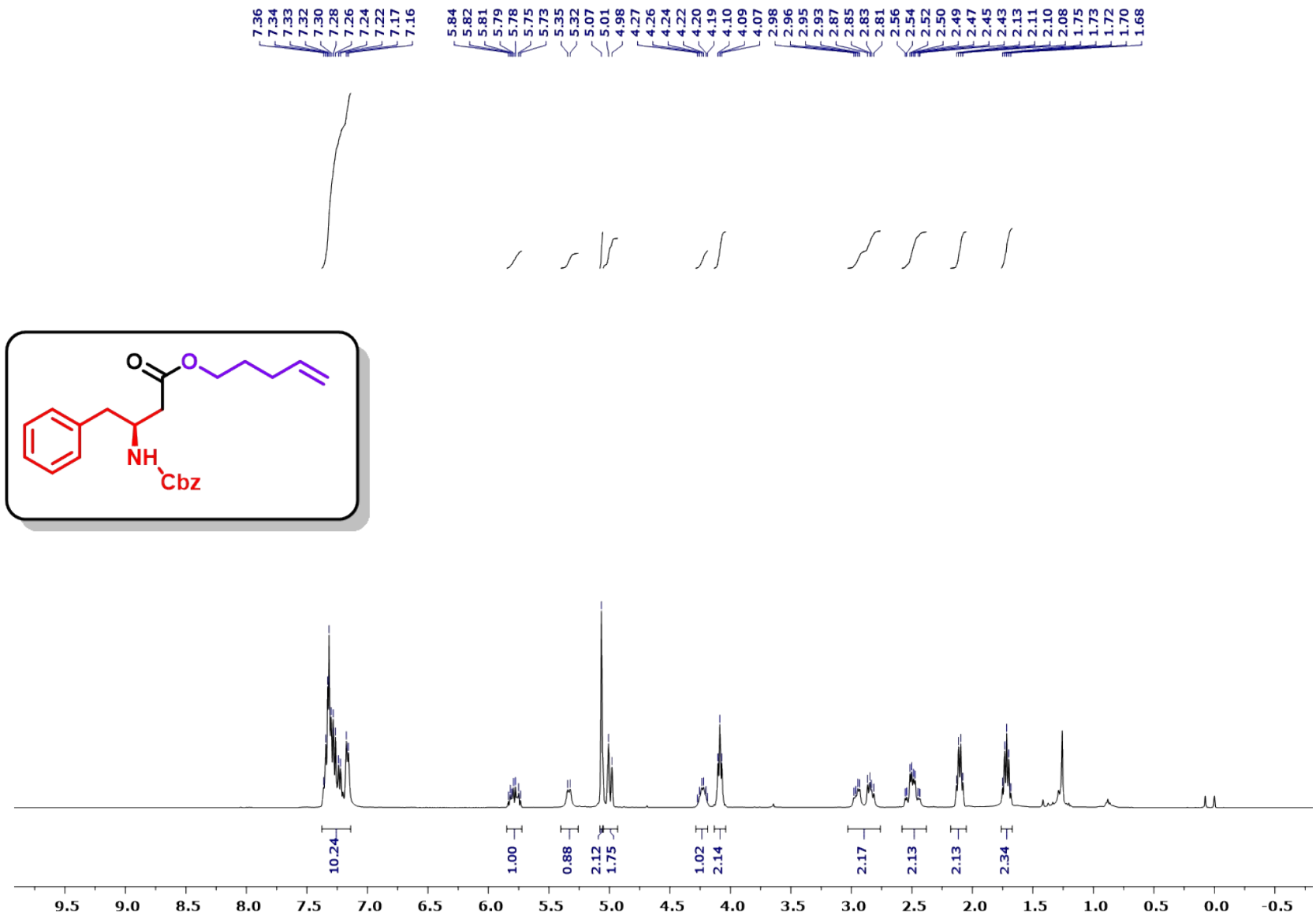

Fig. S48. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of pent-4-en-1-yl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (**6ah**).

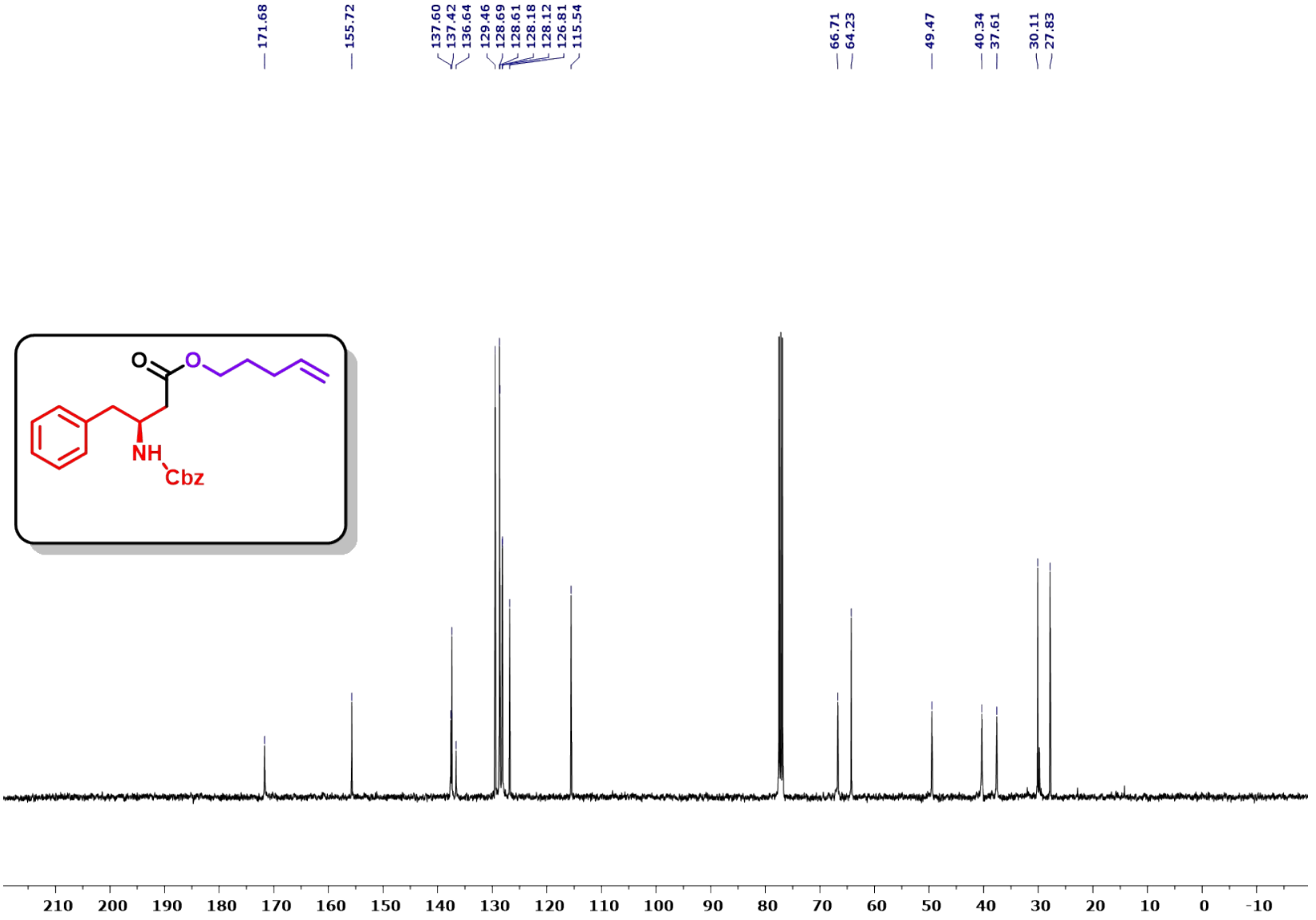

Fig. S49. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of pent-4-en-1-yl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (**6ah**).

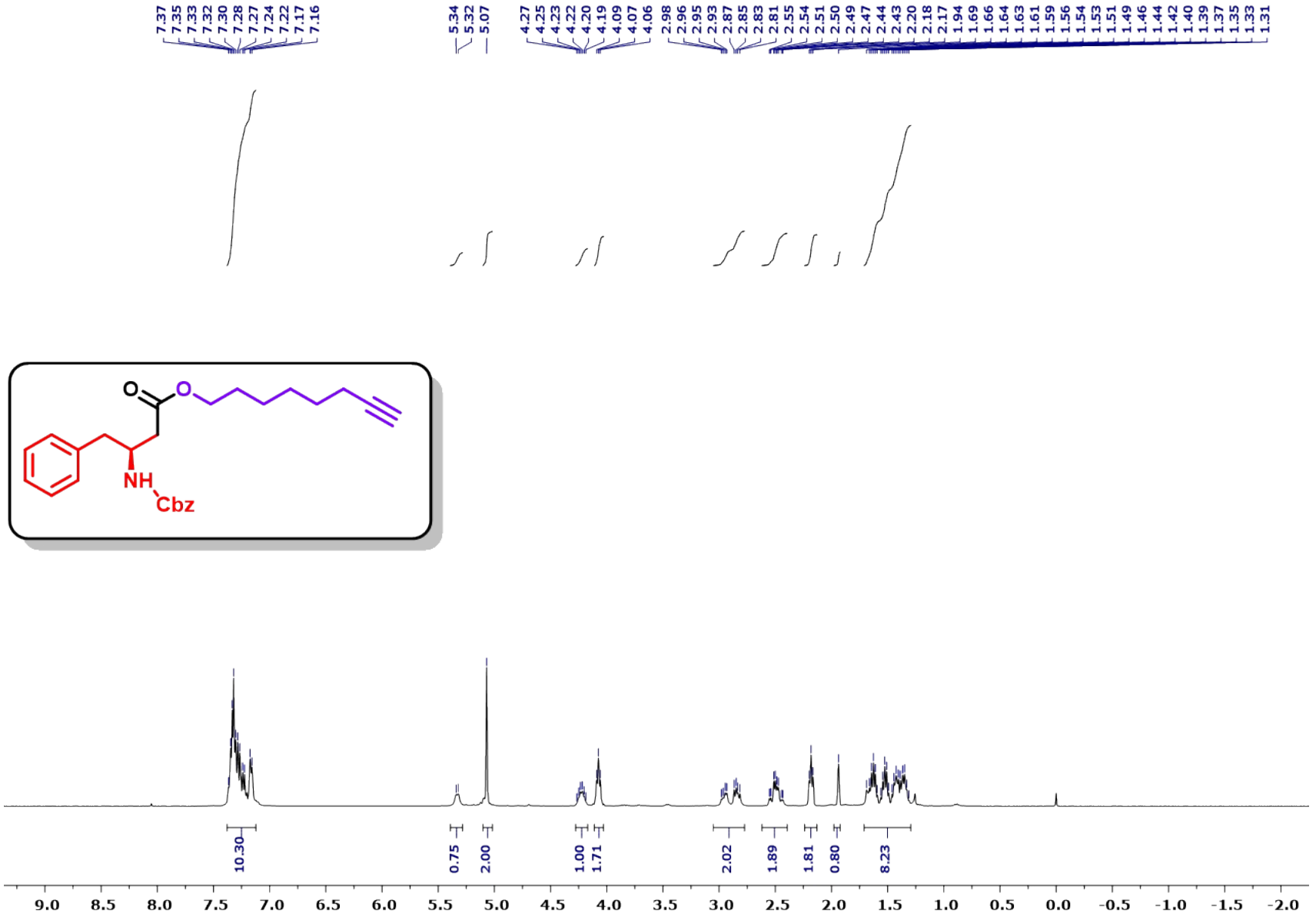

Fig. S50. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of oct-7-yn-1-yl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (**6ai**).

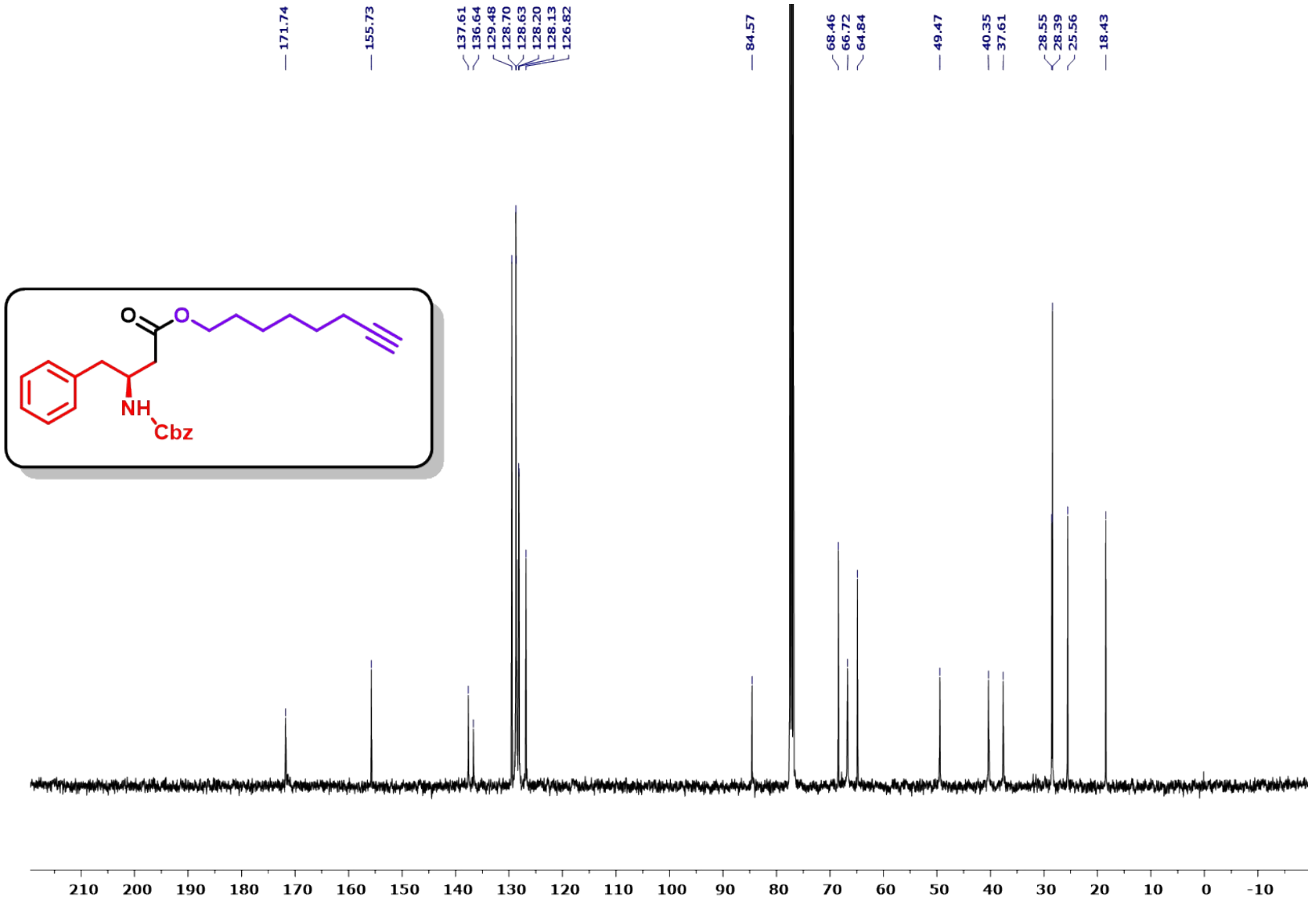

Fig. S51. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of oct-7-yn-1-yl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanoate (**6ai**).

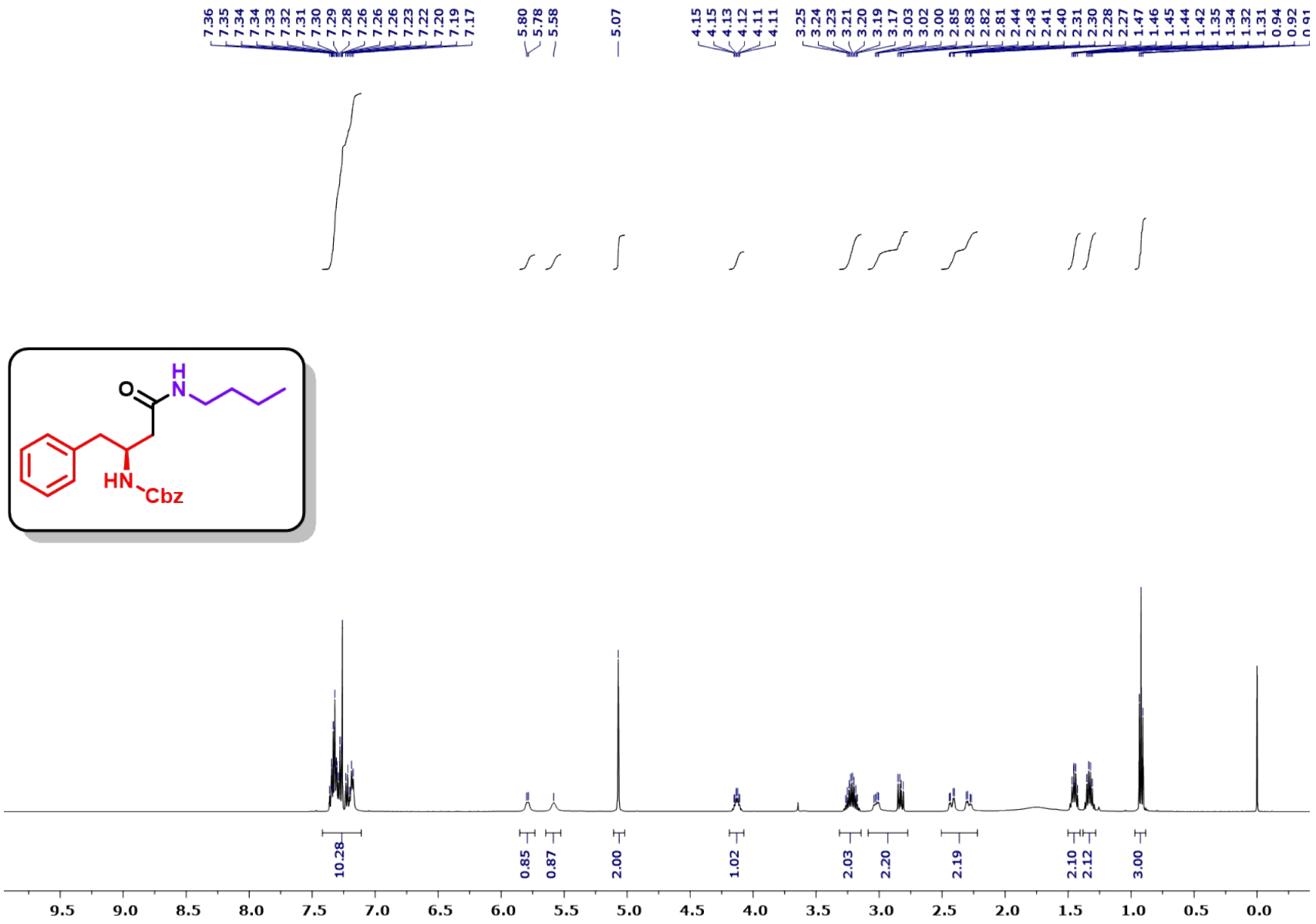

Fig. S52. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of benzyl (*S*)-(4-(butylamino)-4-oxo-1-phenylbutan-2-yl) carbamate (**6aj**).

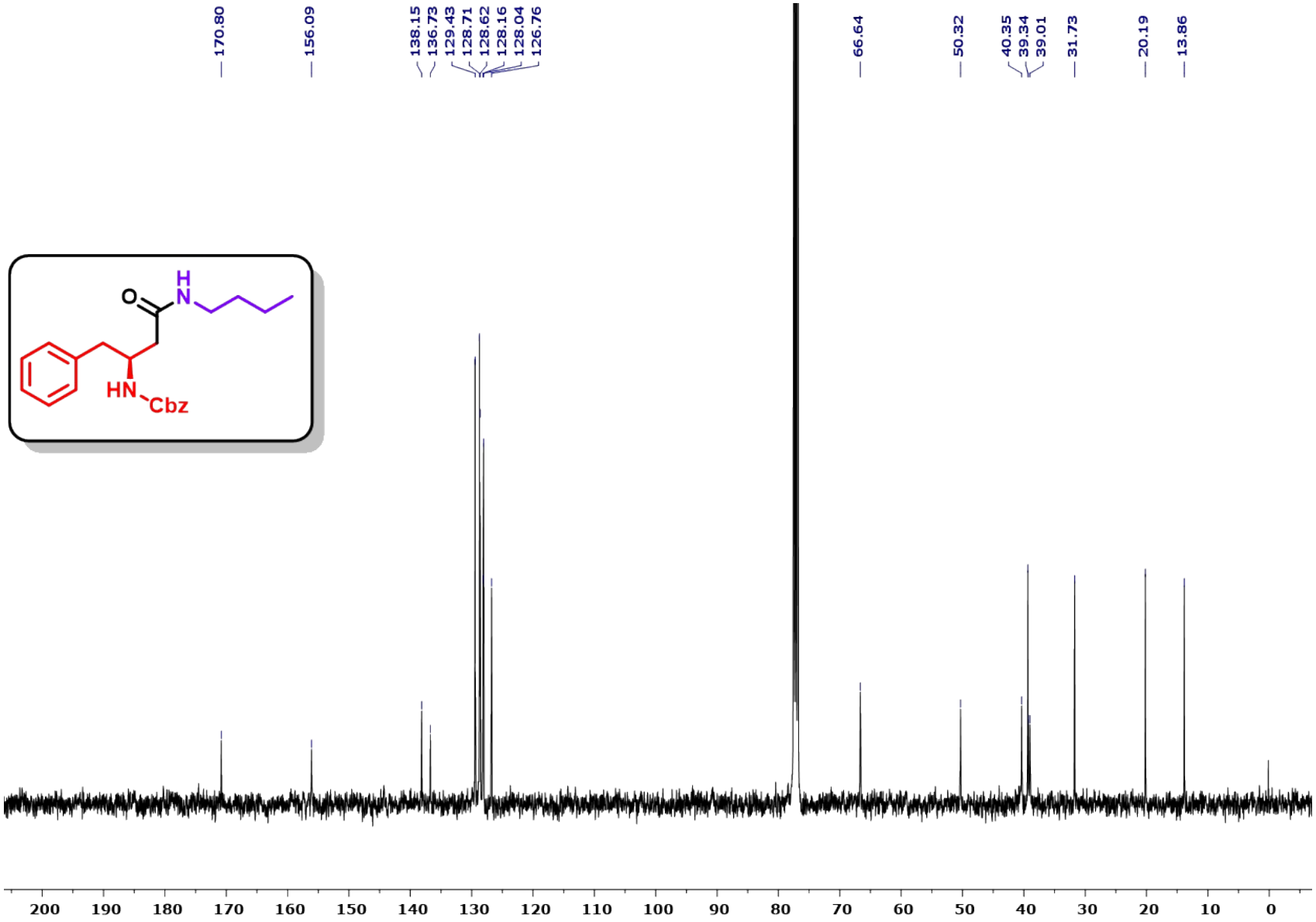

Fig. S53. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of benzyl (*S*)-(4-(butylamino)-4-oxo-1-phenylbutan-2-yl) carbamate (**6aj**).

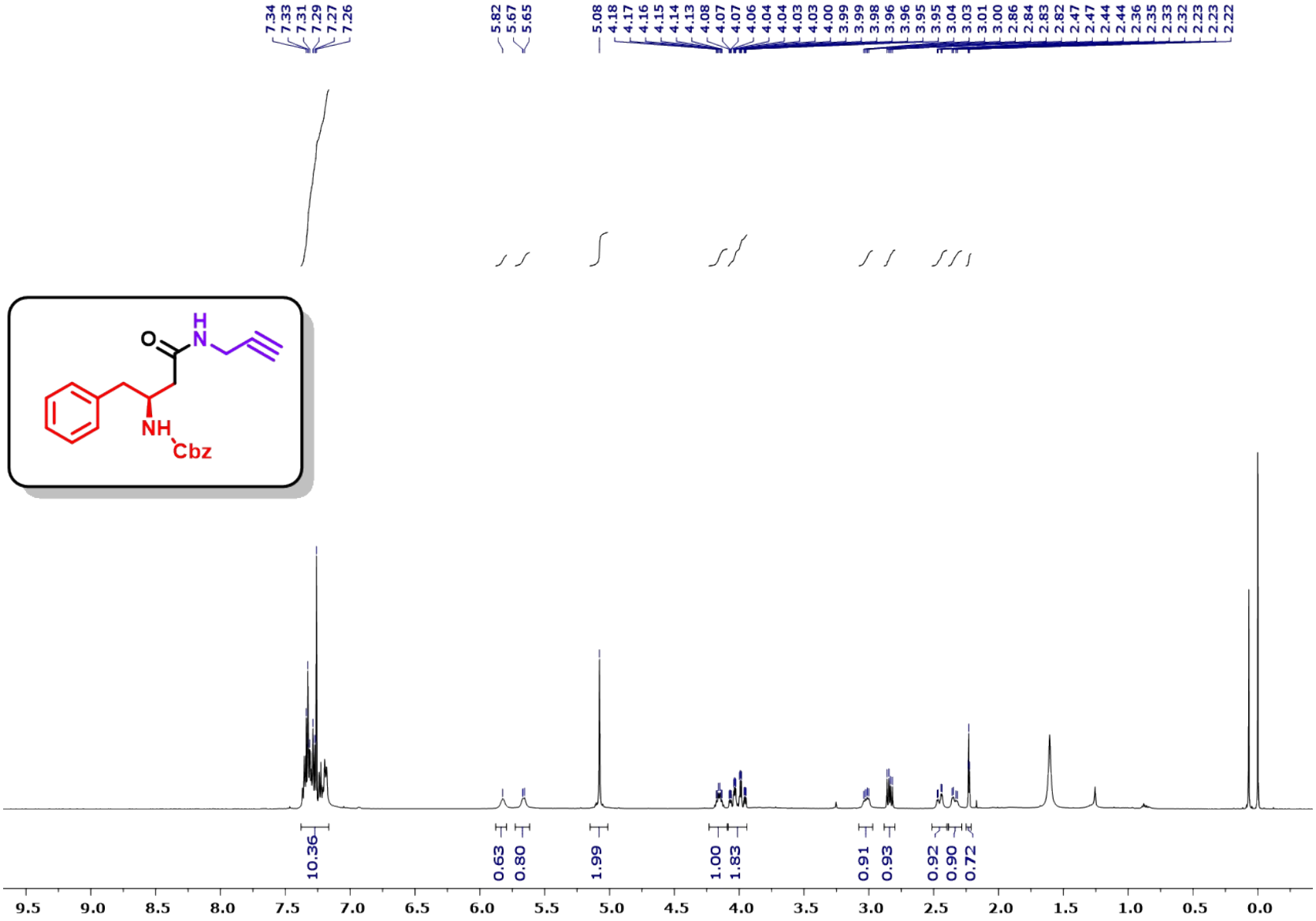

Fig. S54. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of benzyl (*S*)-(4-oxo-1-phenyl-4-(prop-2-yn-1-ylamino) butan-2-yl) carbamate (**6ak**).

