SUPPORTING INFORMATION

Simultaneous Immobilization of CO_2 and H_2S by Propargyl Amines under Mild Conditions: Efficient Synthesis of Thiazolidine-2-ones

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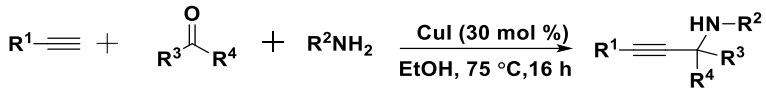
1. General Information

All reagents were purchased from Aladdin, Adamas, Macklin, or Bidepharm and directly used without further purification. Column chromatography separations were carried out on silica gel (200–300 mesh). Melting points were determined on a XT4A melting point apparatus and were uncorrected. Molecular weights were determined by high-resolution mass spectra (ESI) of Agilent Technologies LCMS TOF mass spectrometry or low-resolution mass spectrometry (ESI, LCMS-2020, Shimadzu). The FTIR was determined on a METTLER TOLEDO ReactIR^{TM}15 spectrometer. The NMR spectra were obtained in $CDCl_3$ or DMSO-d_6 on an Agilent 500 MHz DD2 spectrometer and referenced to the residual deuterated solvent or TMS. The NMR results were processed using MestReNova software.

2. Preparations of Starting Substrates and Thiazolidine-2-one

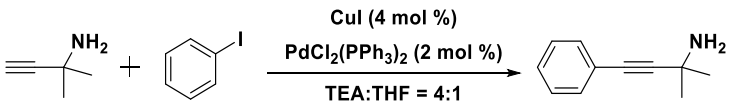
The **1a** and **1aa** were purchased from Macklin and Keychems and used without further purification.

2.1 General Procedure for the Synthesis of 1b-1i, 1k-1q, 1s-1z[1]



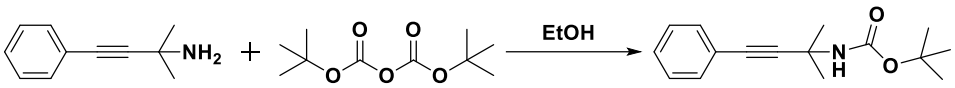
In a typical experiment, CuI (3 mmol) and EtOH (5 mL) were charged in a schlenk flask with a magnetic stirrer. The schlenk flask was sealed and flushed with N_2 and then was charged with alkyne (10 mmol), aldehyde (10 mmol), and amine (10 mmol). The schlenk flask was then placed in a sand bath of 75 °C and was allowed to stir overnight. After the reaction was completed, the crude reaction mixture was purified by silica gel column chromatography to provide the desired propargylic amine.

2.2 Procedure for the Preparation of 1j[2]



To a solution of CuI (38.1 mg, 0.2 mmol, 4.0 mol %), $PdCl_2(PPh_3)_2$ (70.2 mg, 0.1 mmol, 2.0 mol %), and the aryl iodide (5.5 mmol, 1.1 equiv) in THF/Et_3N = 4:1 solution (2.5 mL) under nitrogen, the 2-methylbut-3-yn-2-amine (5 mmol, 1.0 equiv) was added and the mixture was stirred overnight at room temperature. After the reaction was completed, a saturated NH_4Cl aqueous solution was added into the reaction mixture solution, then extracted with Et_2O three times. The combined organic layer was washed with brine and dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography to provide the target product 2-methyl-4-phenylbut-3-yn-2-amine (**1j**).

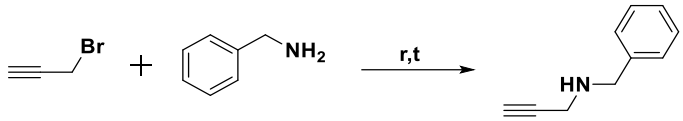
2.3 Procedure for the Preparation of 1r[3]



A mixture of 2-methyl-4-phenylbut-3-yn-2-amine **1j** (0.4773 g, 3 mmol) and $(Boc)_2O$

(0.6543 g, 3 mmol) in ethanol (2 mL) was vigorously stirred at room temperature for 2h. After the completion of the reactions, the reaction mixture was concentrated under vacuum. The crude reaction mixture was purified by silica gel column chromatography to provide the desired tert-butyl (2-methyl-4-phenylbut-3-yn-2-yl)carbamate (**1r**).

2.4 Procedure for the Preparation of 1ab[4]



To a 25 mL round bottom flask equipped with a stir bar was added propargylic bromide (0.9597 g, 8 mmol) was slowly dripped into the pre-added phenylmethanamine (5.1432 g, 48 mmol). Subsequently, the obtained mixture was stirred overnight at room temperature. After the reaction was finished, the resulting mixture solution was extracted in Et_2O and washed with saturated NaCl aqueous solution and the organic phase was dried over $MgSO_4$. The reaction mixture solution was concentrated and purified by silica gel column chromatography to afford N-benzylprop-2-yn-1-amine (**1ab**).