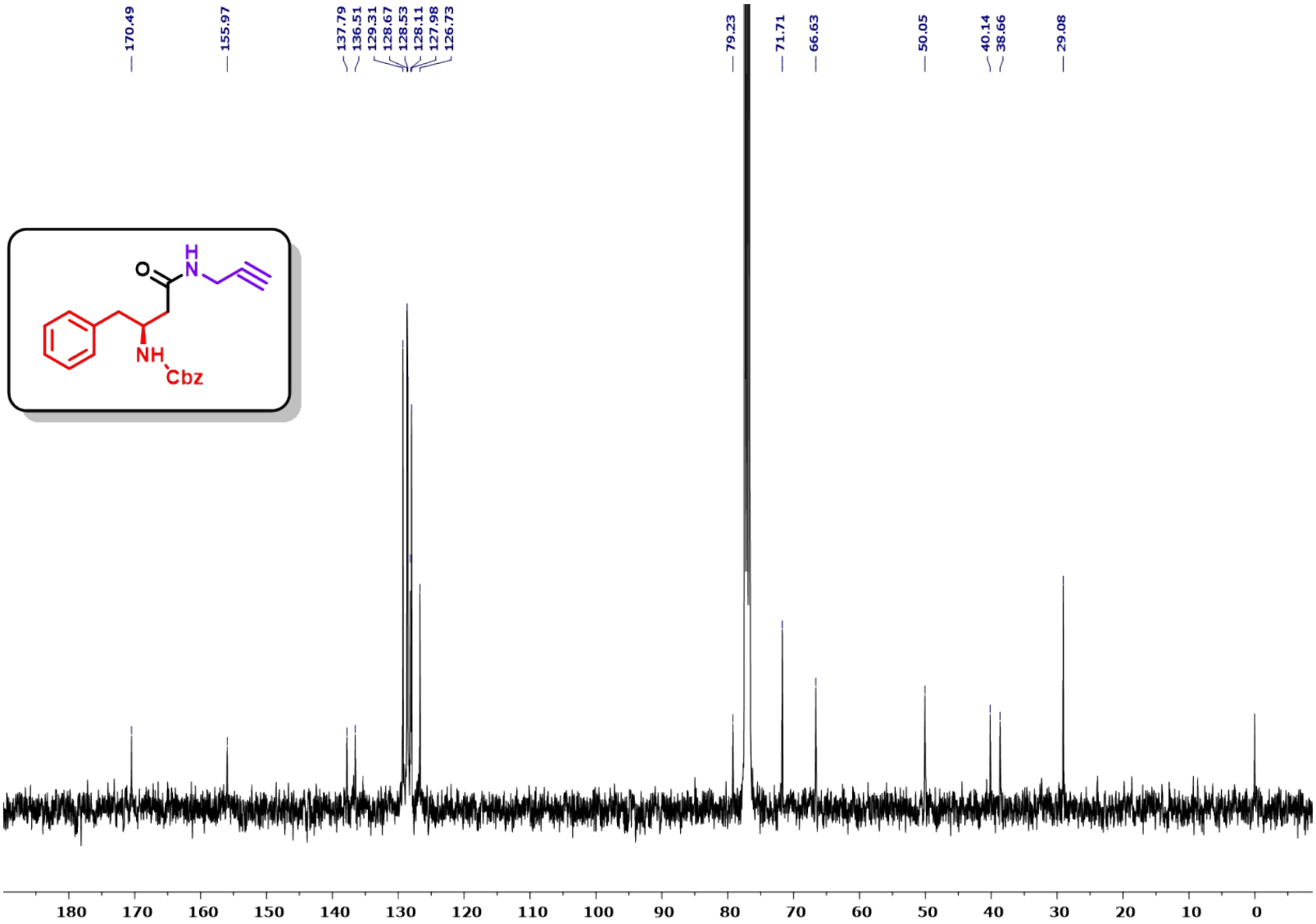

Fig. S55. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of benzyl (*S*)-(4-oxo-1-phenyl-4-(prop-2-yn-1-ylamino) butan-2-yl) carbamate (**6ak**).

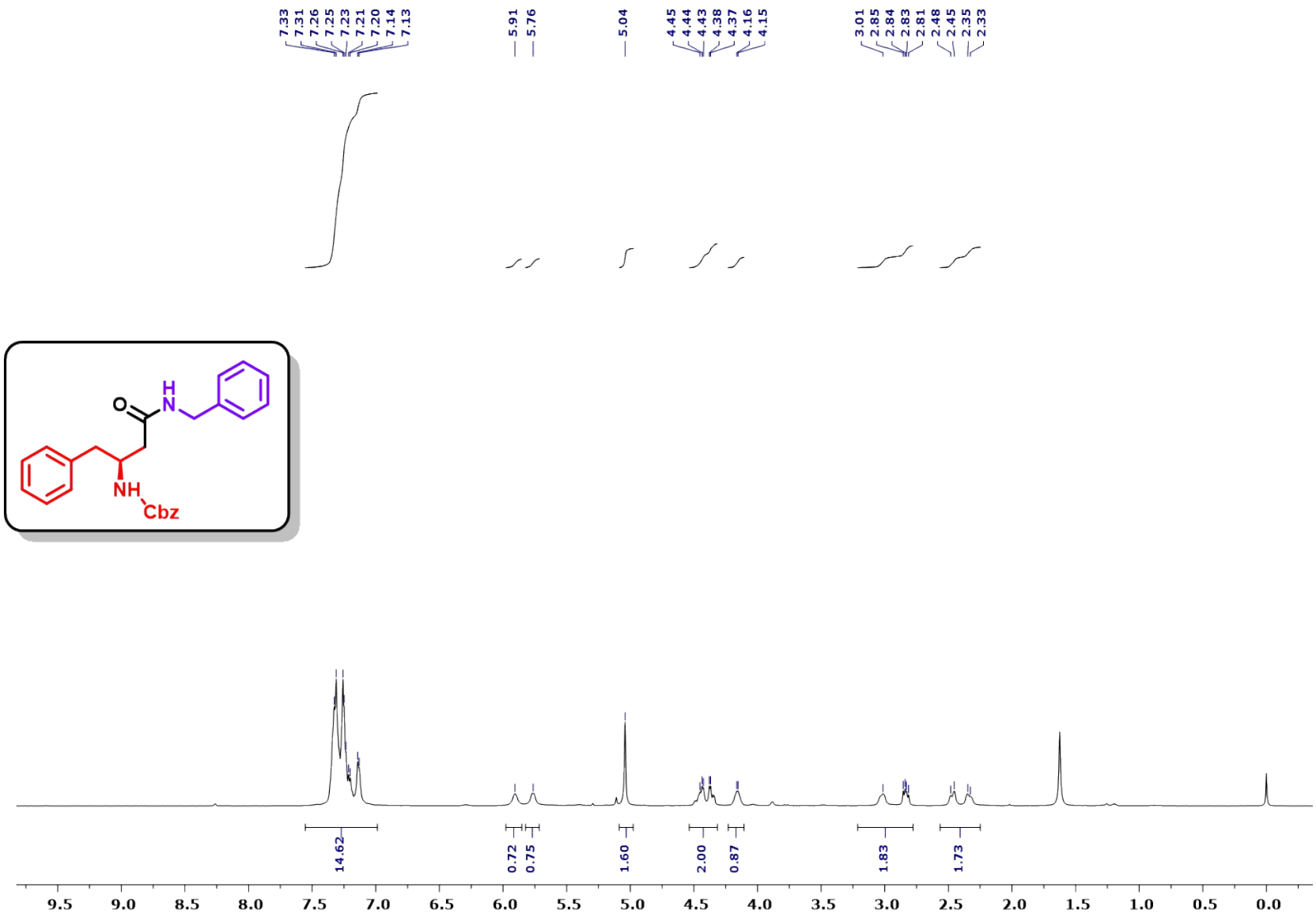

Fig. S56. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of benzyl (*S*)-(4-(benzylamino)-4-oxo-1-phenylbutan-2-yl) carbamate (**6ai**).

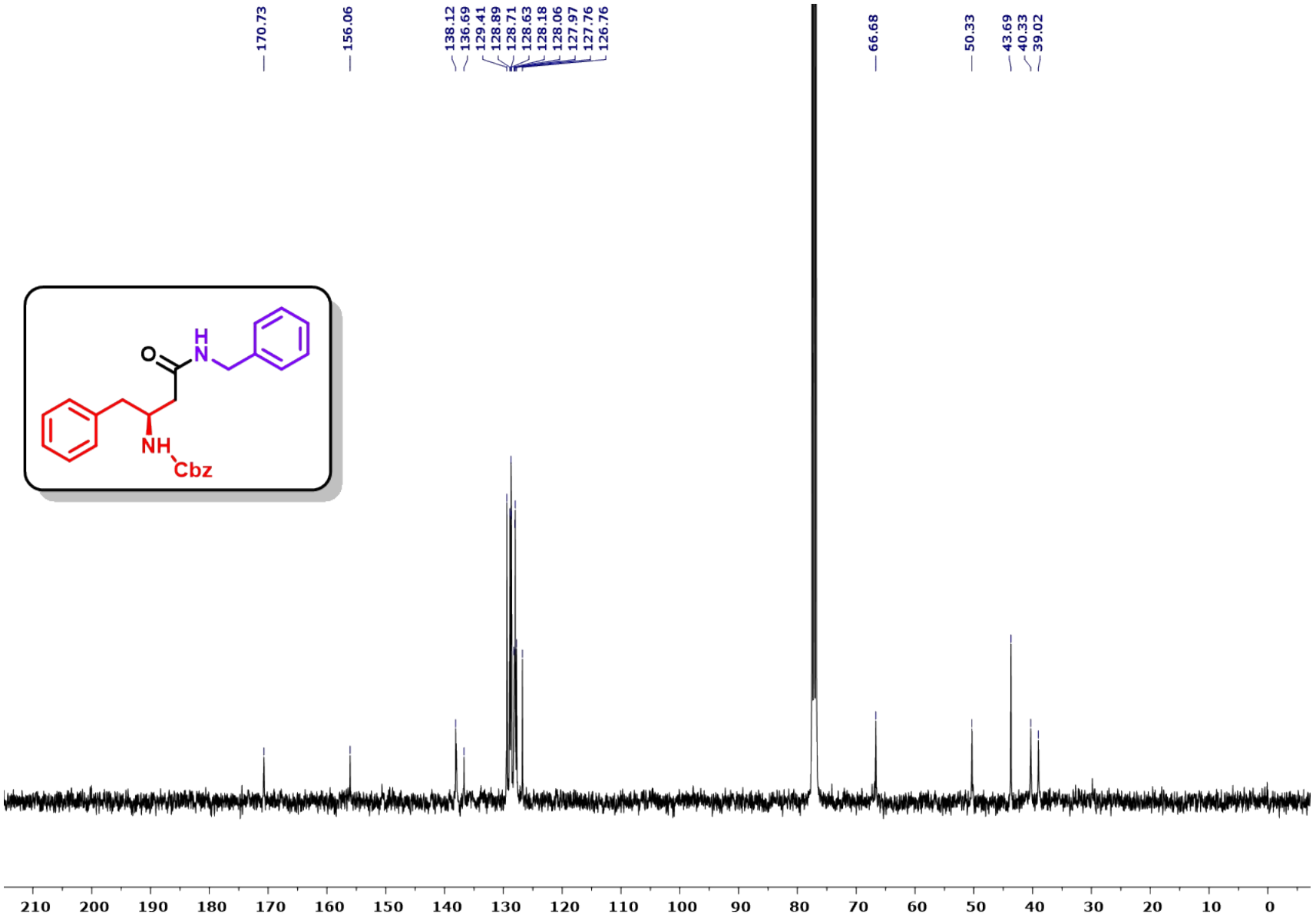

Fig. S57. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of benzyl (*S*)-(4-(benzylamino)-4-oxo-1-phenylbutan-2-yl) carbamate (**6ai**).

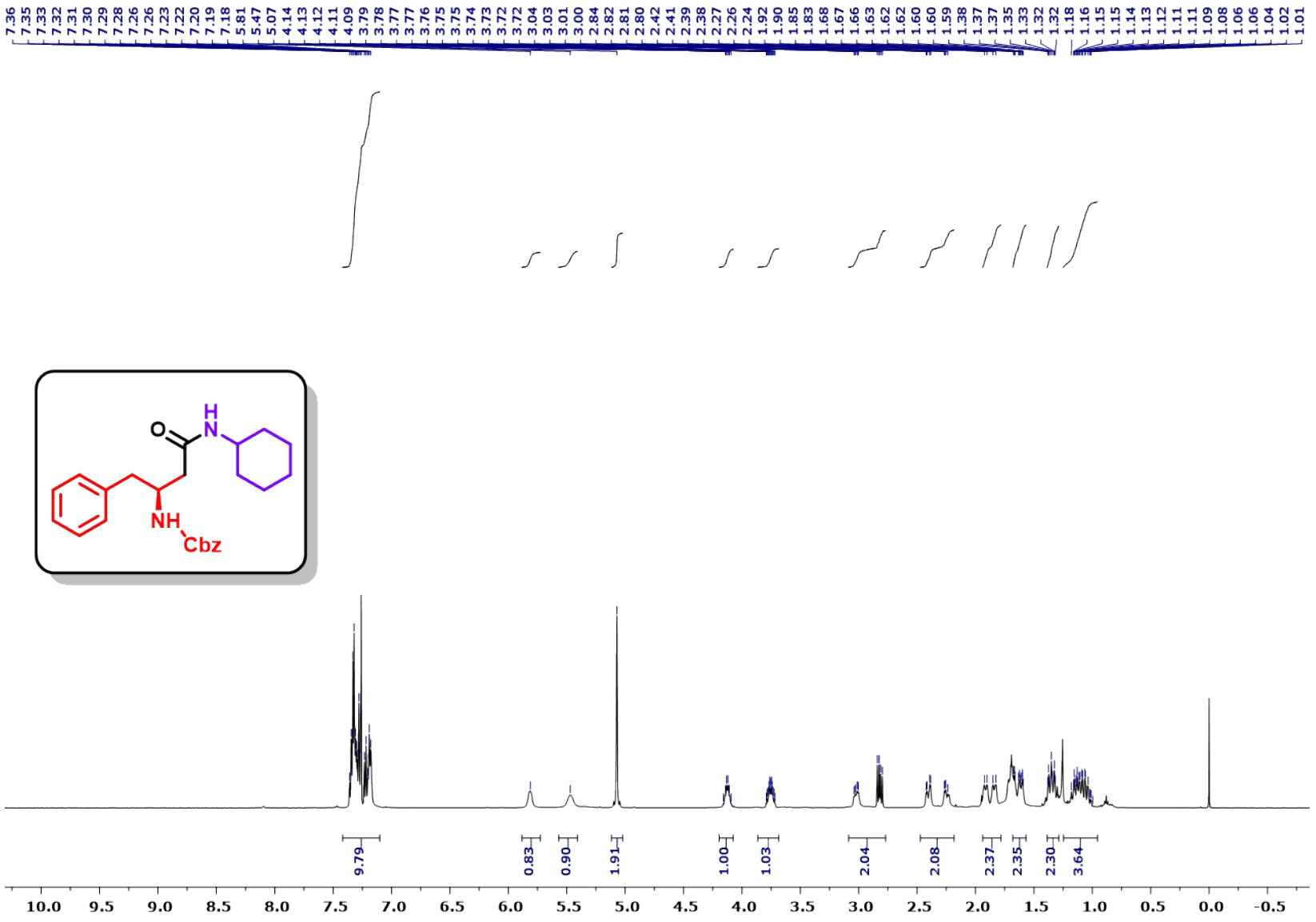

Fig. S58. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of benzyl (*S*)-(4-(cyclohexylamino)-4-oxo-1-phenylbutan-2-yl) carbamate (**6am**).

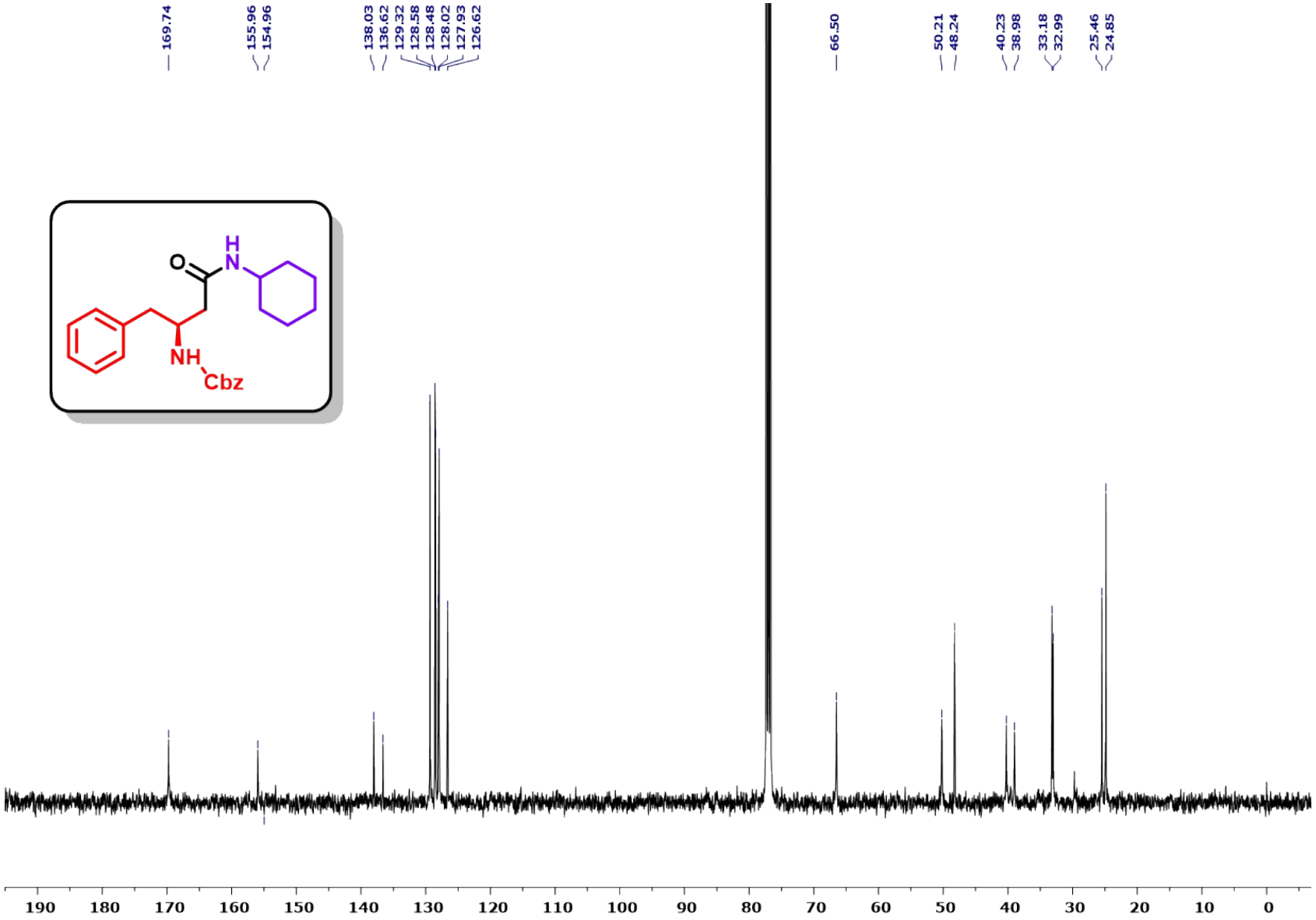

Fig. S59. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of benzyl (*S*)-(4-(cyclohexylamino)-4-oxo-1-phenylbutan-2-yl) carbamate (**6am**).

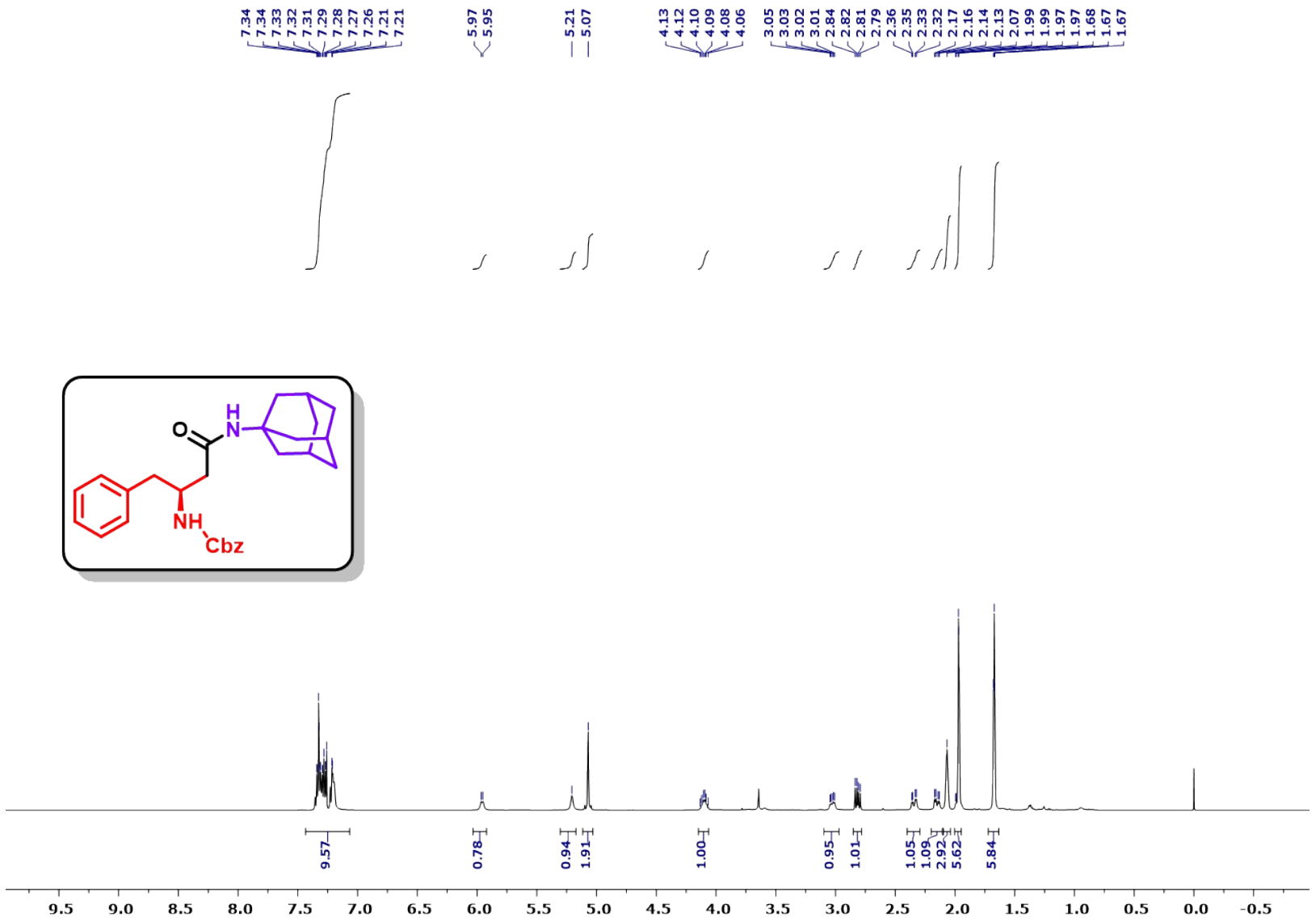

Fig. S60. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of benzyl((*S*)-4-(((3R,5R,7R)-adamantan-1-yl) amino)-4-oxo-1-phenylbutan-2-yl) carbamate (**6an**).

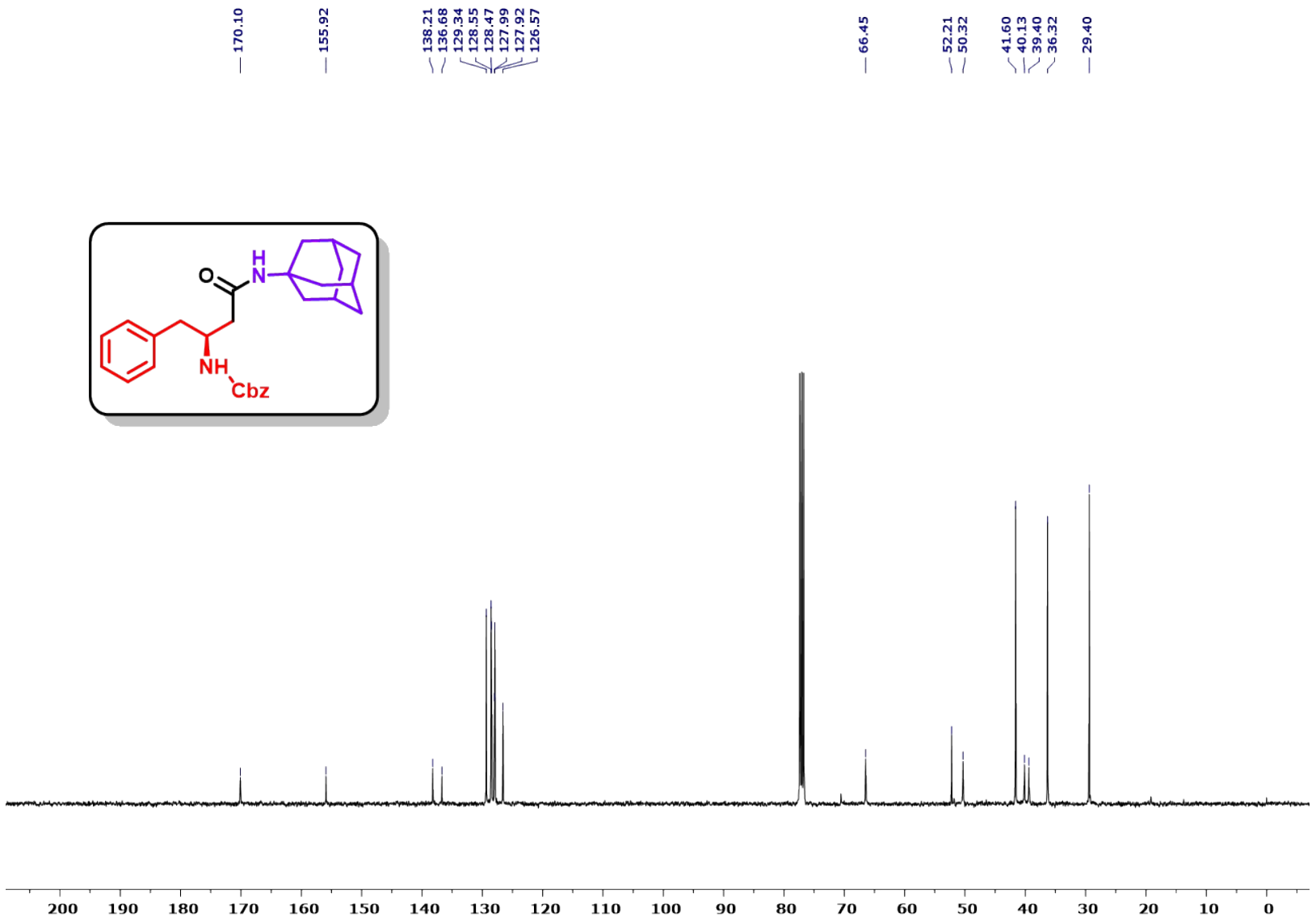

Fig. S61. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of benzyl((*S*)-4-(((3R,5R,7R)-adamantan-1-yl) amino)-4-oxo-1-phenylbutan-2-yl) carbamate (**6an**).

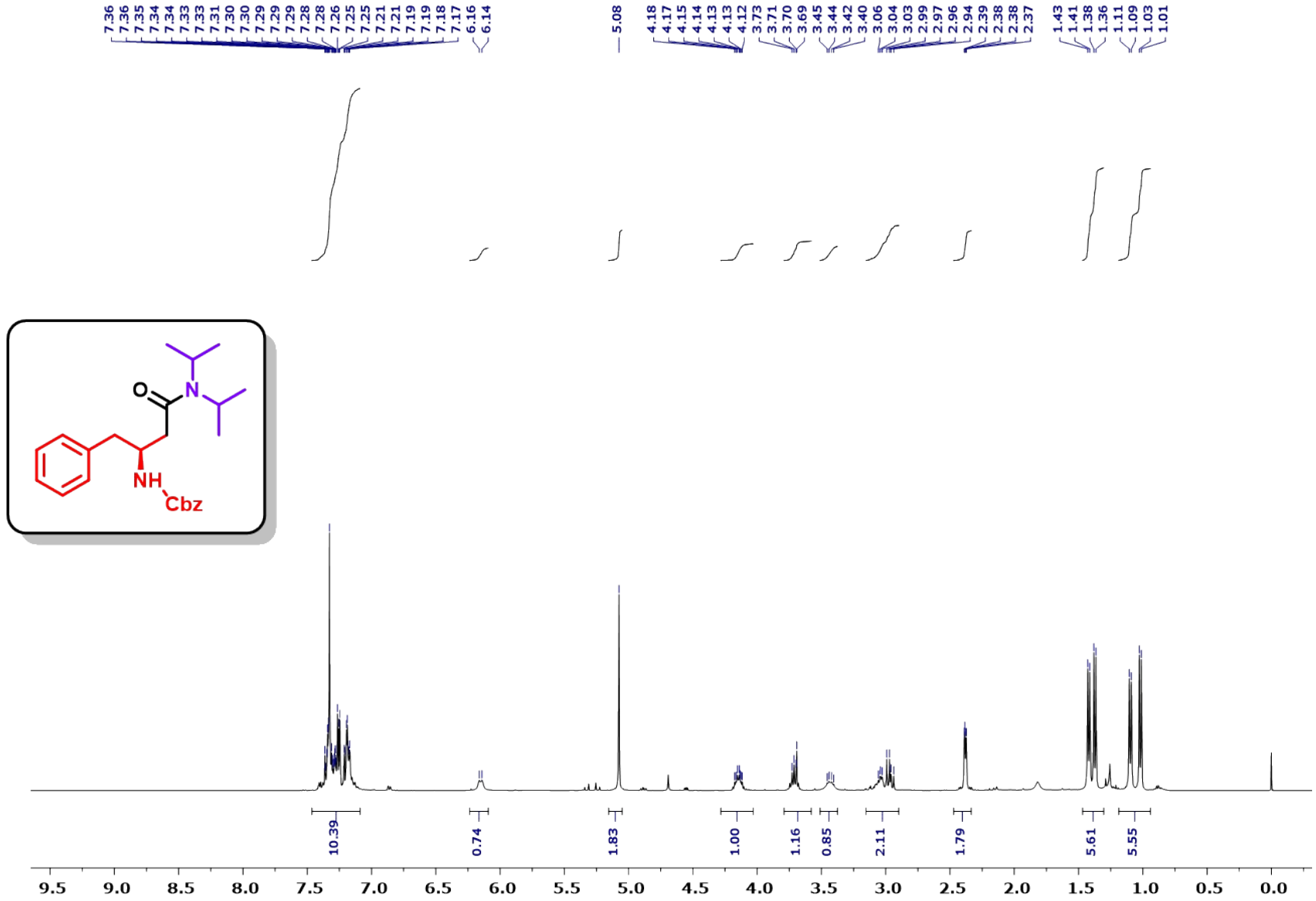

Fig. S62. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of benzyl (*S*)-(4-(diisopropylamino)-4-oxo-1-phenylbutan-2-yl) carbamate (**6ao**).

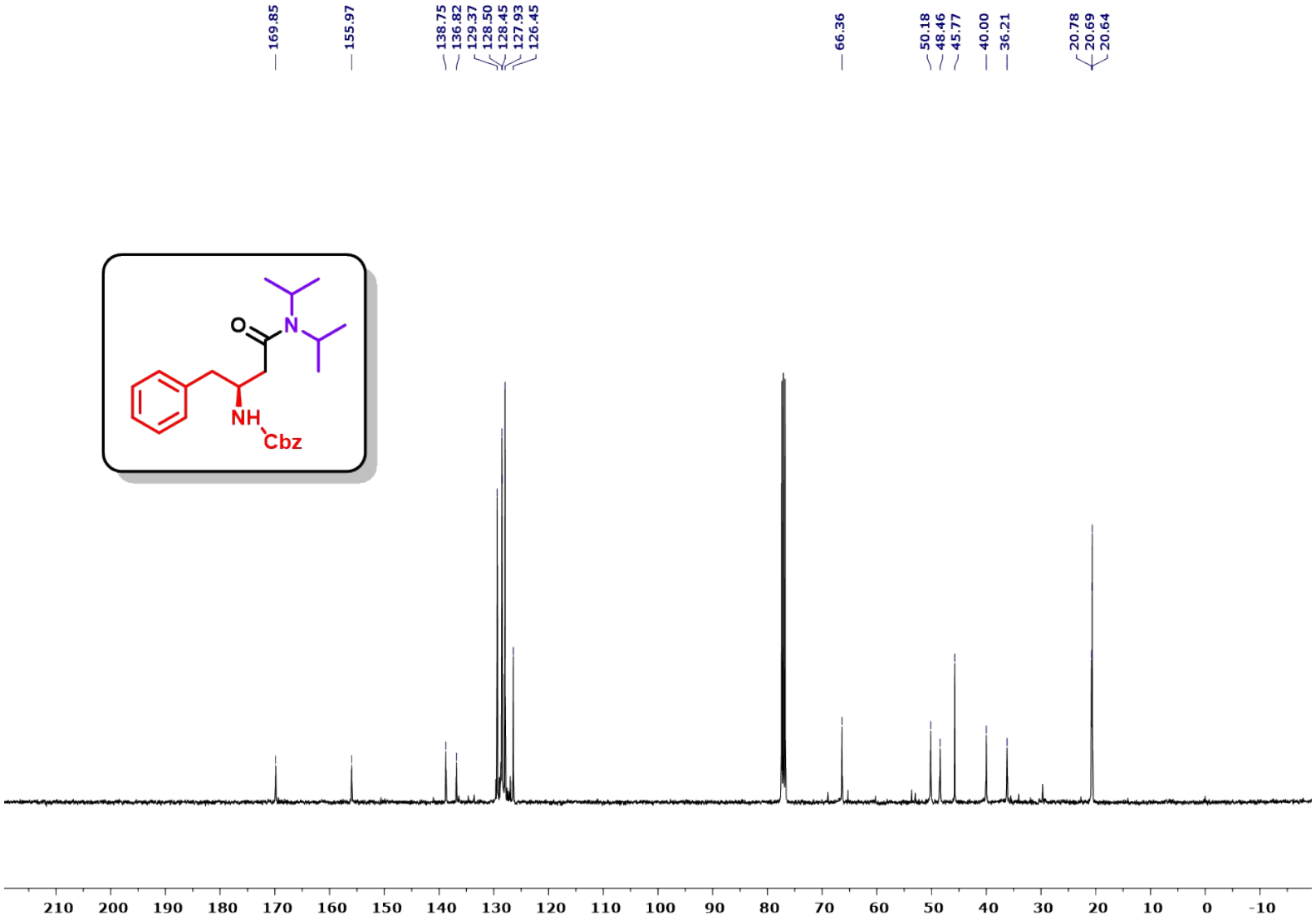

Fig. S63. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of benzyl (*S*)-(4-(diisopropylamino)-4-oxo-1-phenylbutan-2-yl) carbamate (**6ao**).

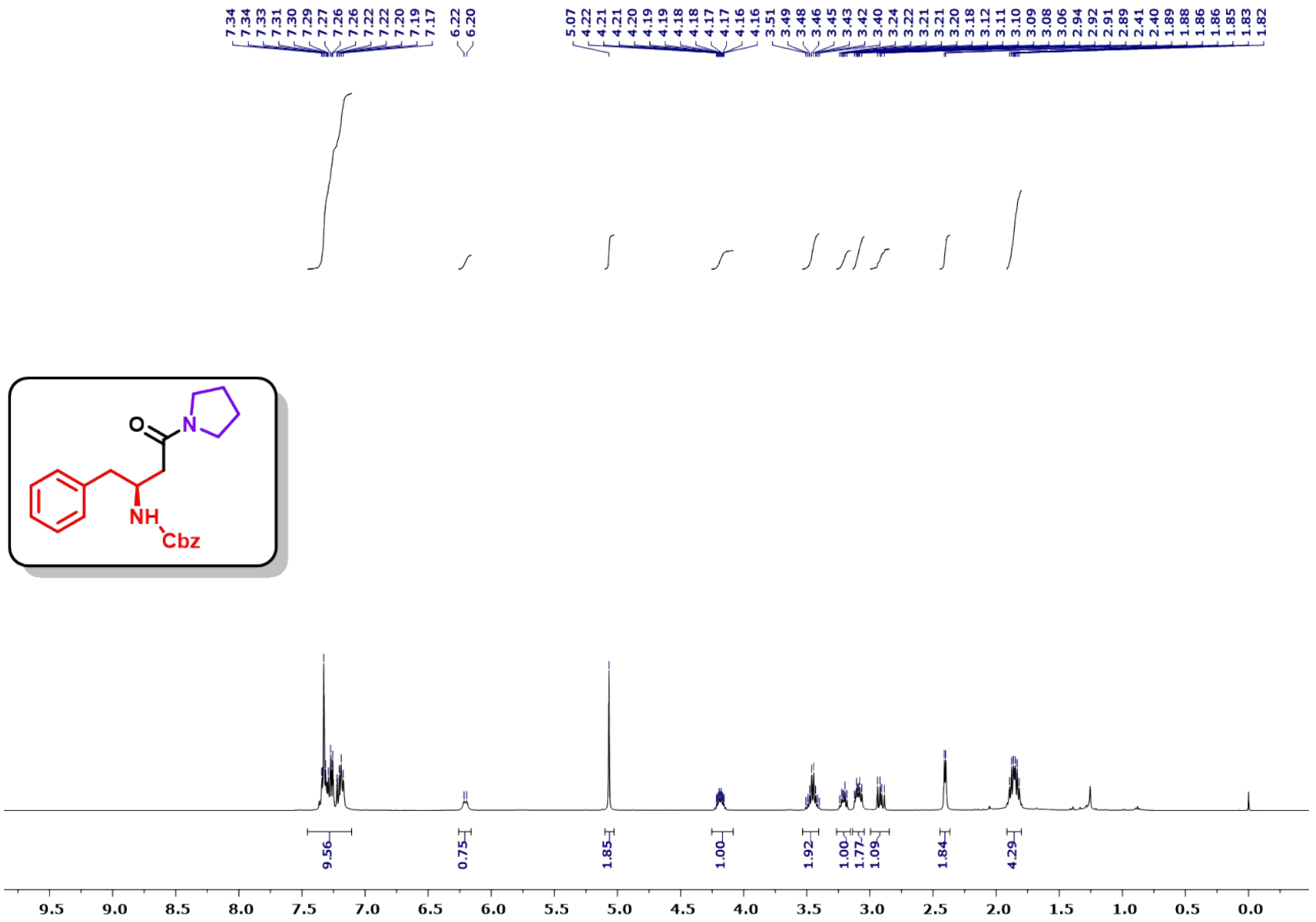

Fig. S64. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of benzyl (*S*)-(4-oxo-1-phenyl-4-(pyrrolidin-1-yl) butan-2-yl) carbamate (**6ap**).

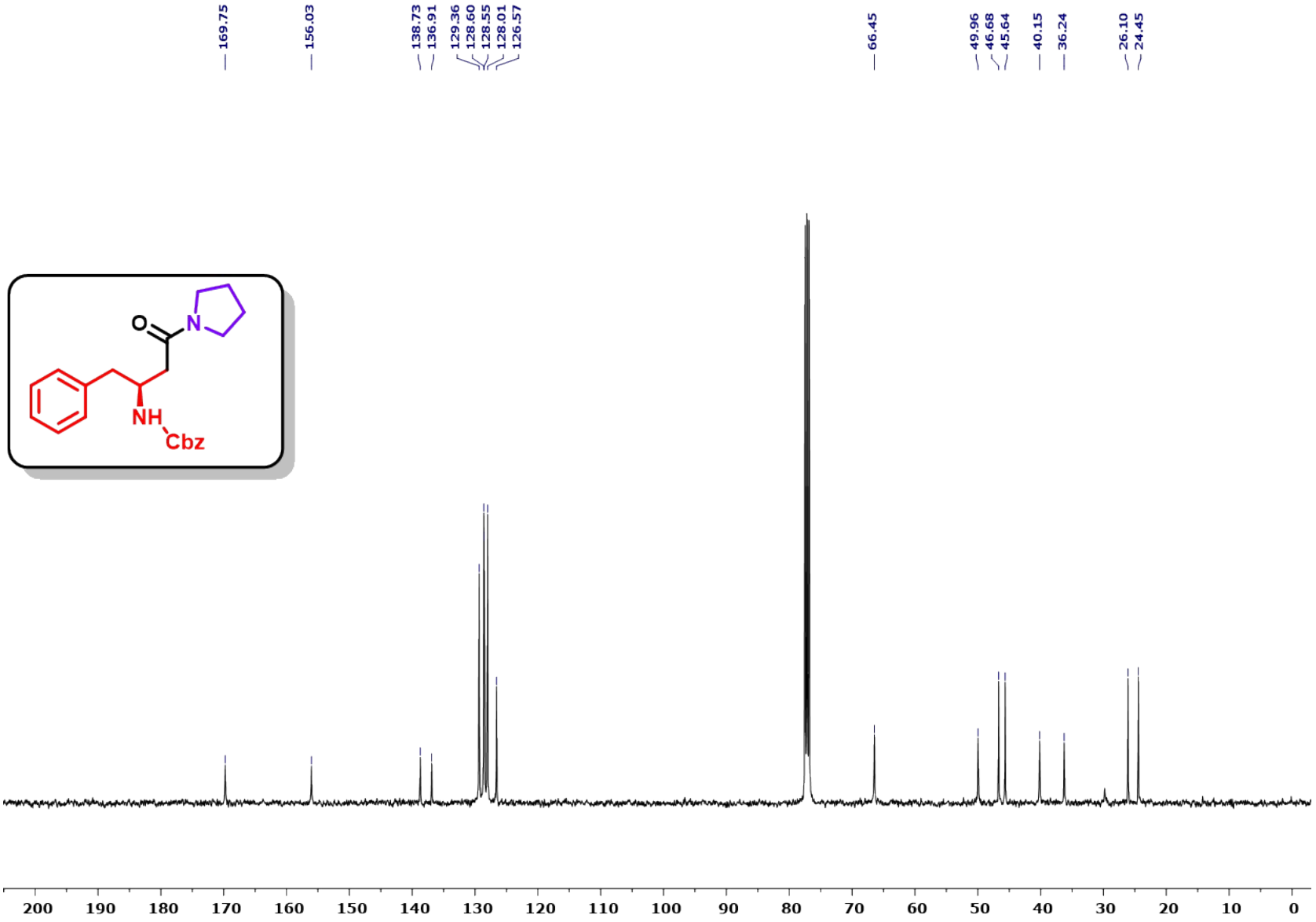

Fig. S65. ^{13}C NMR (101 MHz, $CDCl_3$**)** spectra of benzyl (*S*)-(4-oxo-1-phenyl-4-(pyrrolidin-1-yl) butan-2-yl) carbamate (**6ap**).

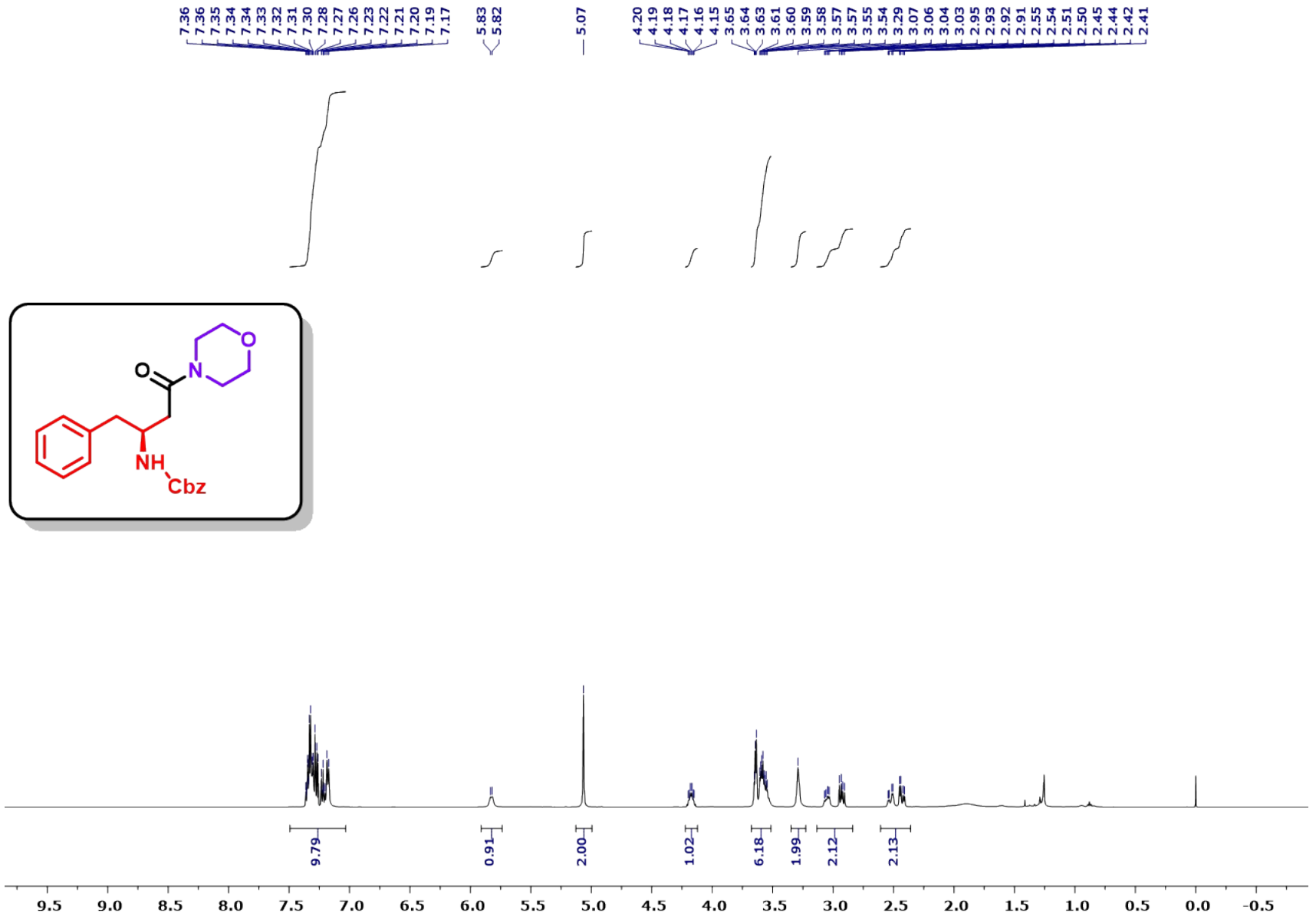

Fig. S66. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of benzyl (*S*)-(4-morpholino-4-oxo-1-phenylbutan-2-yl) carbamate (**6aq**).

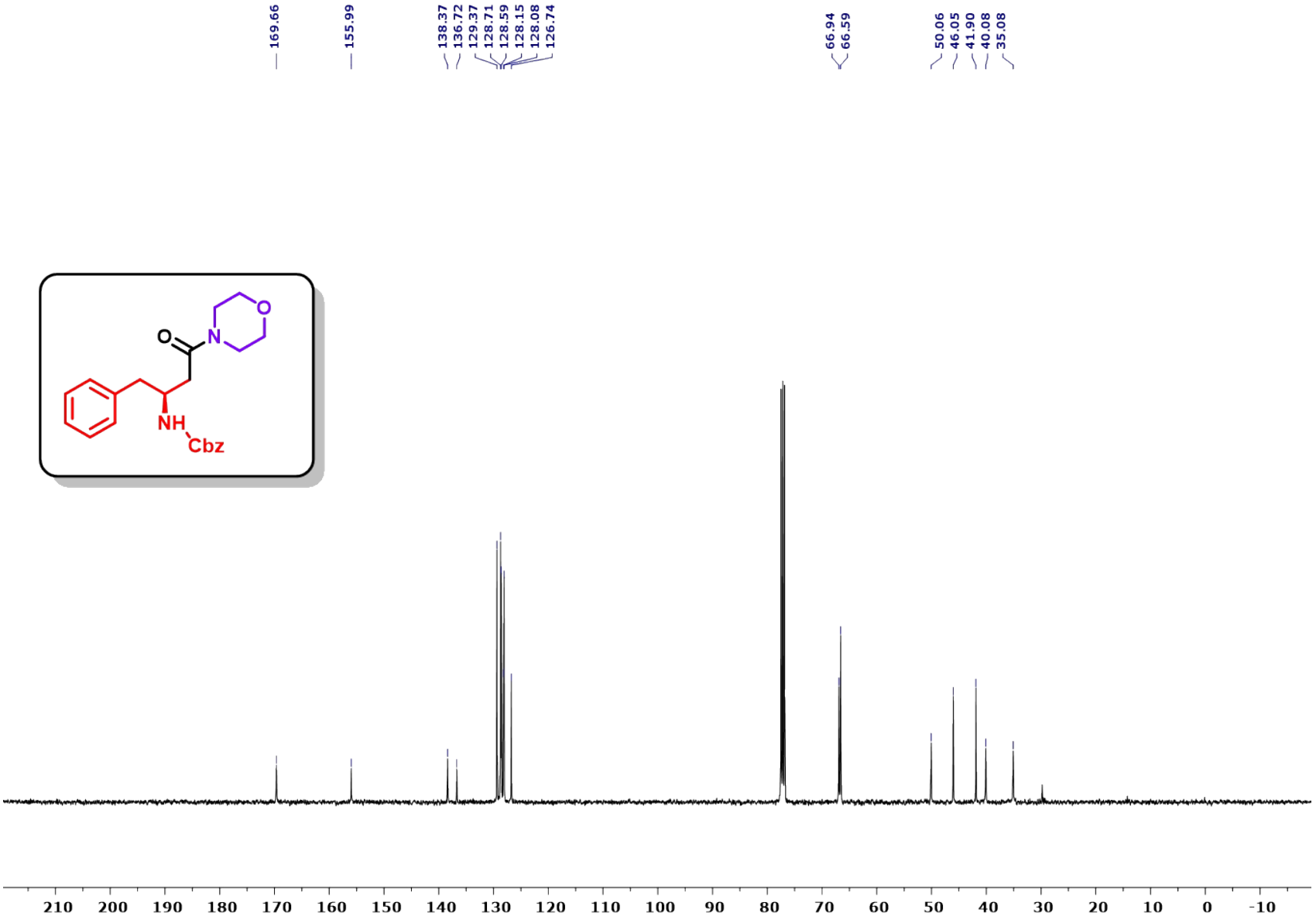

Fig. S67. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of benzyl (*S*)-(4-morpholino-4-oxo-1-phenylbutan-2-yl) carbamate (**6aq**).

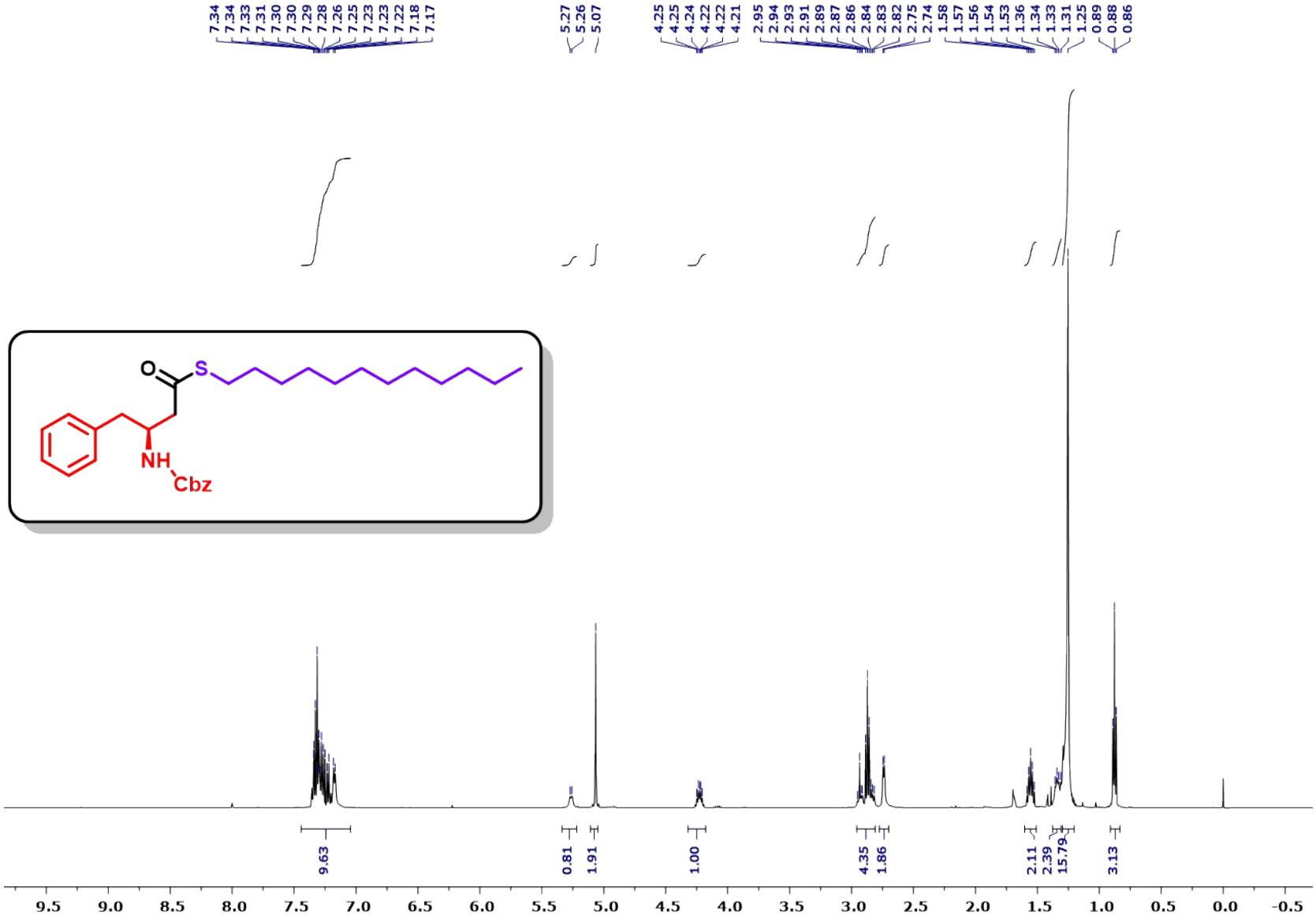

Fig. S68. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of *S*-dodecyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanethioate (**6ar**).

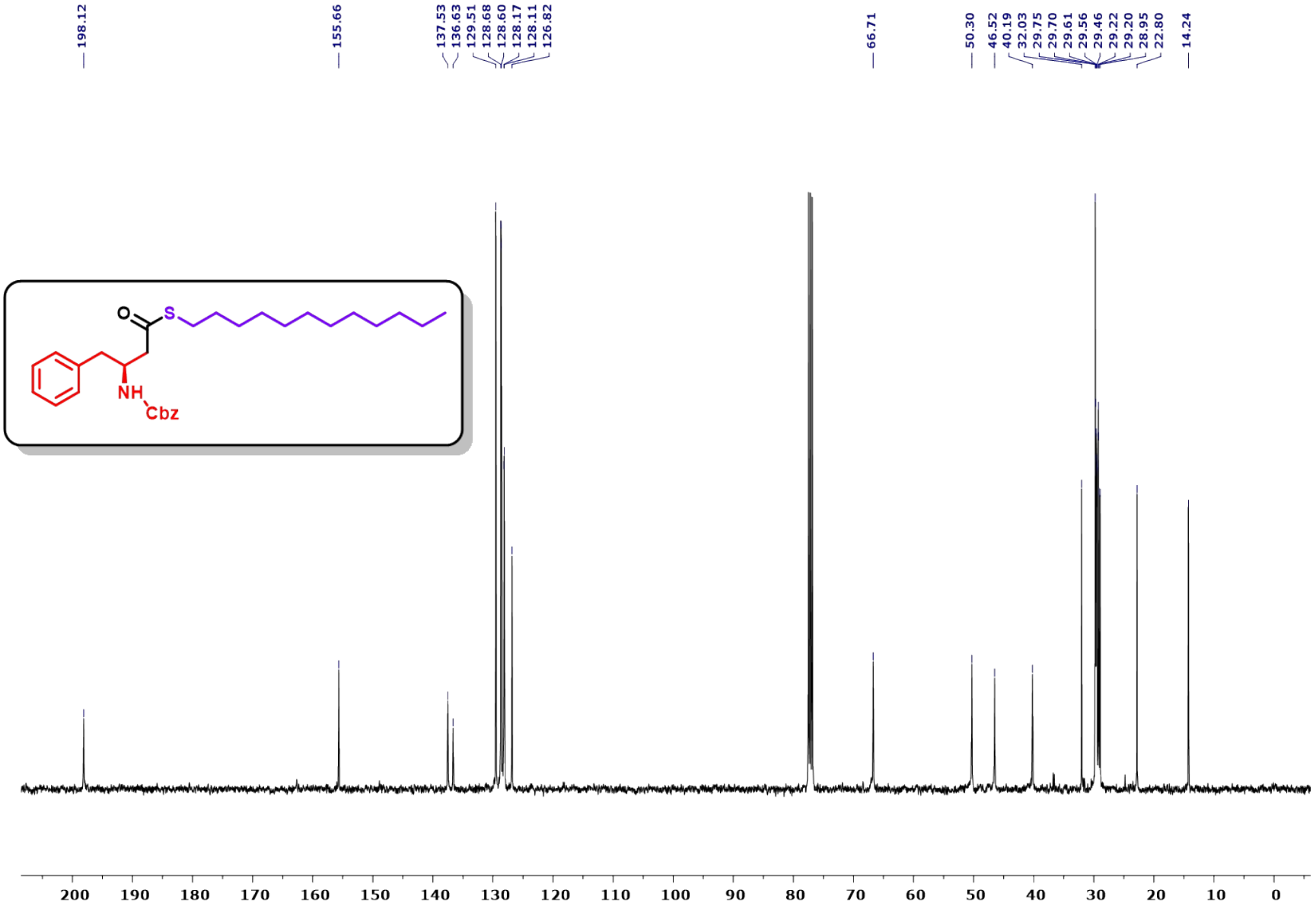

Fig. S69. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of *S*-dodecyl (*S*)-3-(((benzyloxy)carbonyl) amino)-4-phenylbutanethioate (**6ar**).

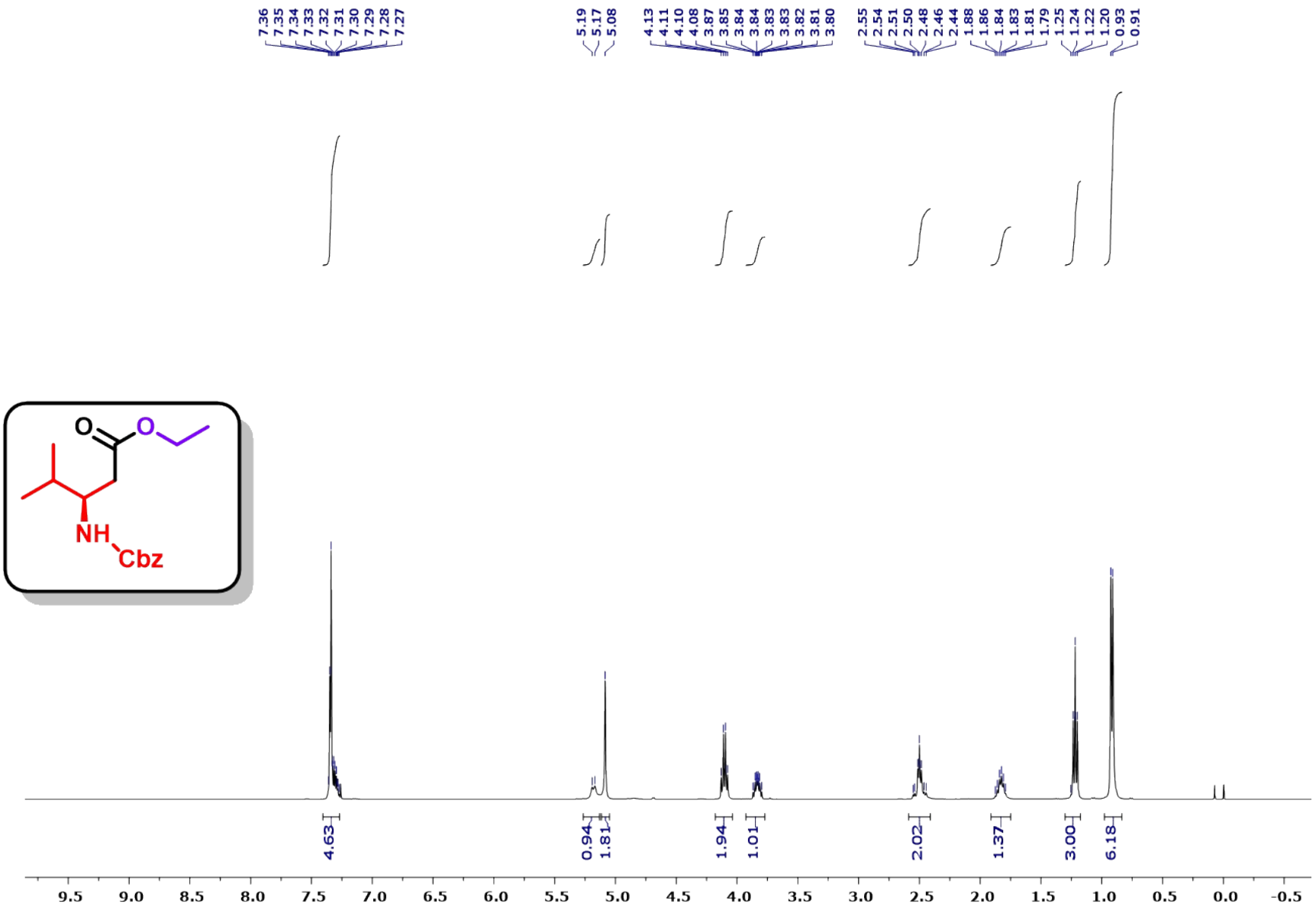

Fig. S70. ^{1}H NMR (400 MHz, $CDCl_3$**)** spectra of ethyl (*R*)-3-(((benzyloxy)carbonyl) amino)-4-methylpentanoate (**6ba**).

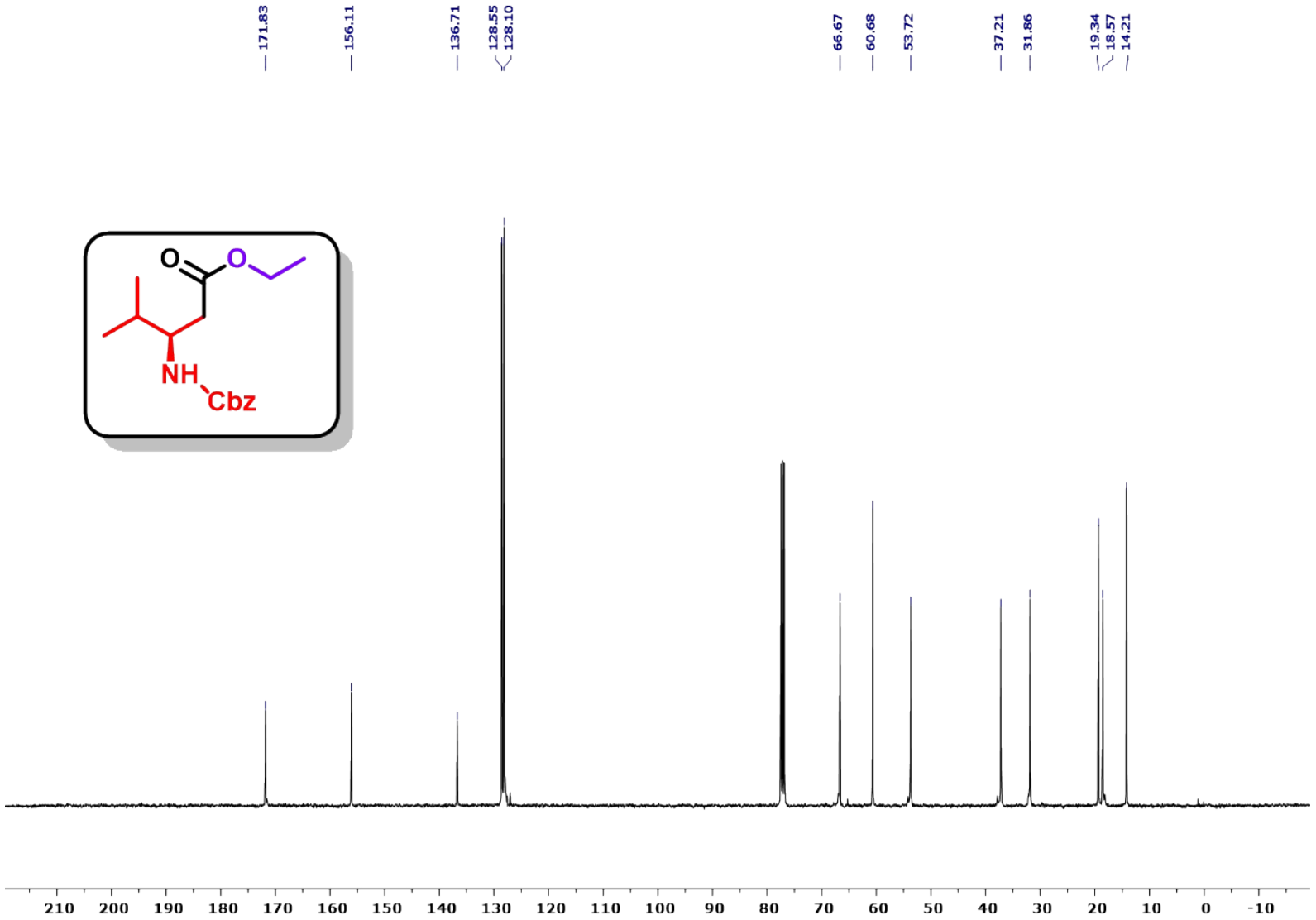

Fig. S71. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of ethyl (*R*)-3-(((benzyloxy)carbonyl) amino)-4-methylpentanoate (**6ba**).

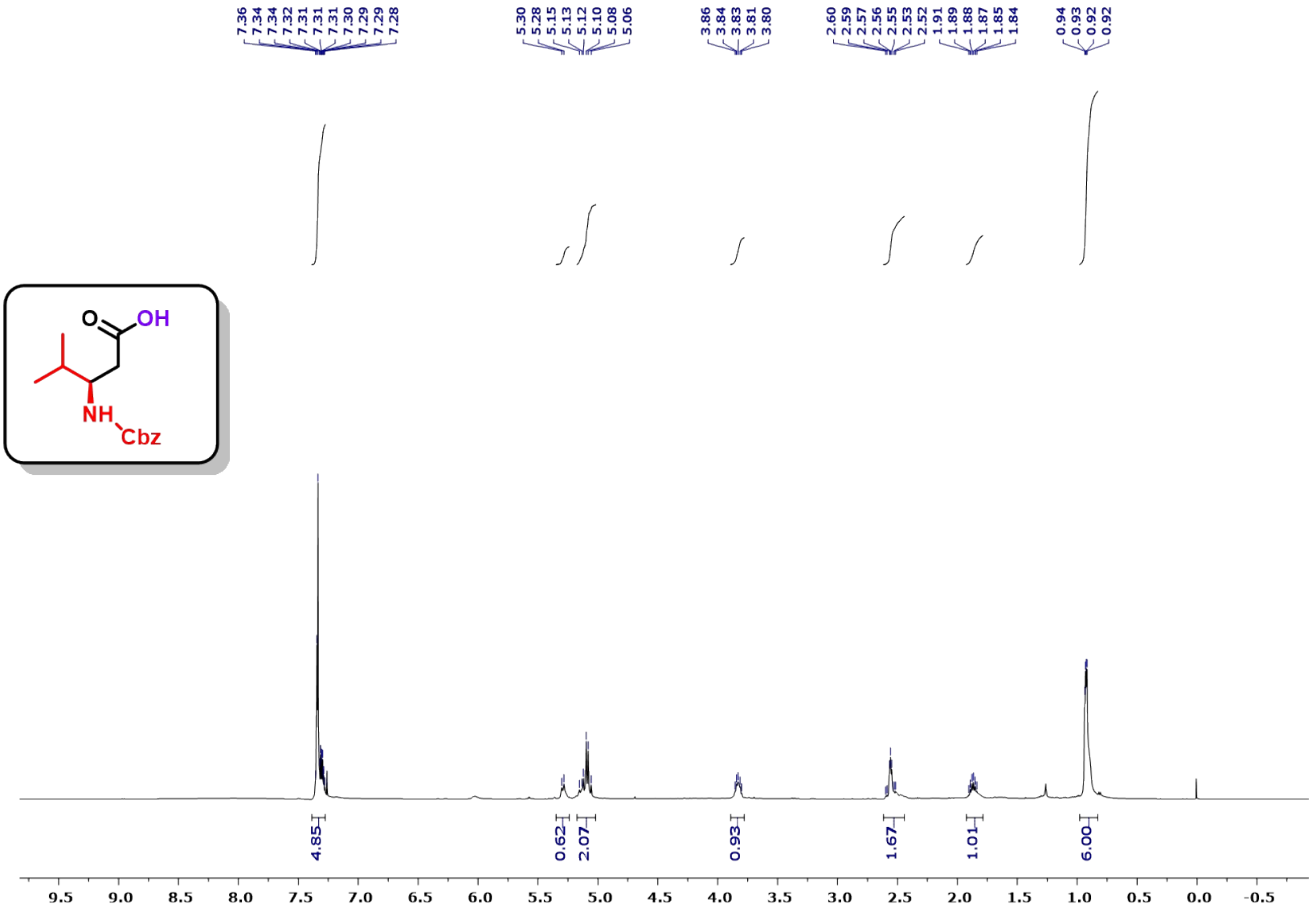

Fig. S72. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of (*R*)-3-(((benzyloxy)carbonyl) amino)-4-methylpentanoic acid (**6bc**).

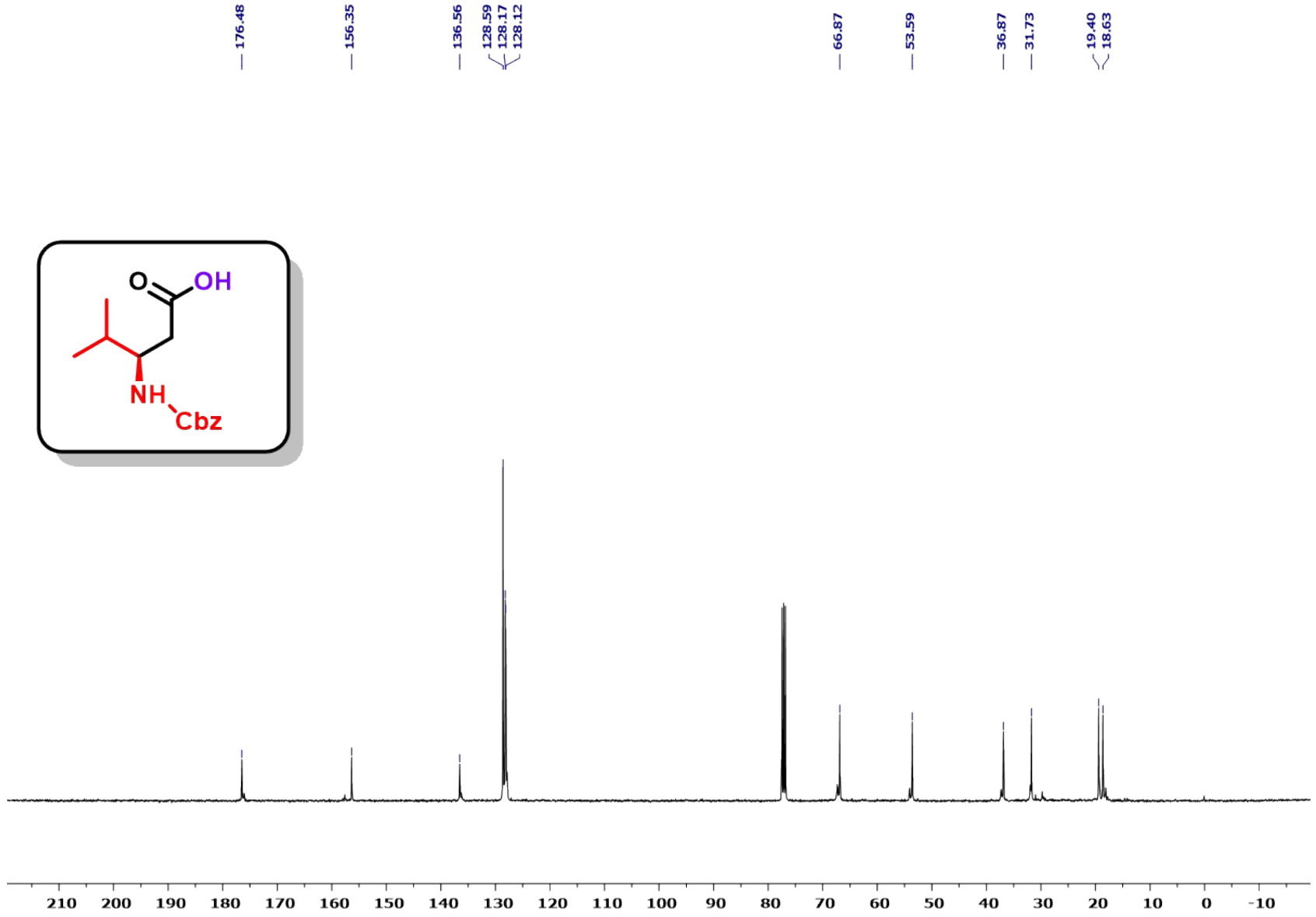

Fig. S73. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of (*R*)-3-(((benzyloxy)carbonyl) amino)-4-methylpentanoic acid (**6bc**).

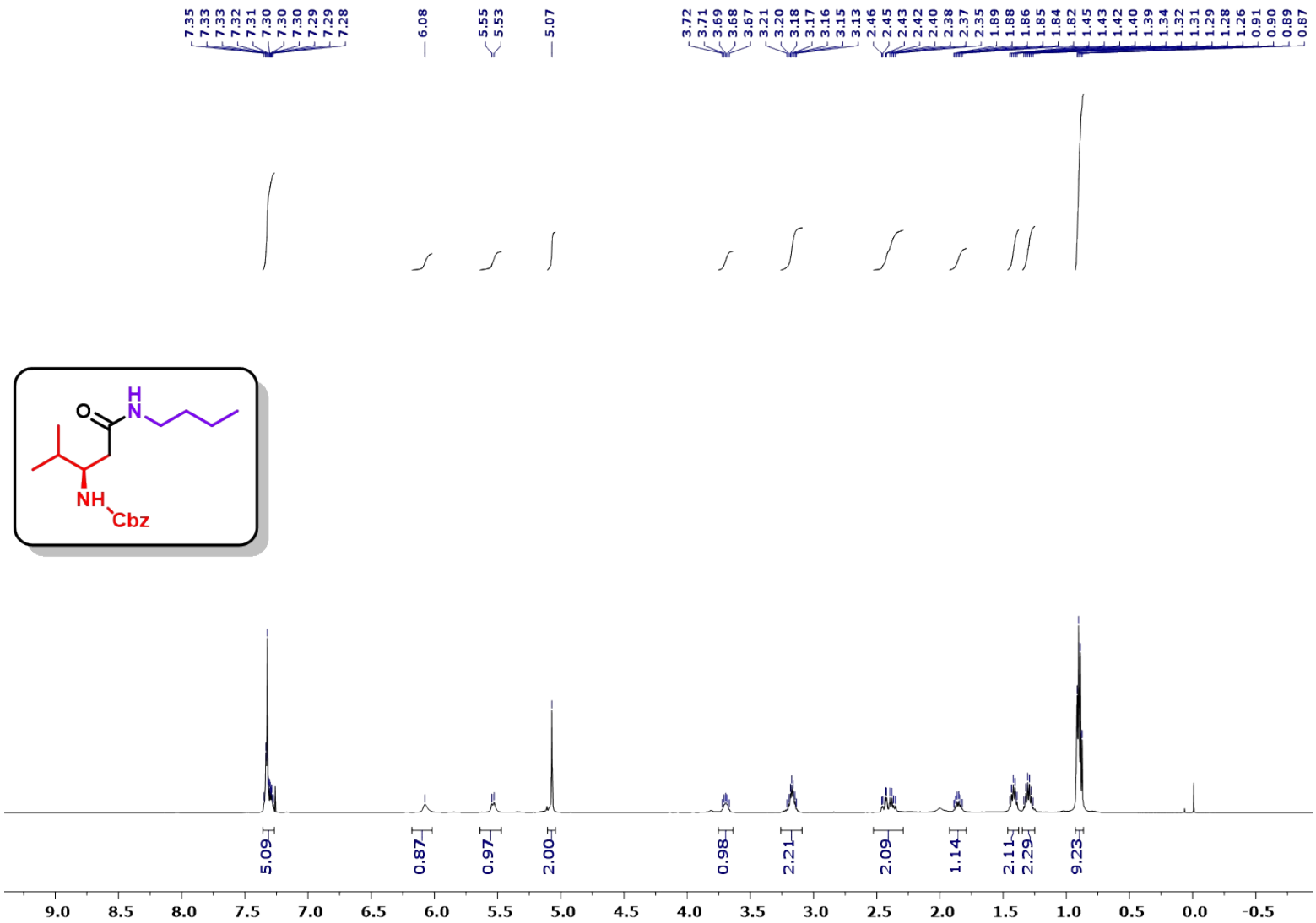

Fig. S74. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of benzyl (*R*)-(1-(butylamino)-4-methyl-1-oxopentan-3-yl) carbamate (**6bj**).

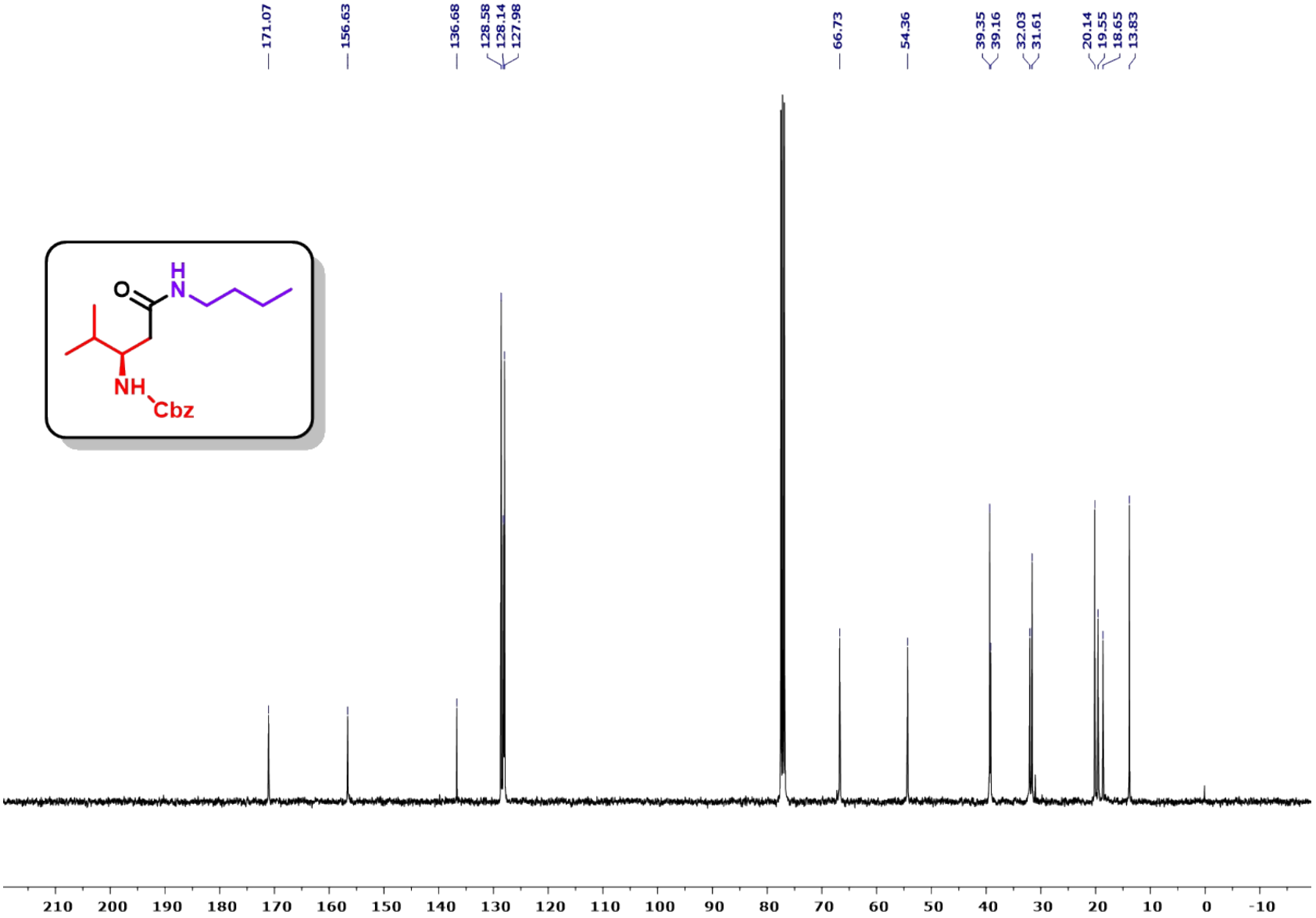

Fig. S75. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of benzyl (*R*)-(1-(butylamino)-4-methyl-1-oxopentan-3-yl) carbamate (**6bj**).

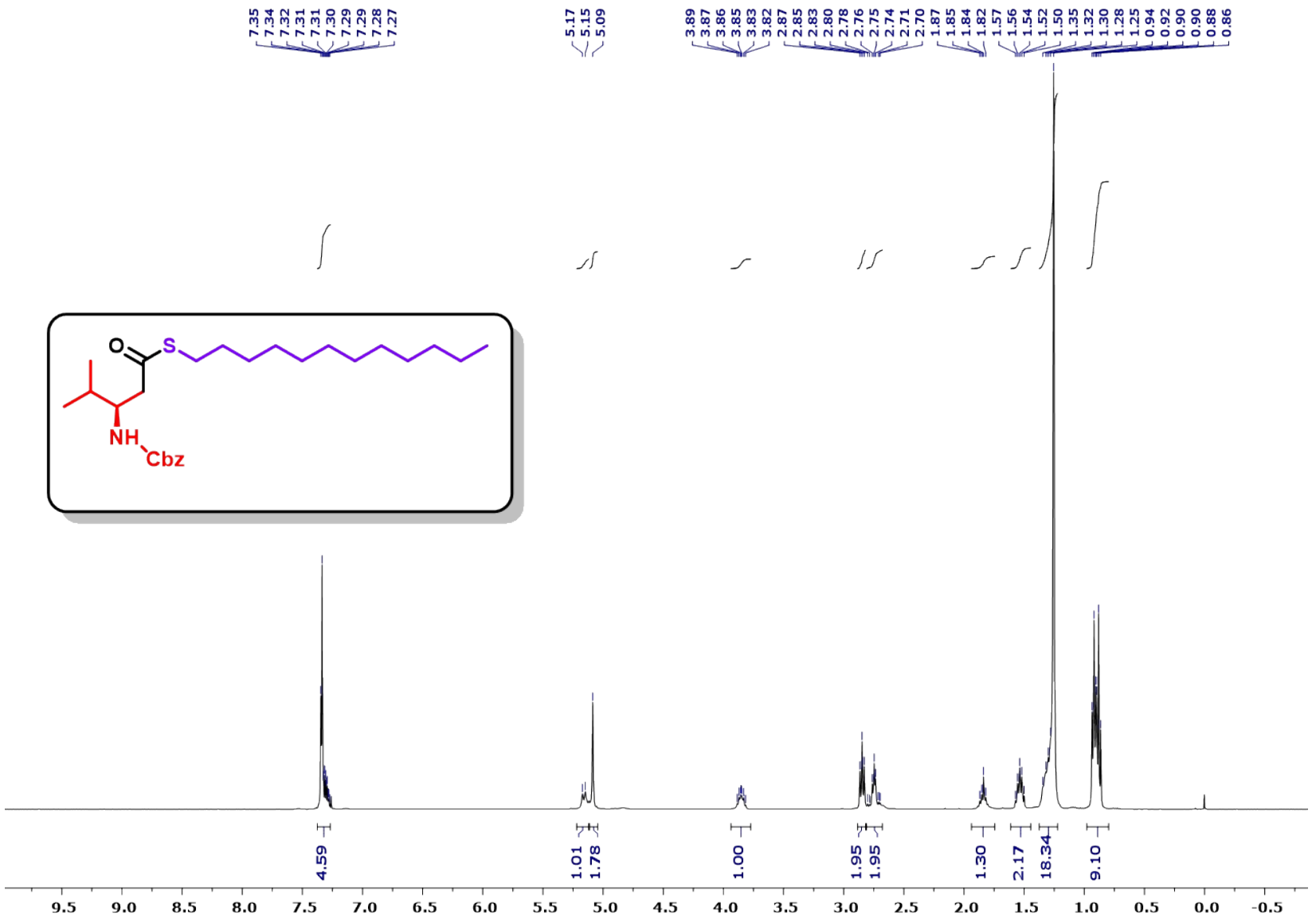

Fig. S76. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of *S*-dodecyl (*R*)-3-(((benzyloxy)carbonyl) amino)-4-methylpentanethioate (**6br**).

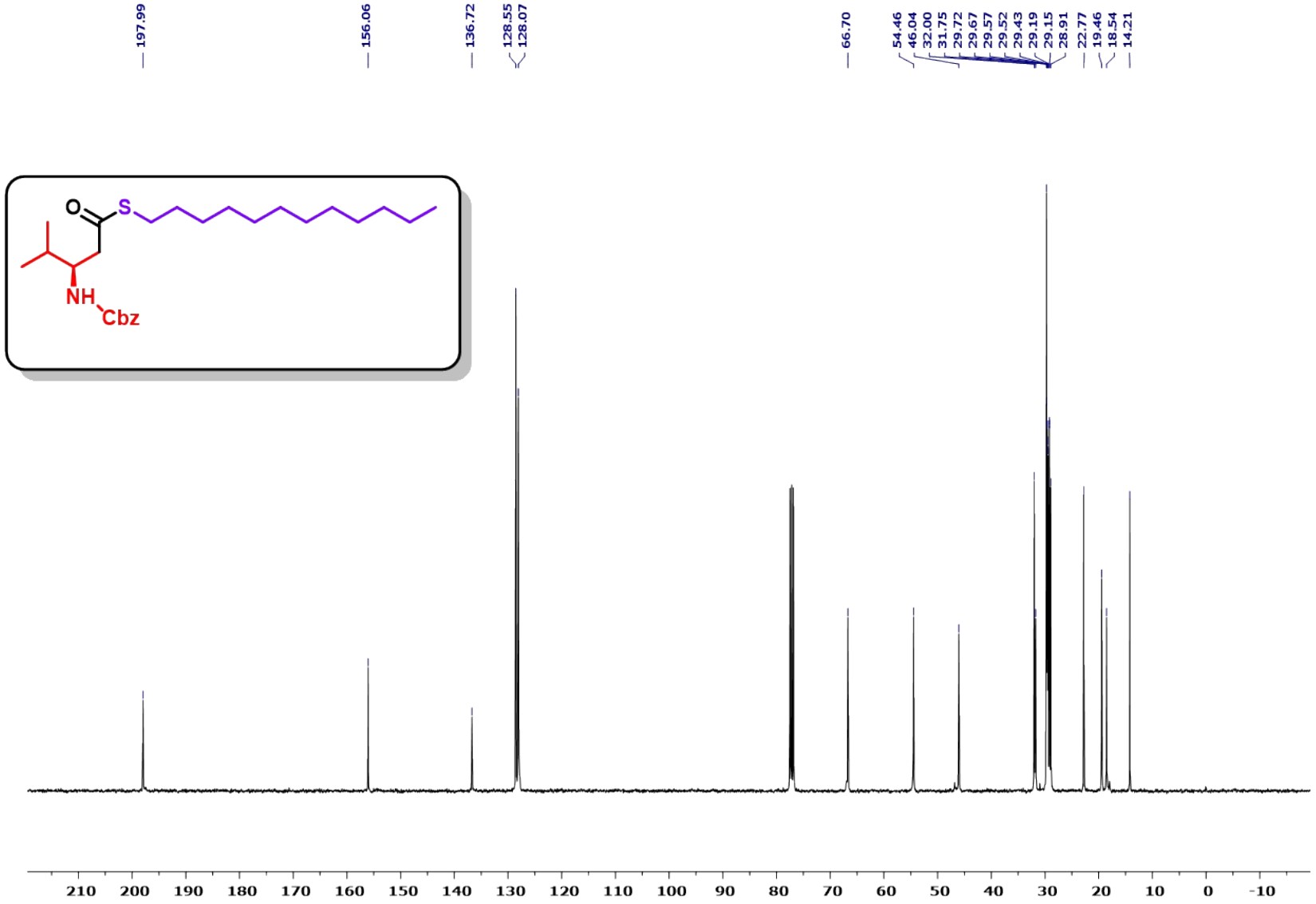

Fig. S77. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of *S*-dodecyl (*R*)-3-(((benzyloxy)carbonyl) amino)-4-methylpentanethioate (**6br**).

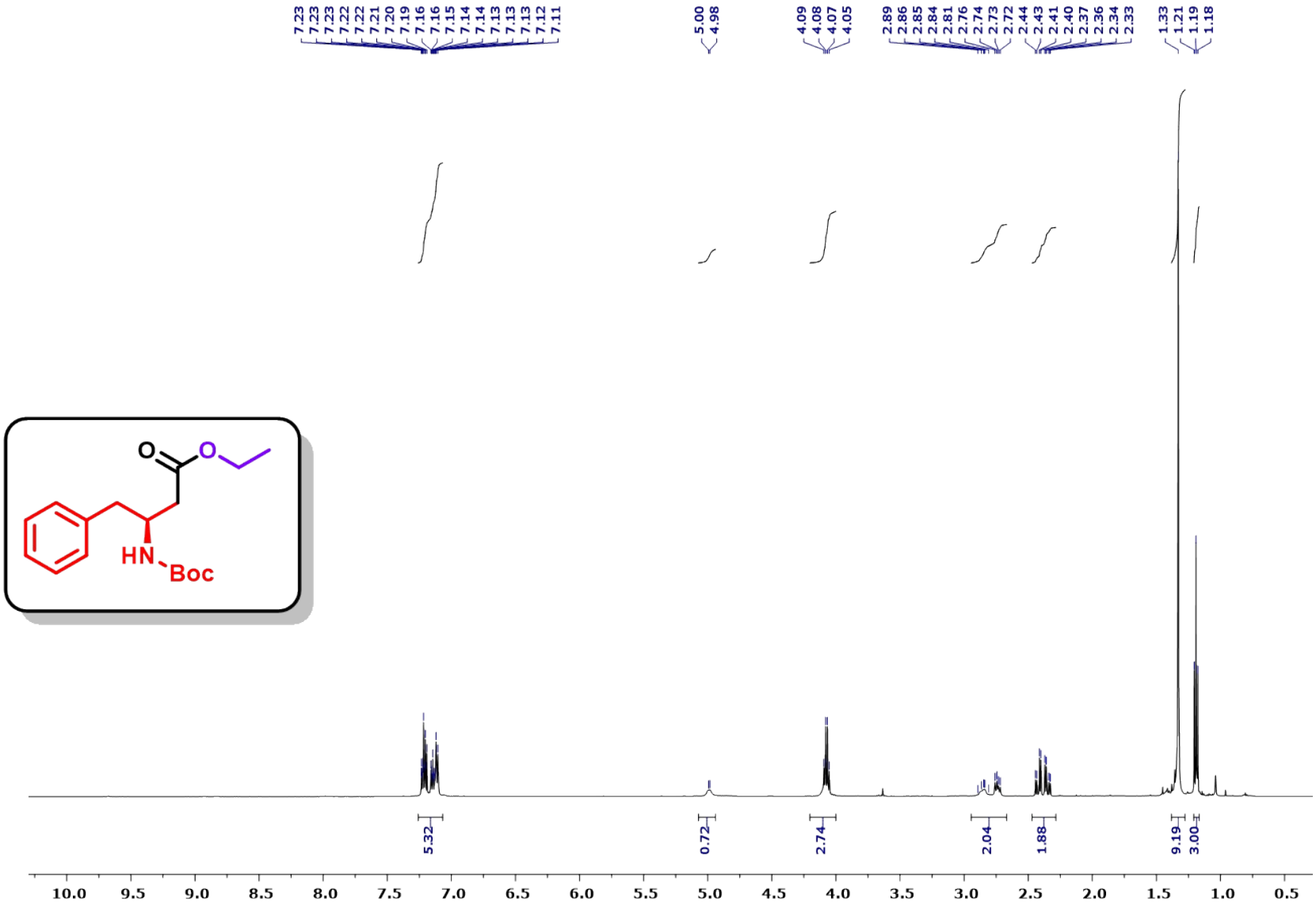

Fig. S78. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of ethyl (*S*)-3-((tert-butoxycarbonyl) amino)-4-phenylbutanoate (**6ca**).

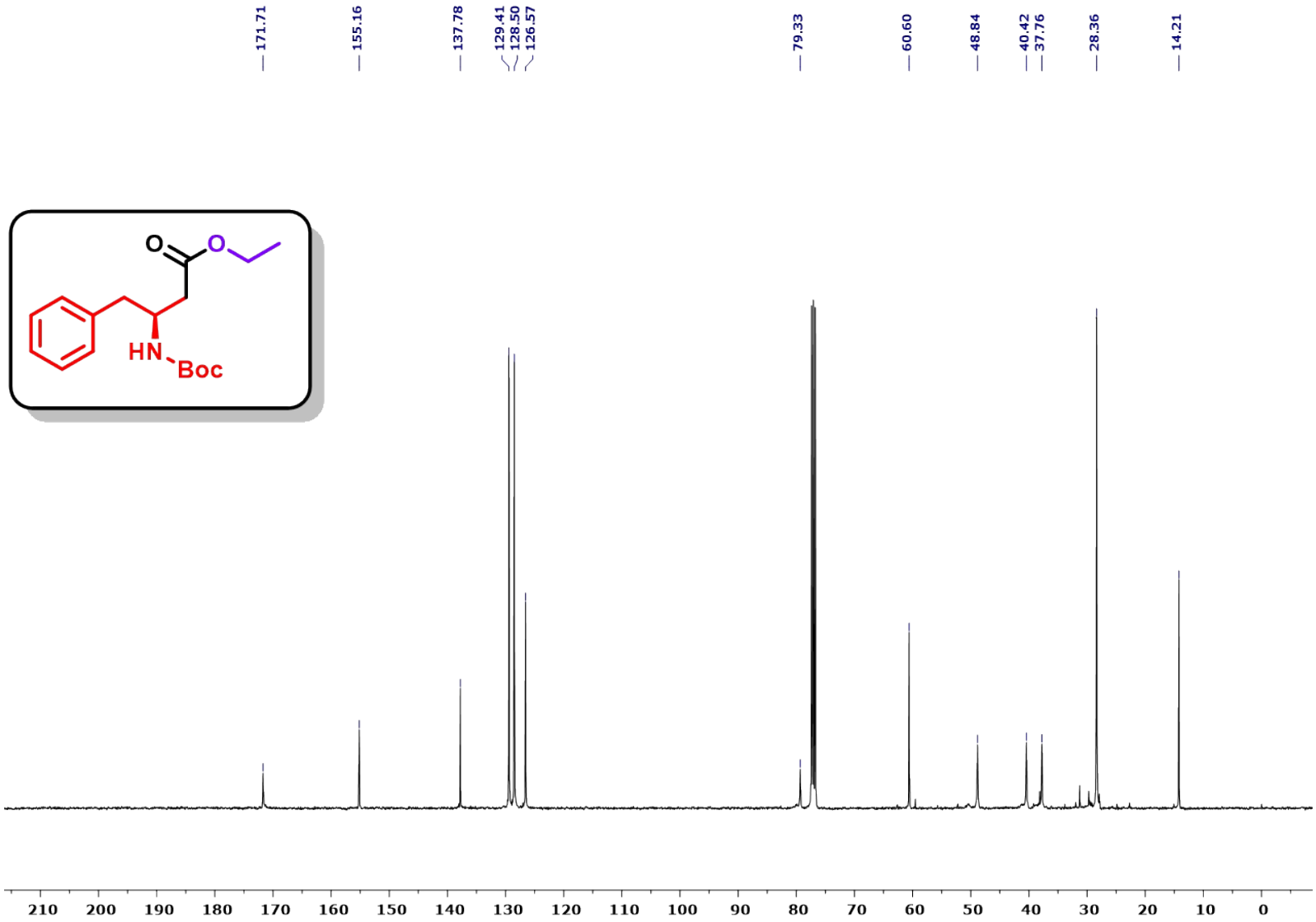

Fig. S79. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of ethyl (S)-3-((tert-butoxycarbonyl) amino)-4-phenylbutanoate (**6ca**).

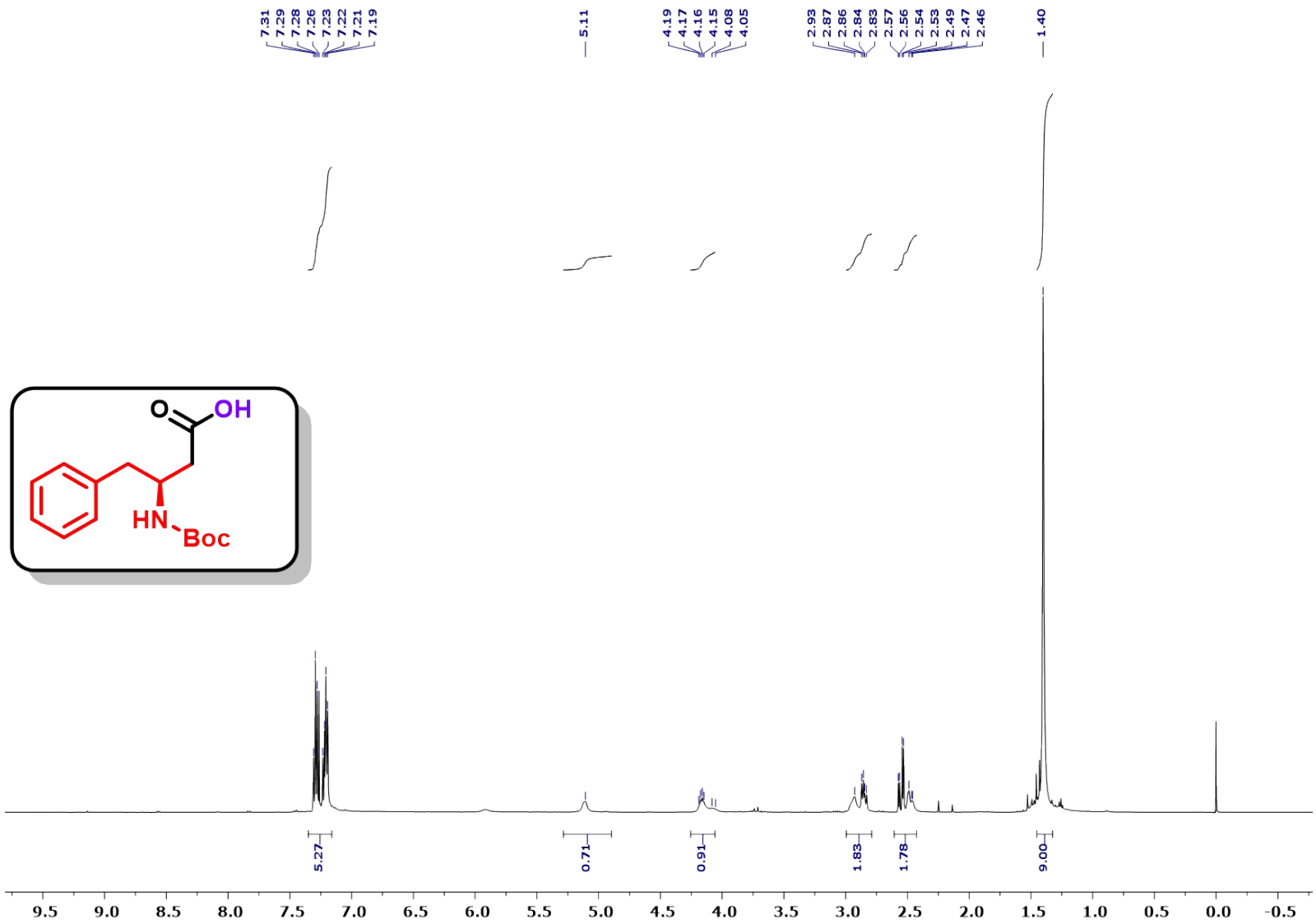

Fig. S80. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of (*S*)-3-((*tert*-butoxycarbonyl) amino)-4-phenylbutanoic acid (**6cc**).

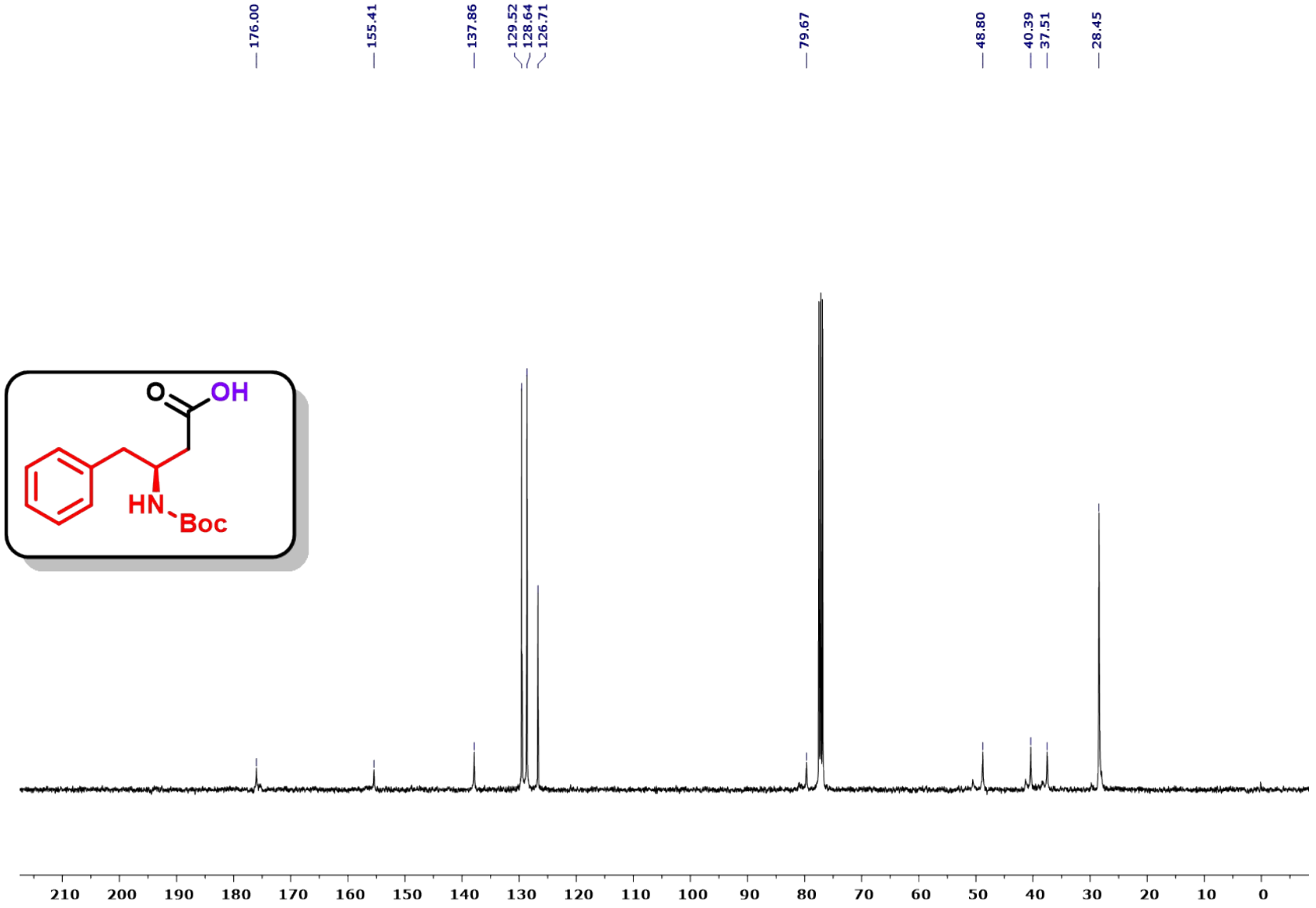

Fig. S81. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of (*S*)-3-((*tert*-butoxycarbonyl) amino)-4-phenylbutanoic acid (**6cc**).

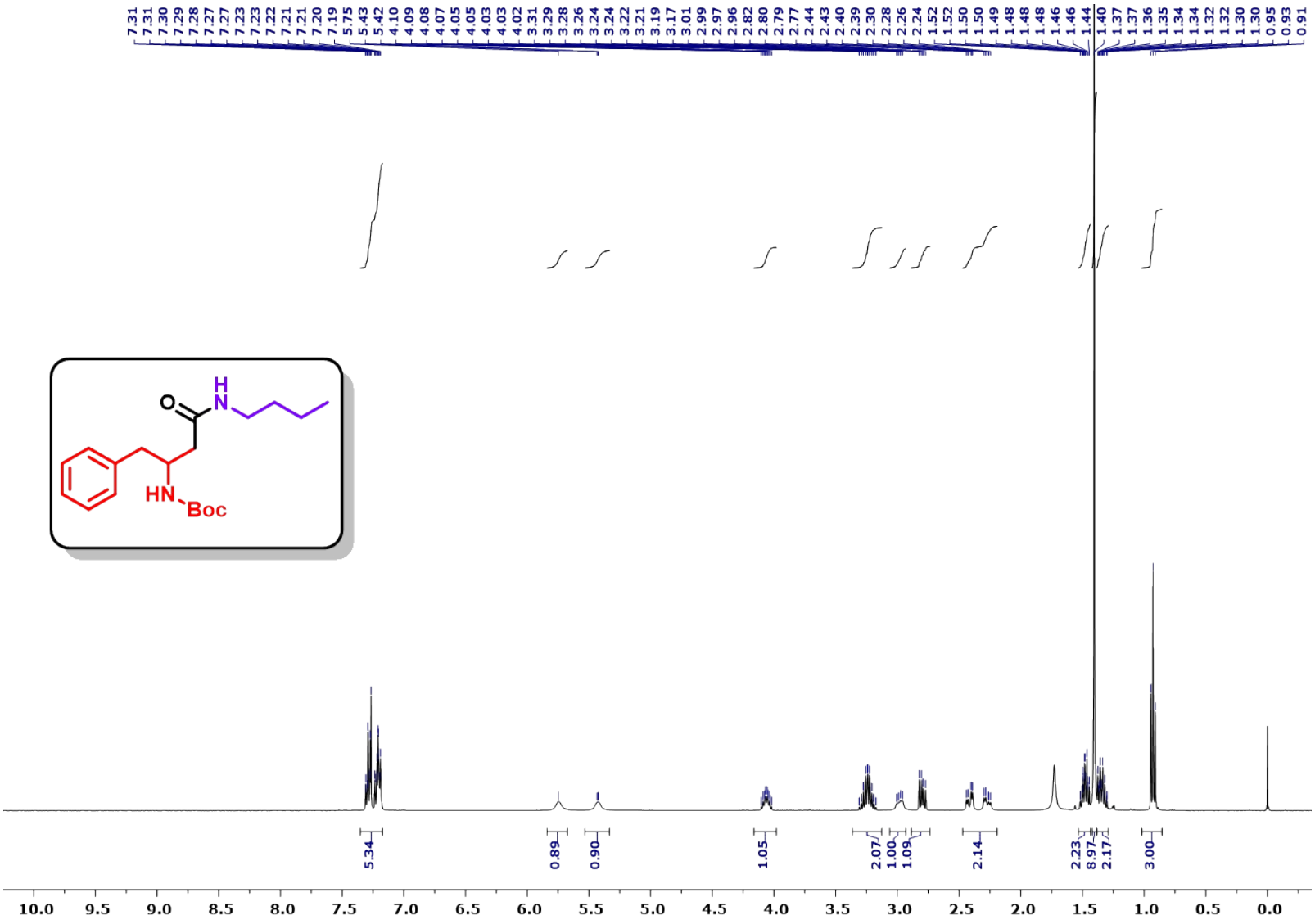

Fig. S82. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of *tert*-butyl (*S*)-(4-(butylamino)-4-oxo-1-phenylbutan-2-yl) carbamate (**6cj**).

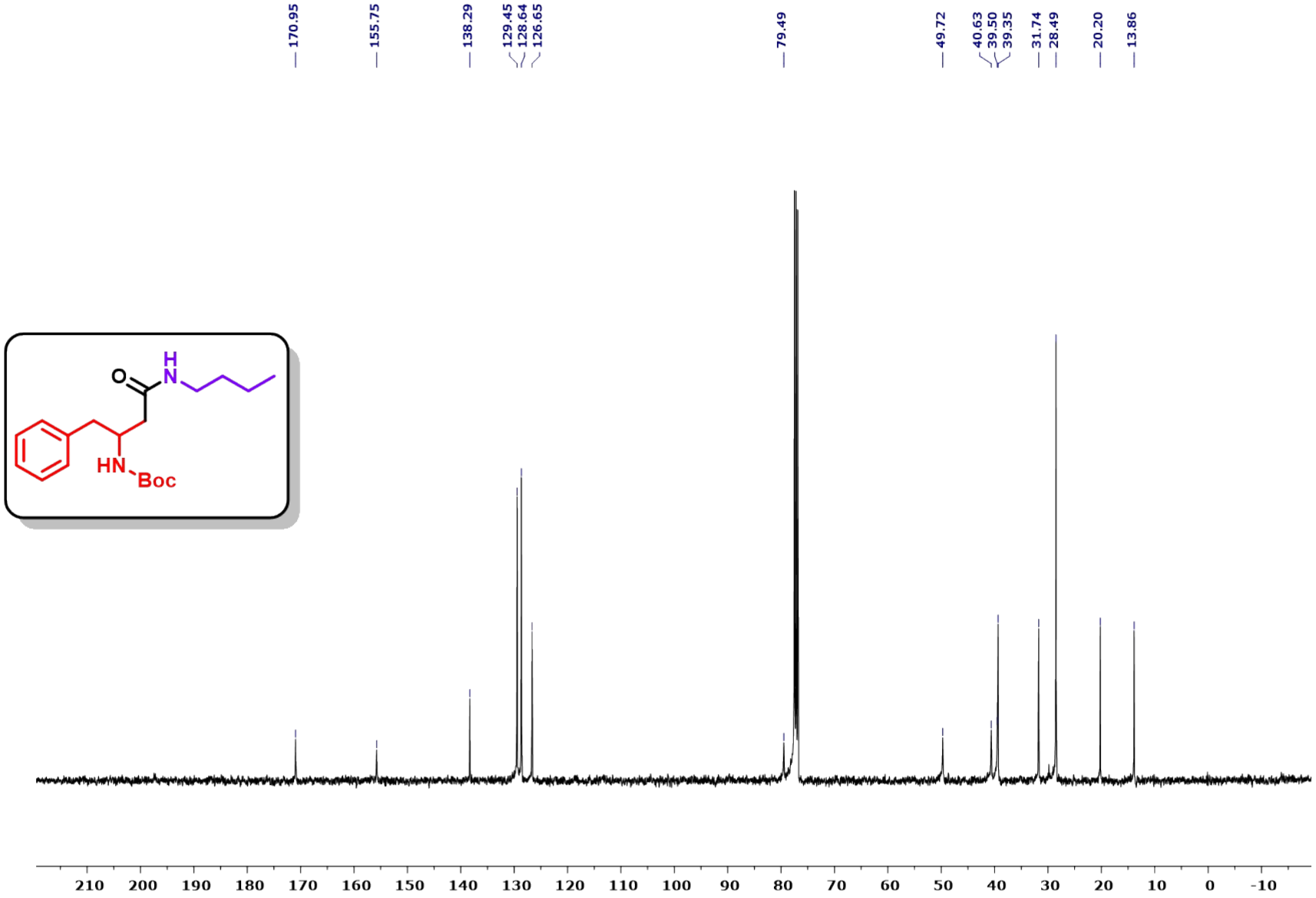

Fig. S83. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of *tert*-butyl (S)-(4-(butylamino)-4-oxo-1-phenylbutan-2-yl) carbamate (**6cj**).

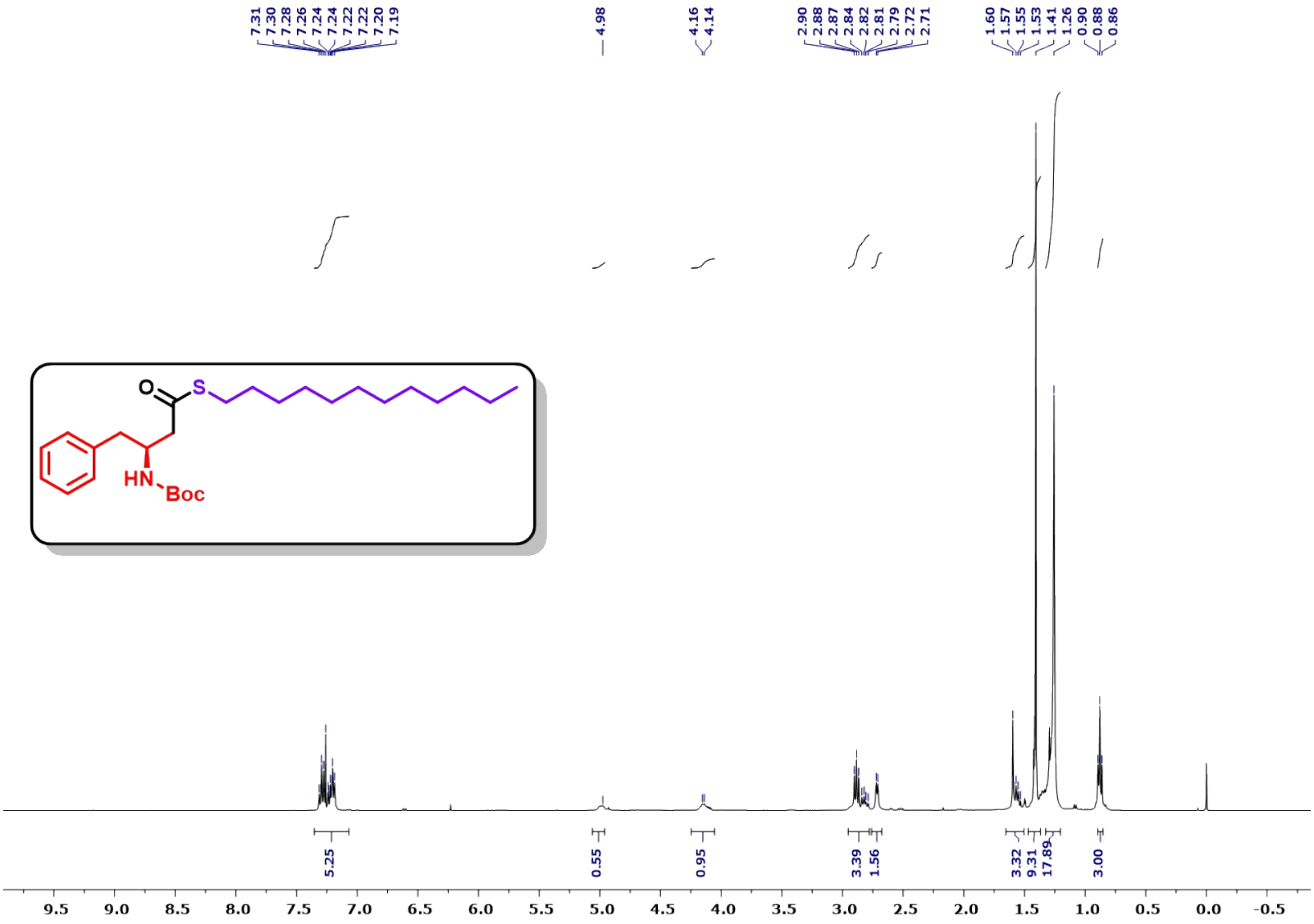

Fig. S84. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of *S*-dodecyl (*S*)-3-((*tert*-butoxycarbonyl) amino)-4-phenylbutanethioate (**6cr**).

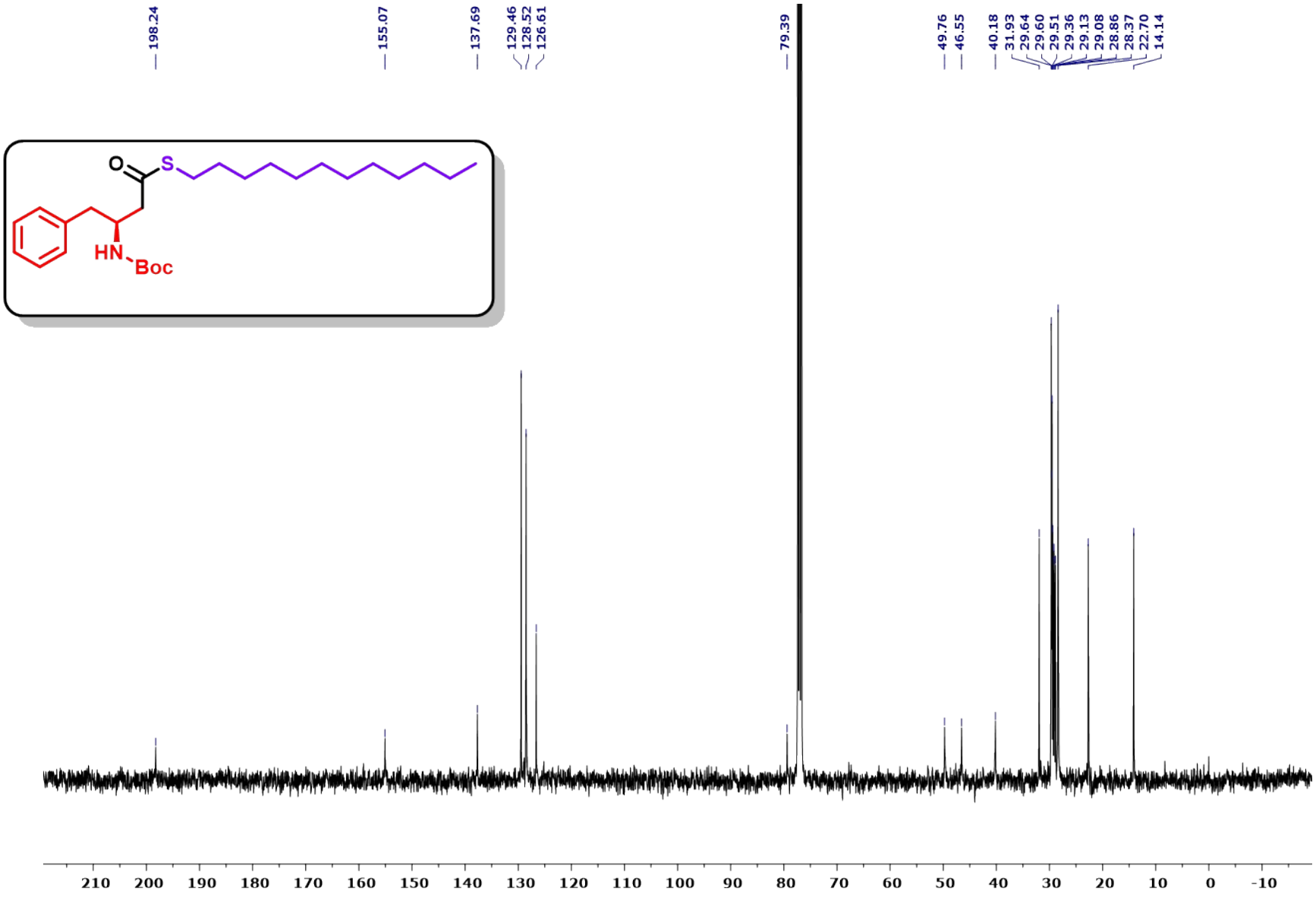

Fig. S85. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of *S*-dodecyl (*S*)-3-((*tert*-butoxycarbonyl) amino)-4-phenylbutanethioate (**6cr**).

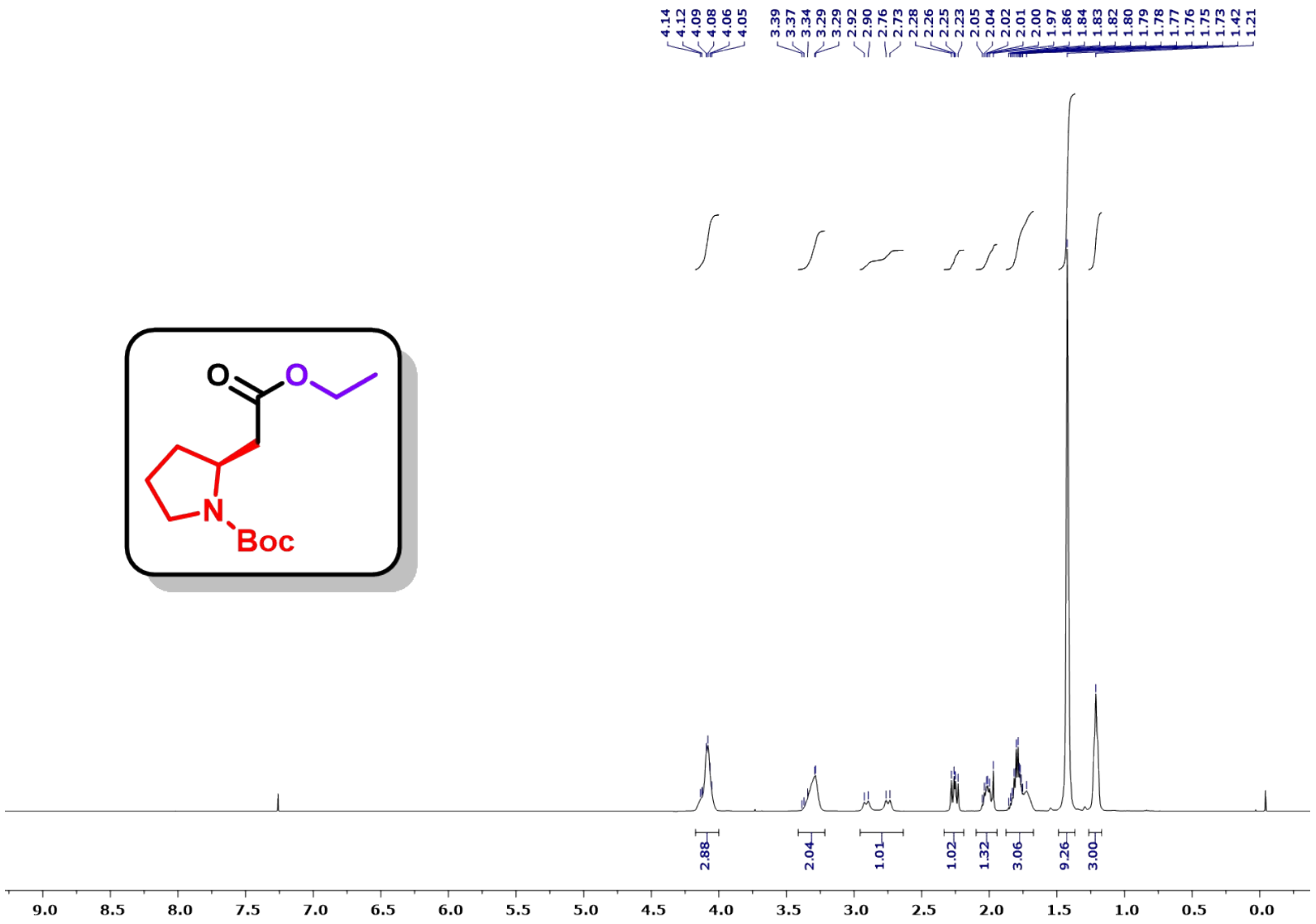

Fig. S86. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of *tert*-butyl (*S*)-2-(2-ethoxy-2-oxoethyl) pyrrolidine-1-carboxylate (**6da**).

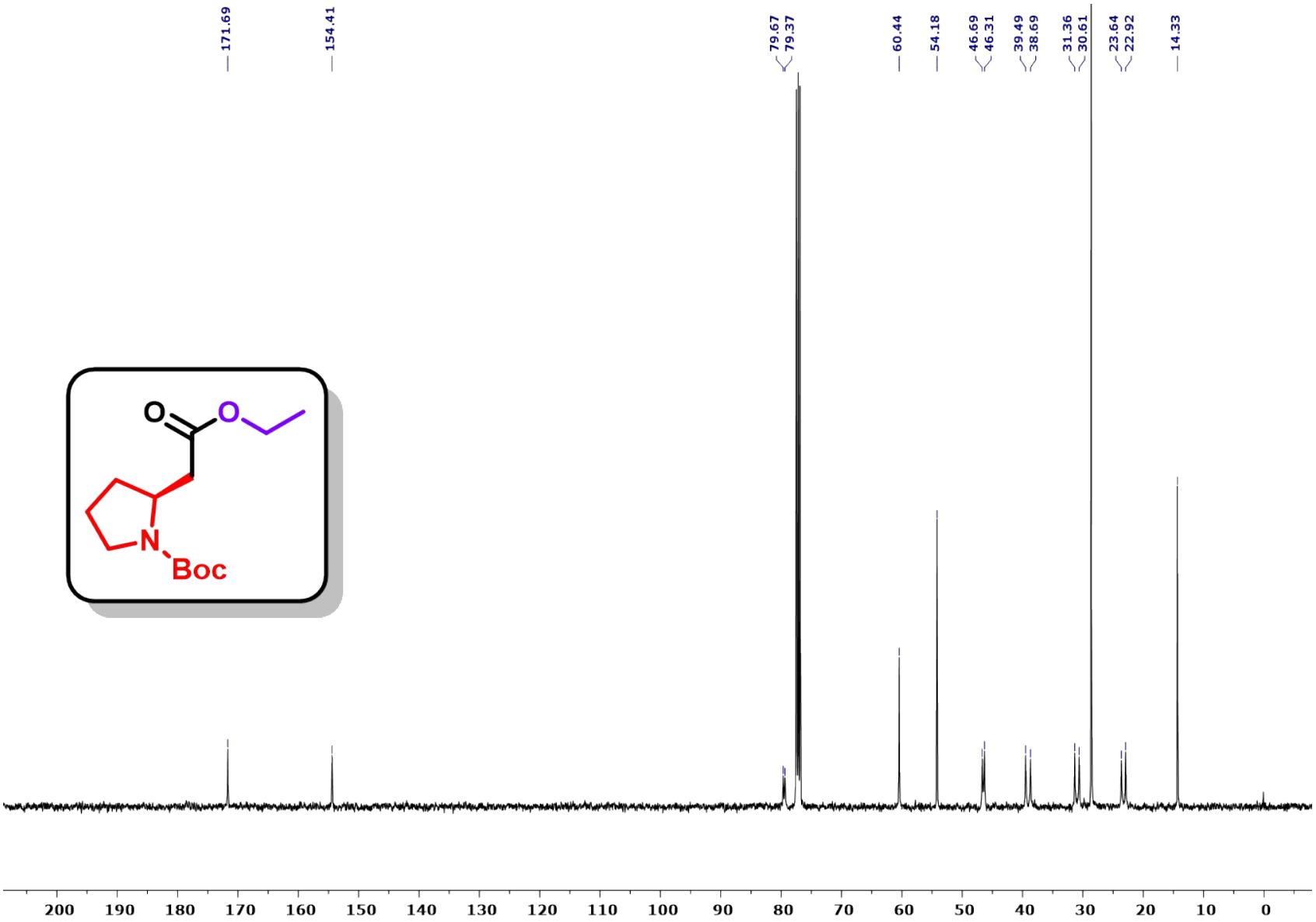

Fig. S87. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of *tert*-butyl (*S*)-2-(2-ethoxy-2-oxoethyl) pyrrolidine-1-carboxylate (**6da**).

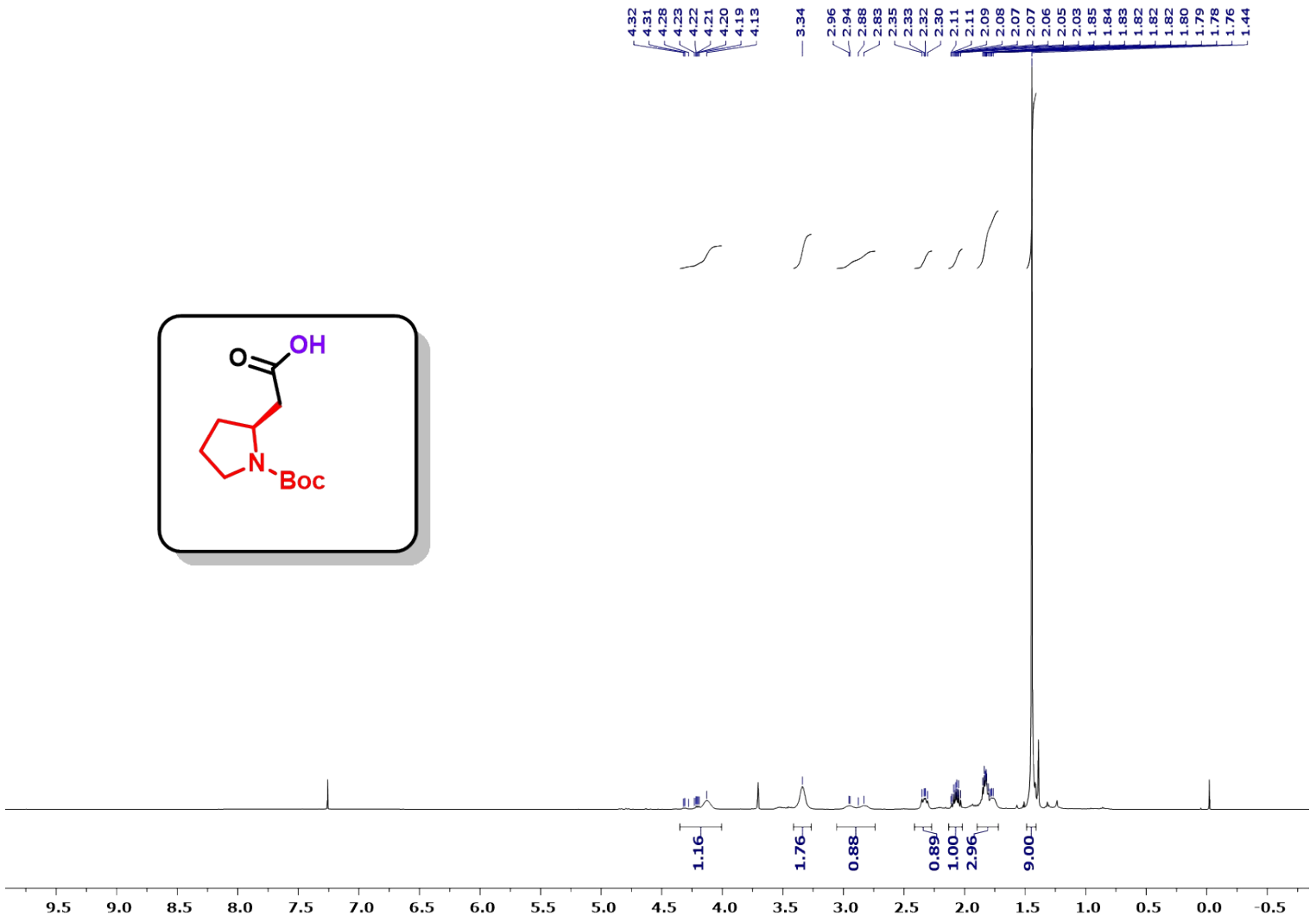

Fig. S88. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of (*S*)-2-(1-(*tert*-butoxycarbonyl) pyrrolidin-2-yl) acetic acid (**6dc**).

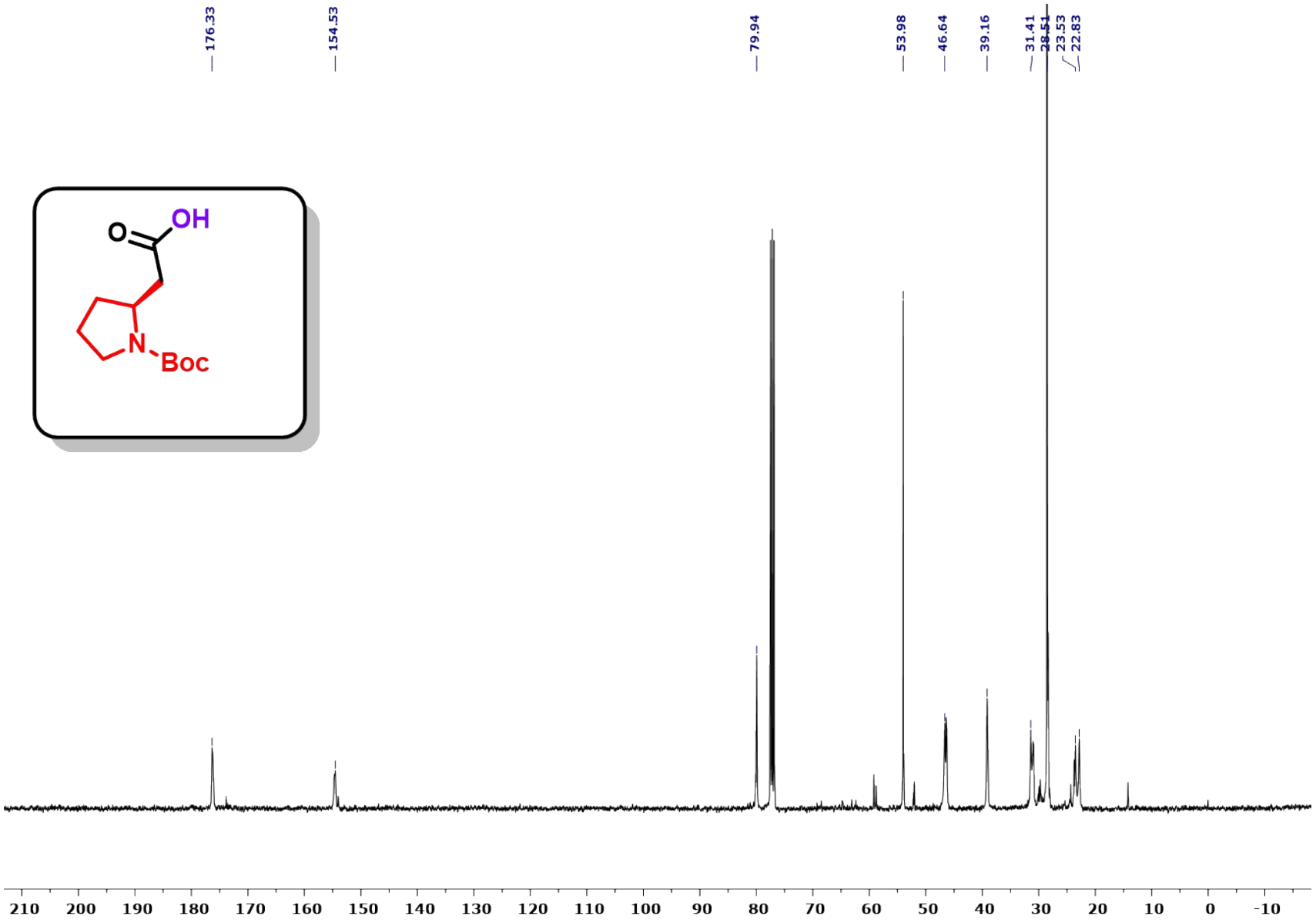

Fig. S89. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of (*S*)-2-(1-(*tert*-butoxycarbonyl) pyrrolidin-2-yl) acetic acid (**6dc**).

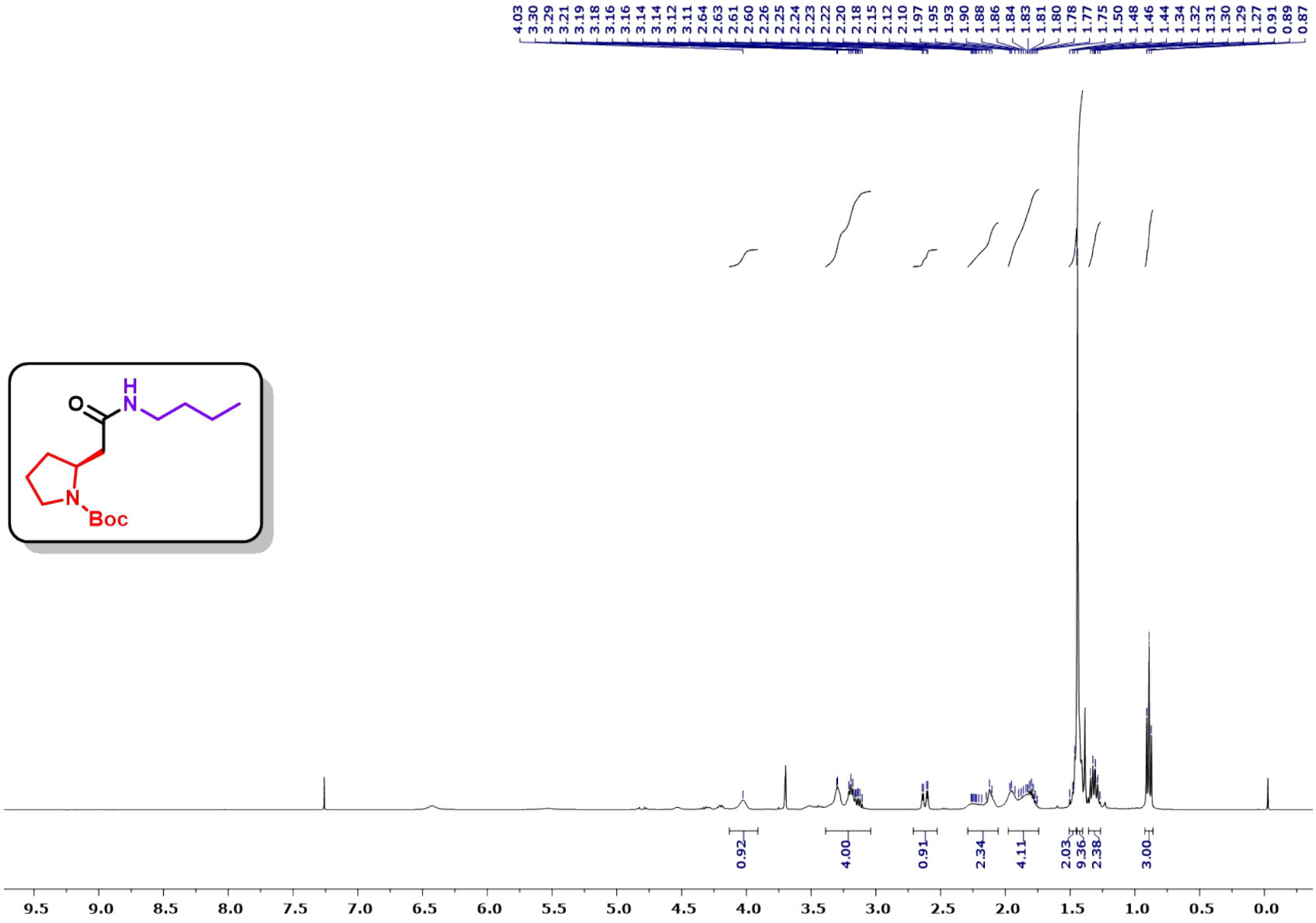

Fig. S90. ^{1}H NMR (400 MHz, $CDCl_3$) spectra of *tert*-butyl (*S*)-2-(2-(butylamino)-2-oxoethyl) pyrrolidine-1-carboxylate (**6dj**).

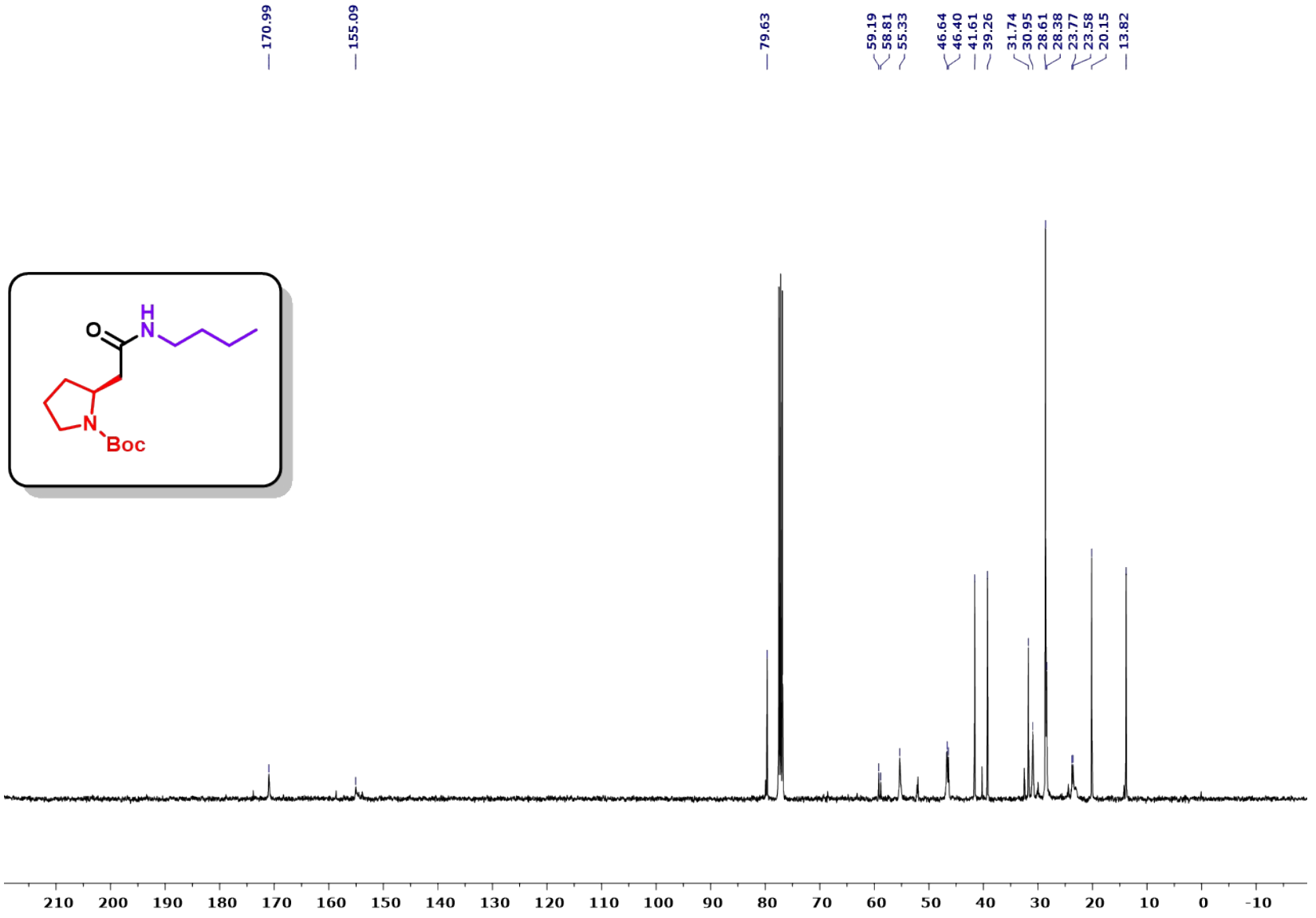

Fig. S91. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of *tert*-butyl (*S*)-2-(2-(butylamino)-2-oxoethyl) pyrrolidine-1-carboxylate (**6dj**).

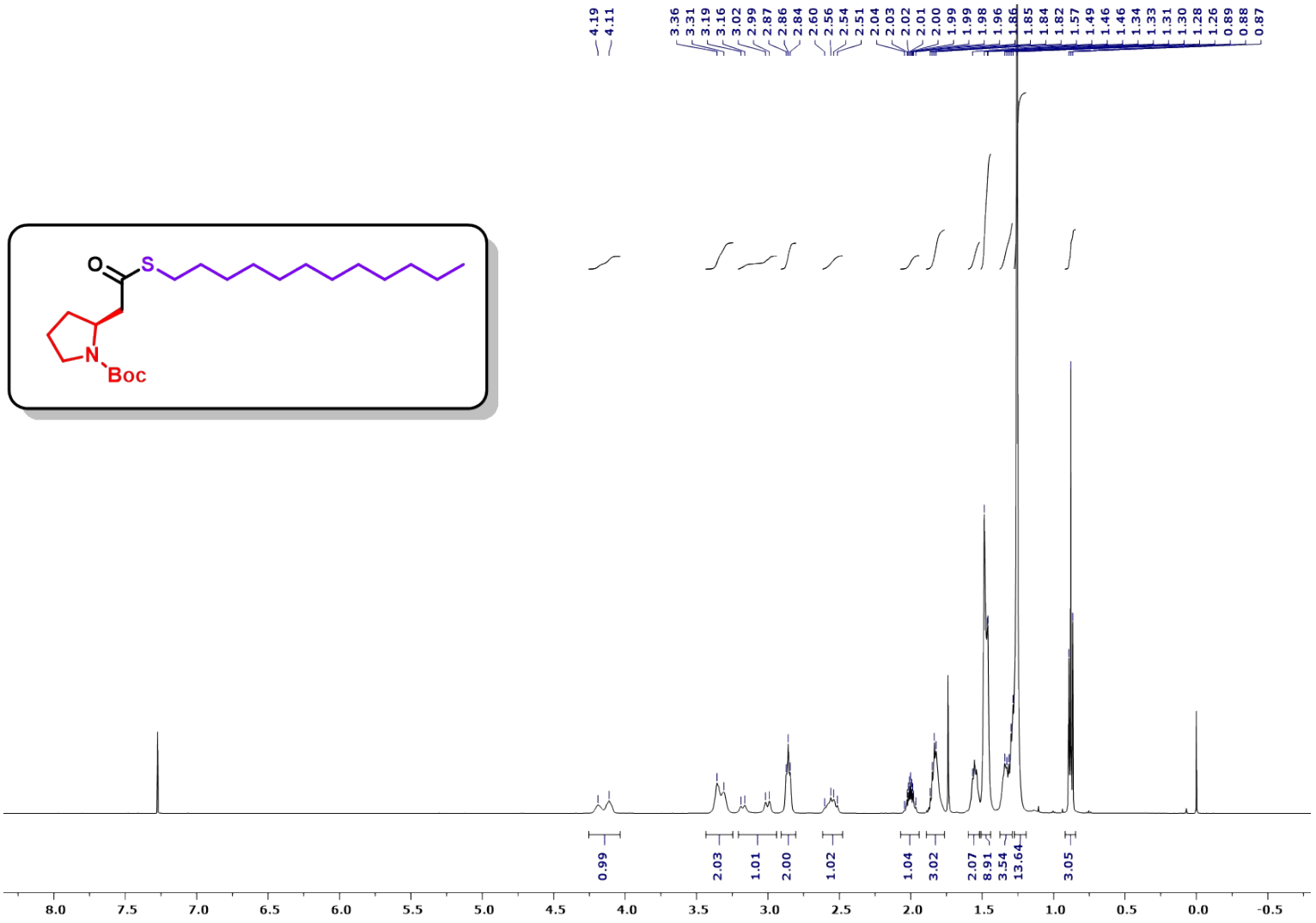

Fig. S92. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of *tert*-butyl (*S*)-2-(2-(dodecylthio)-2-oxoethyl) pyrrolidine-1-carboxylate (**6dr**).

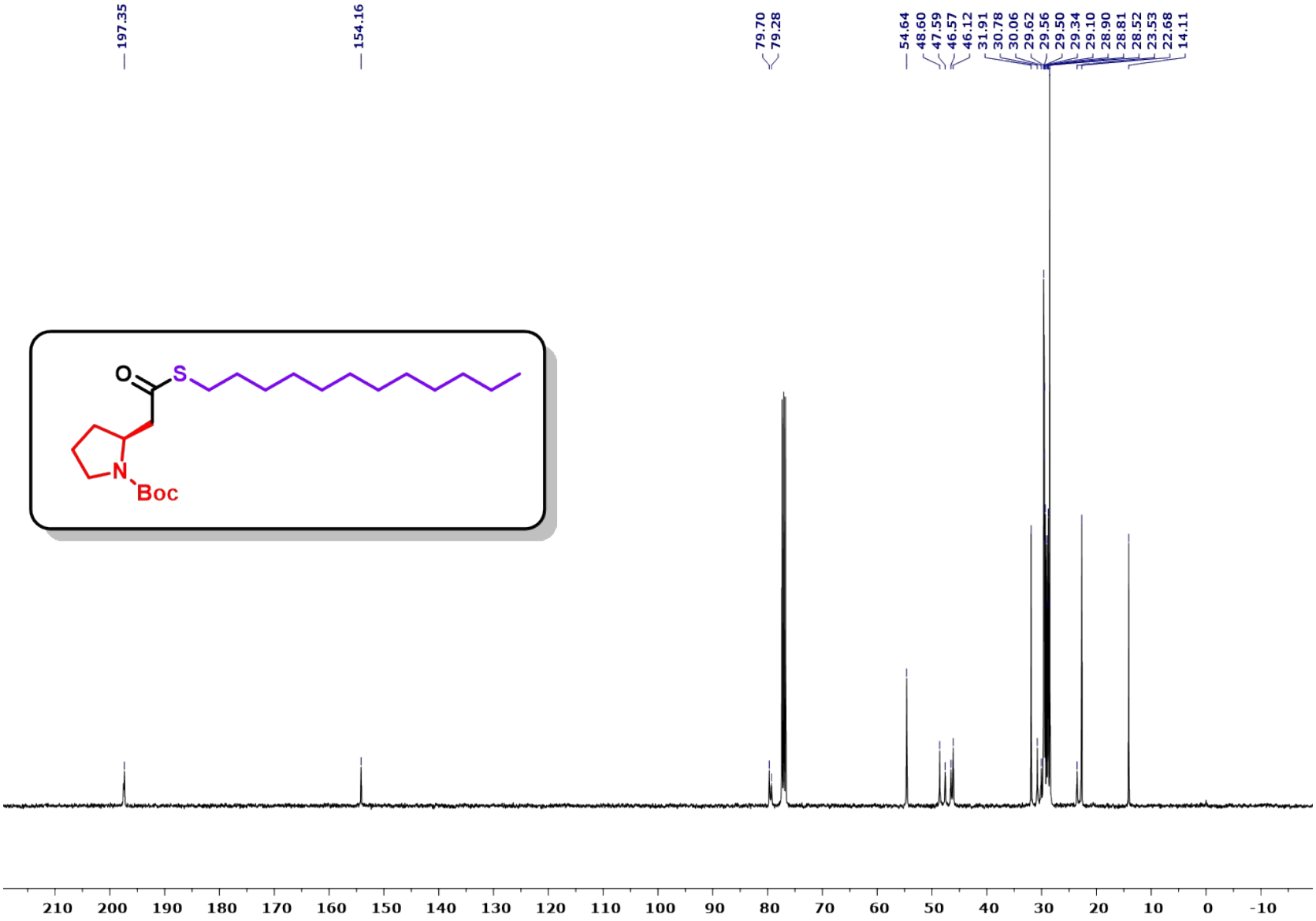

Fig. S93. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of *tert*-butyl (*S*)-2-(2-(dodecylthio)-2-oxoethyl) pyrrolidine-1-carboxylate (**6dr**).

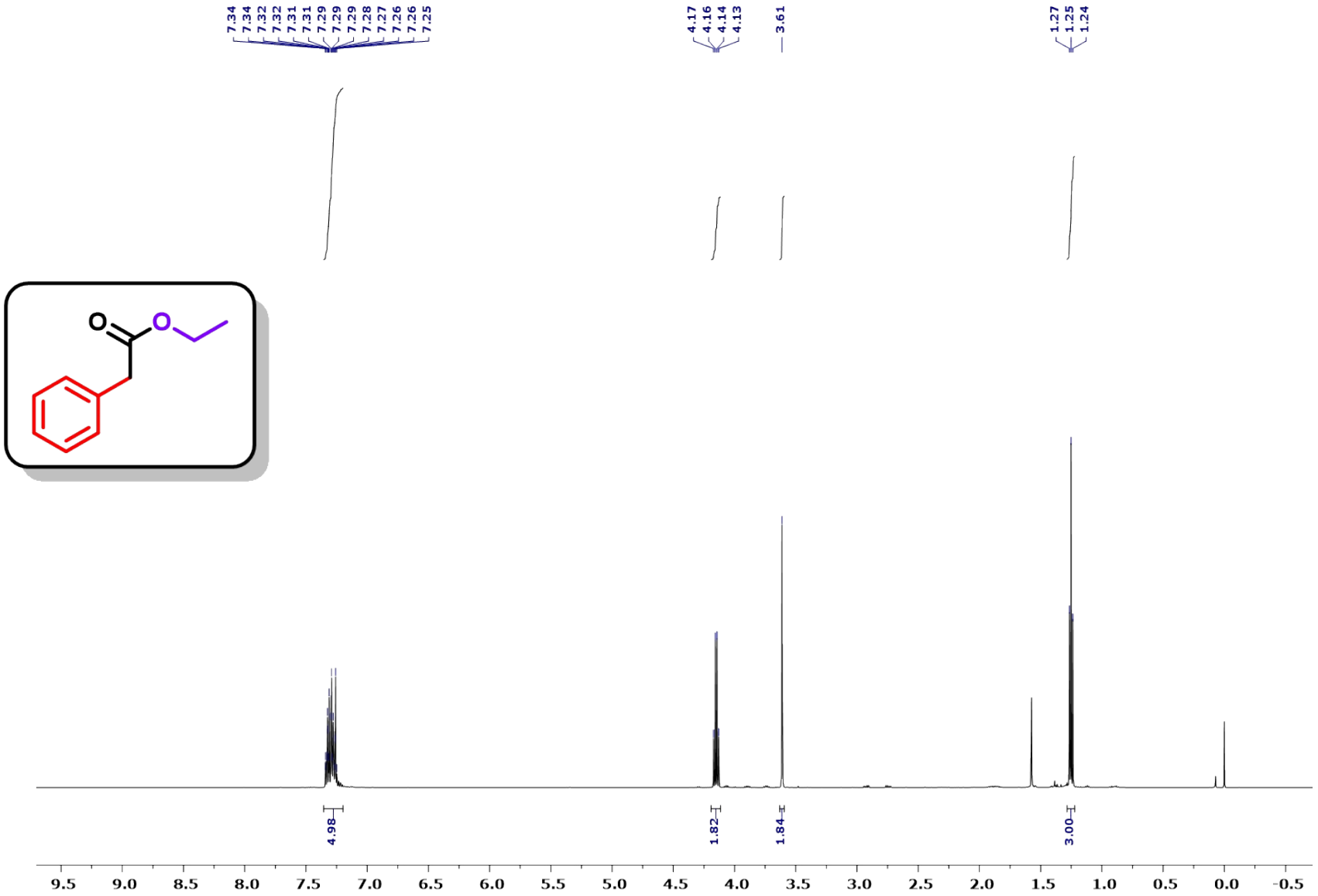

Fig. S94. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of ethyl 2-phenylacetate (**6ea**).

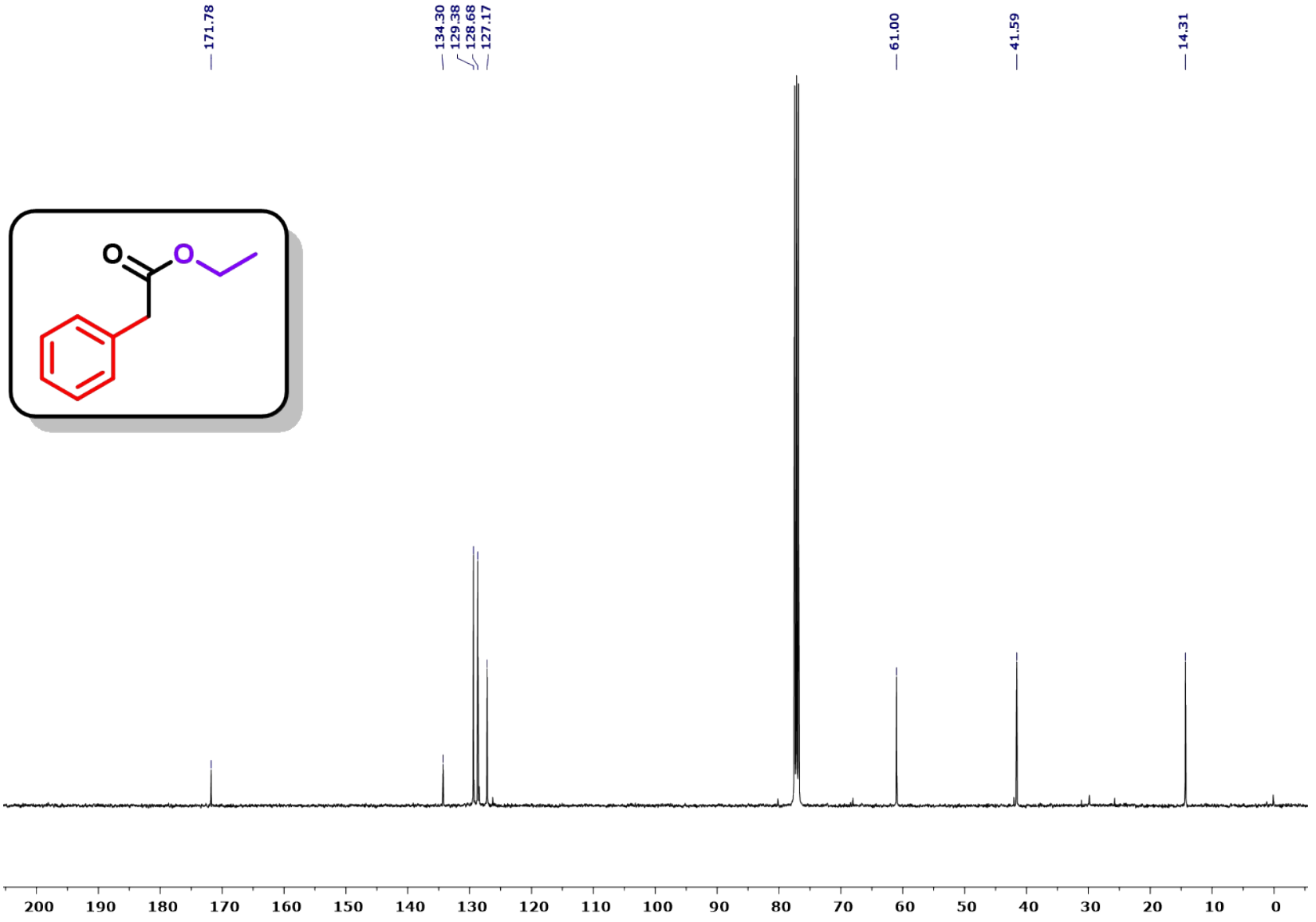

Fig. S95. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of ethyl 2-phenylacetate (**6ea**).

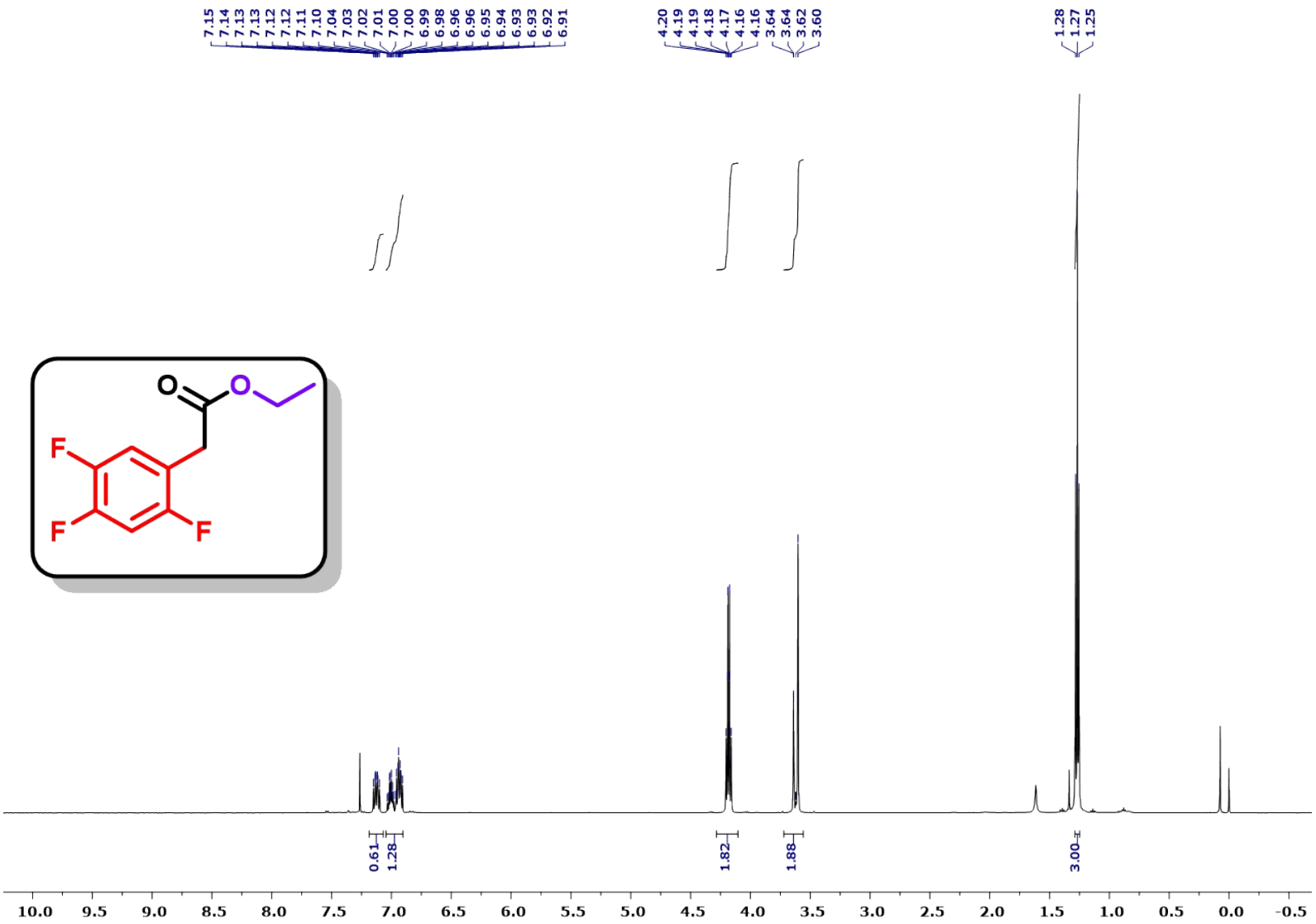

Fig. S96. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of ethyl 2-(2,4,5-trifluorophenyl) acetate (**6fa**).

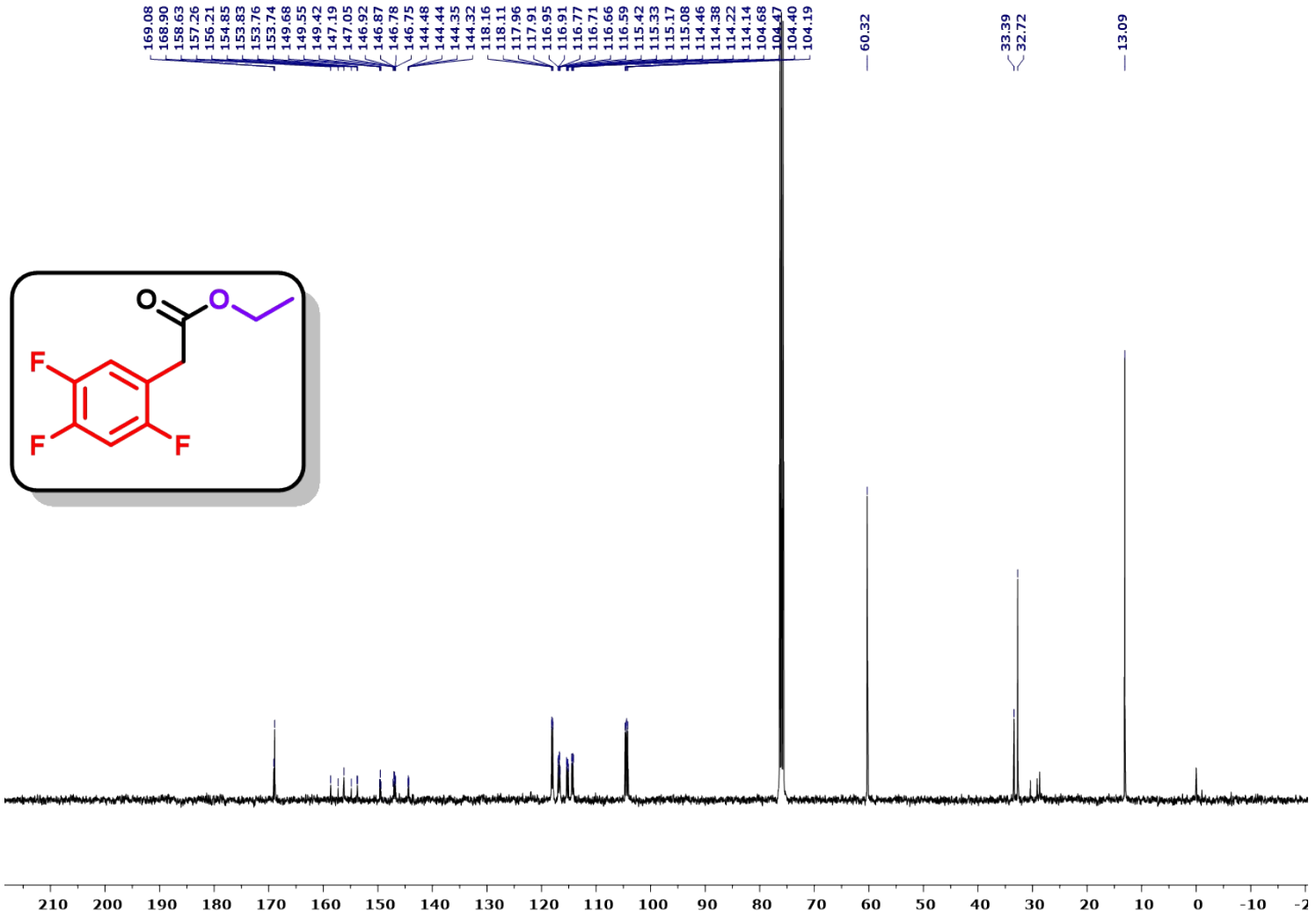

Fig. S97. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of ethyl 2-(2,4,5-trifluorophenyl) acetate (**6fa**).

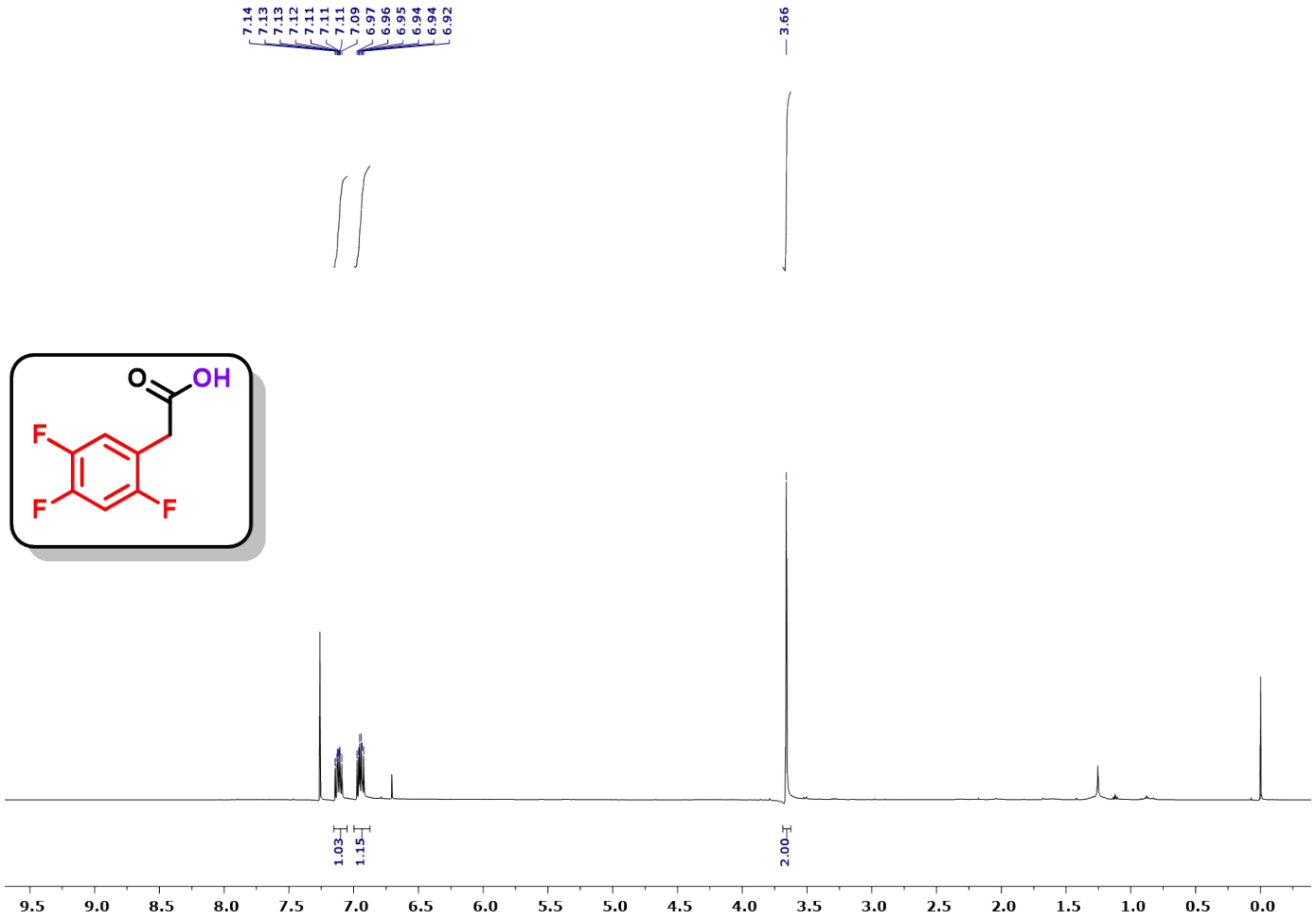

Fig. S98. ^{1}H NMR (500 MHz, $CDCl_3$) spectra of 2-(2,4,5-trifluorophenyl) acetic acid (**6fc**).

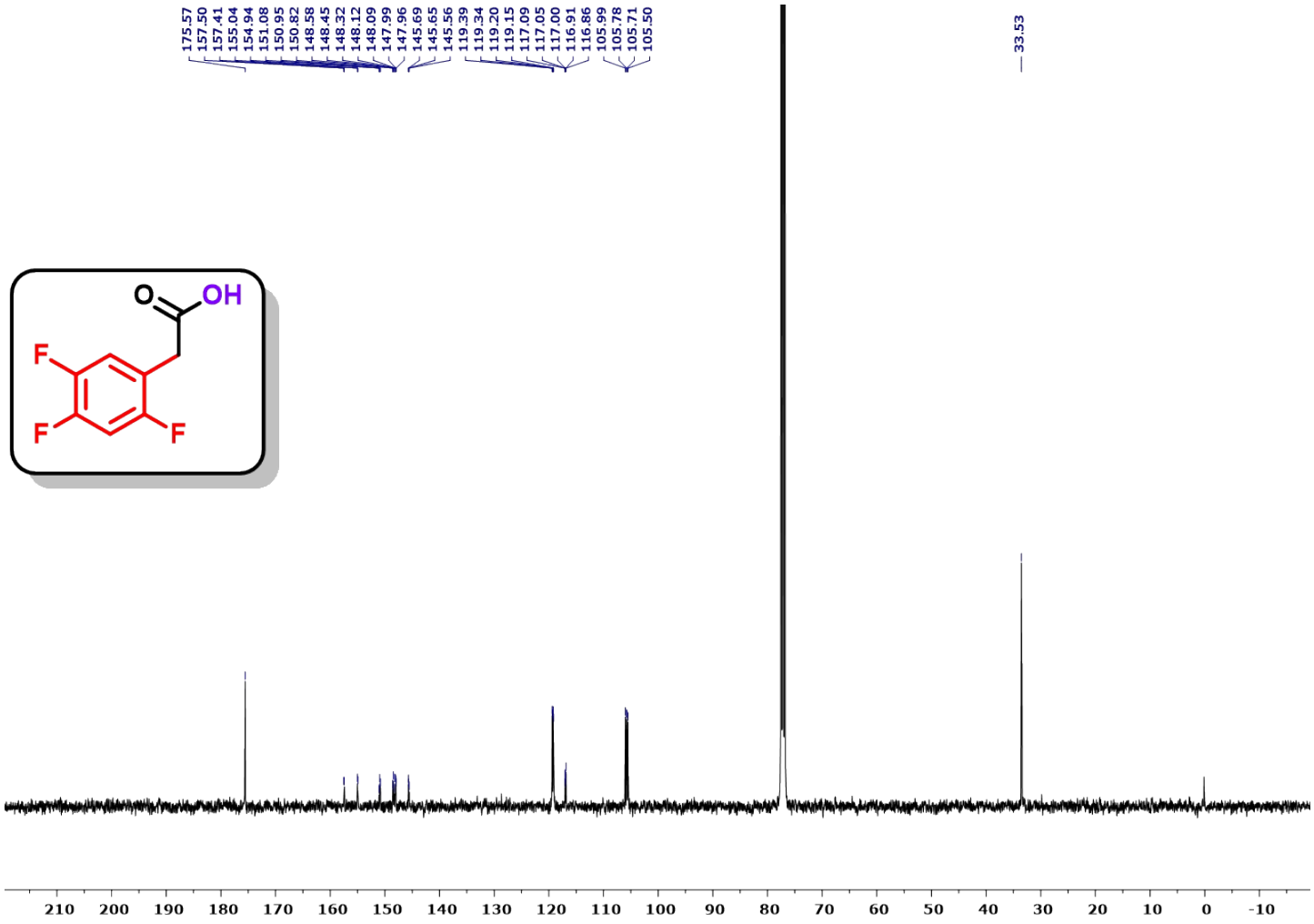

Fig. S99. ^{13}C NMR (101 MHz, $CDCl_3$) spectra of 2-(2,4,5-trifluorophenyl) acetic acid (**6fc**).

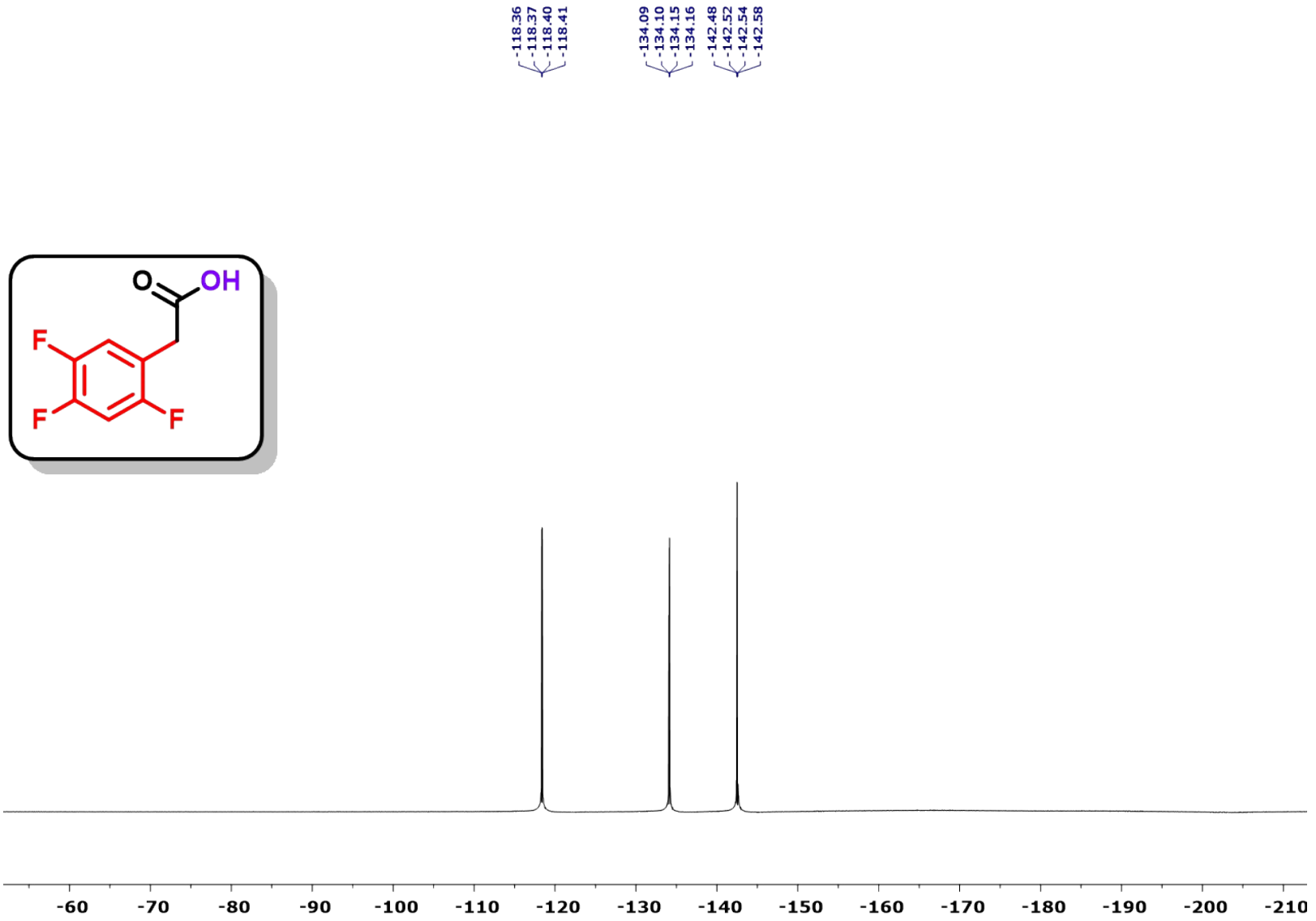

Fig. S100. ^{19}F NMR (376 MHz, $CDCl_3$) spectra of 2-(2,4,5-trifluorophenyl) acetic acid (**6fc**).

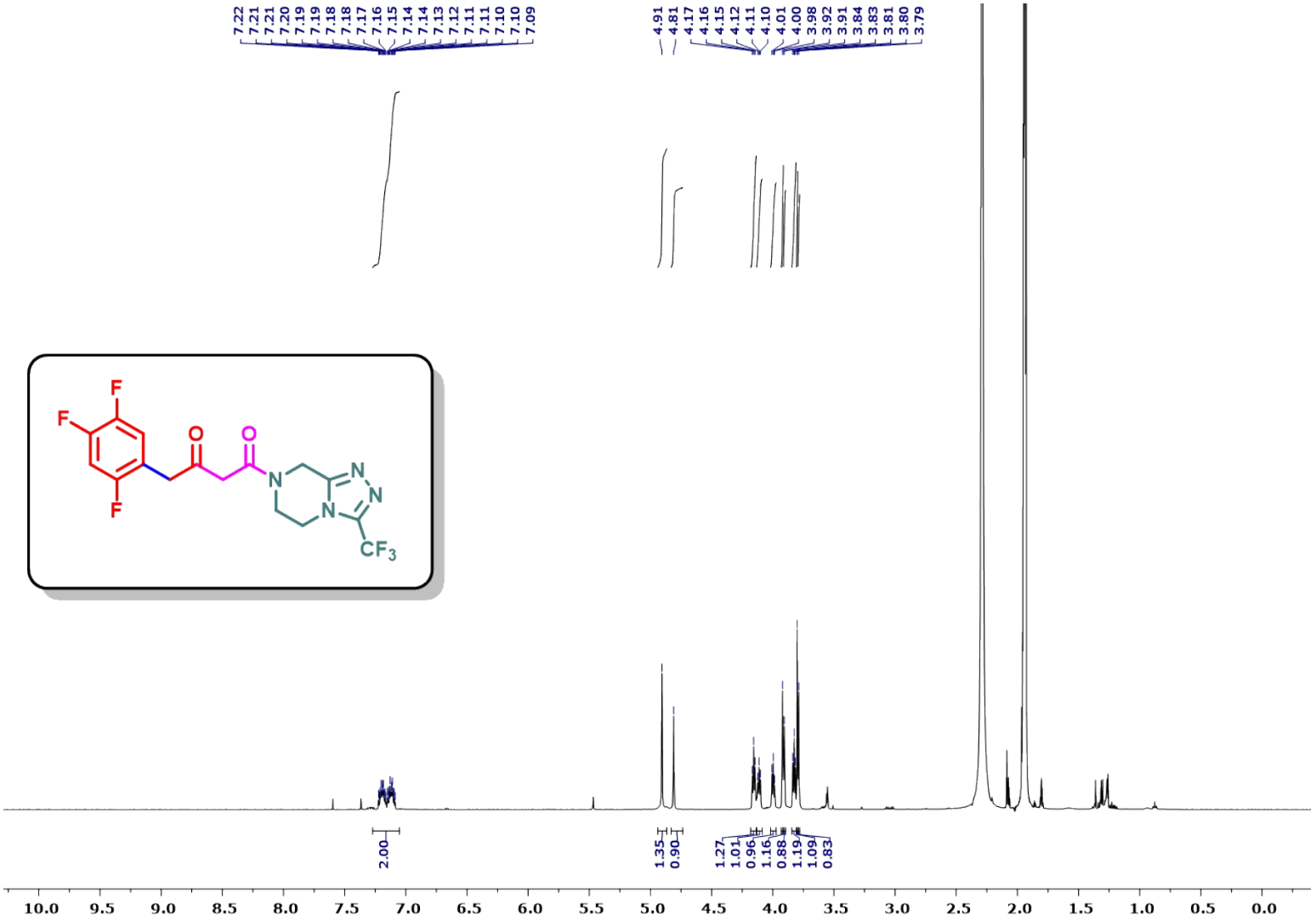

Fig. S101. ^{1}H NMR (500 MHz, CD_3CN) spectra of 1-(3-(trifluoromethyl)-5,6-dihydro-[1,2,4]triazolo[4,3-a]pyrazin-7(8H)-yl)-4-(2,4,5-trifluorophenyl)butane-1,3-dione (**9**).

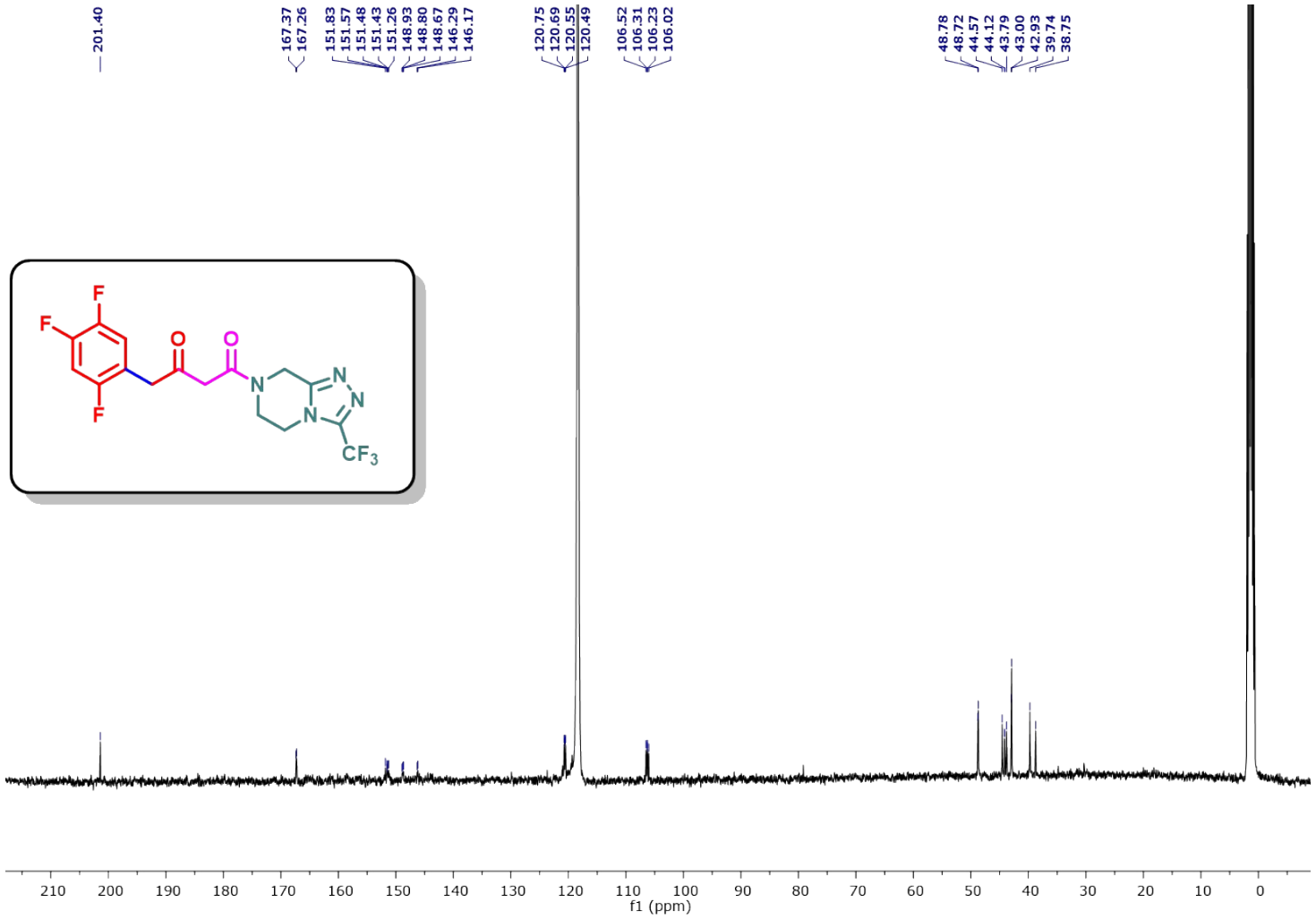

Fig. S102. ^{13}C NMR (101 MHz, CD_3CN) spectra of 1-(3-(trifluoromethyl)-5,6-dihydro-[1,2,4]triazolo[4,3-a]pyrazin-7(8H)-yl)-4-(2,4,5-trifluorophenyl)butane-1,3-dione (**9**).

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