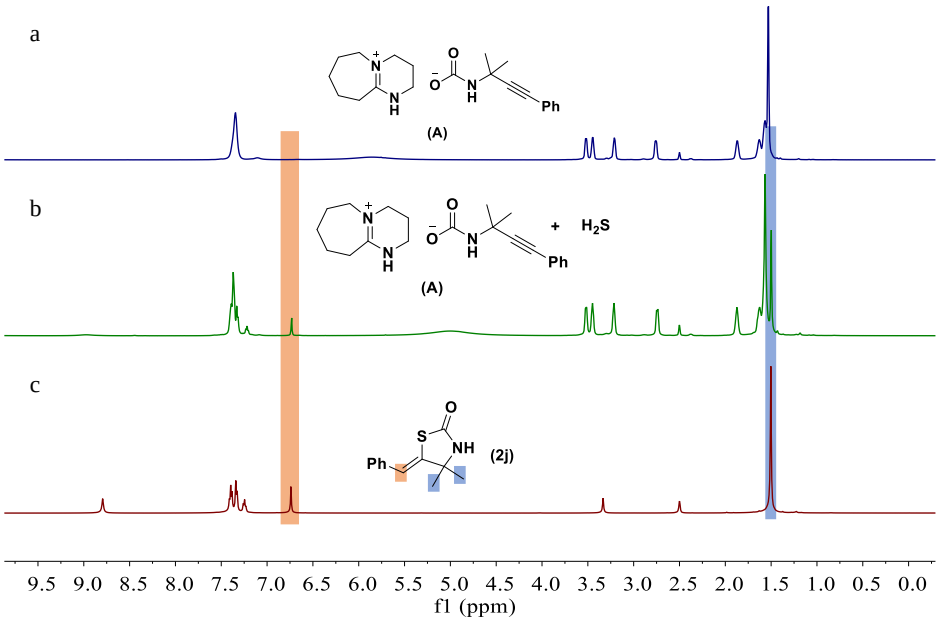
2.5 General Procedure for Preparing the Thiazolidine-2-one Derivatives by the Reaction of Propargylic Amines with H_2S and CO_2

As an example, the procedure using 2-methylbut-3-yn-2-amine(**1a**) as the substrate was described, and those for other substrates were similar to **1a**. **1a** (0.1662 g, 2.0 mmol), DBU (0.1827 g, 1.2 mmol), DMSO (2 mL) were loaded into a 10 mL stainless-steel batch reactor equipped with a magnetic stirrer. The air in the reactor was removed by blowing N_2 into the reactor. Subsequently, 1.0 MPa H_2S was charged and stirred. After the pressure of the reactor did not change, 1.0 MPa CO_2 was charged. The reactor was placed in a constant temperature sand bath and the reaction mixture solution was stirred for 24 h. After the reaction was completed, a saturated NaCl aqueous solution was added to the reaction mixture solution, then extracted with EtOAc three times. The combined organic layer was washed with brine and dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure. The crude mixture was purified by silica gel column chromatography (PE/EA=2/1) to give the desired product 4,4-dimethyl-5-methylenethiazolidin-2-one (**2a**).

3. NMR, FTIR and NOE experiments

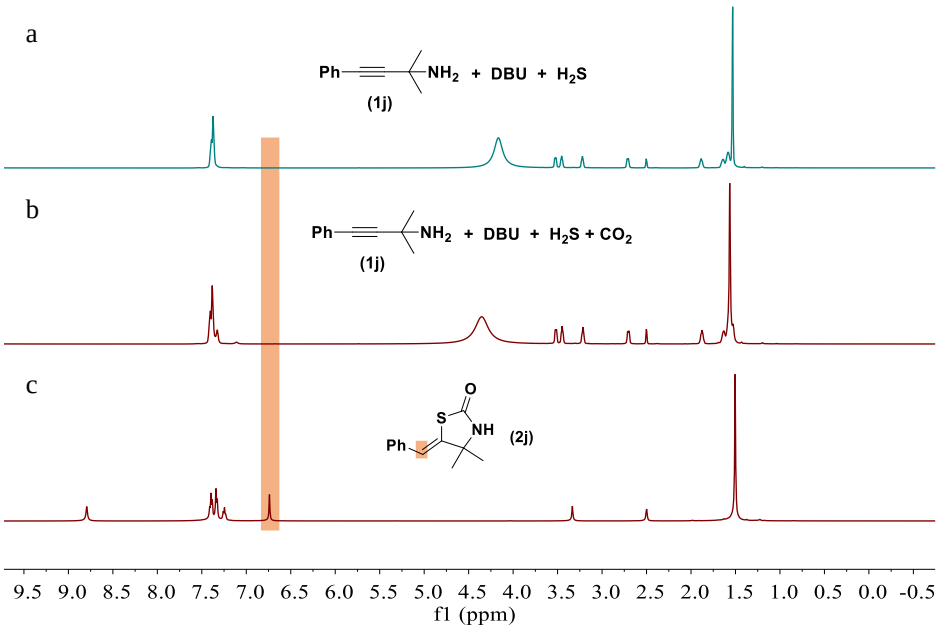
3.1 NMR experiments

The ^{1}HNMR spectra of **1j** and DBU were acquired when 2-methyl-4-phenylbut-3-yn-2-amine (**1j**) (10.0 mg) or DBU (10.0 mg) were dissolved in the NMR tube containing DMSO-d_6 (0.3 mL) respectively, which were shown in Figure 1a and Figure 1b. The ^{1}HNMR spectra of the mixture were acquired after **1j** (6.0 mg) and DBU (5.7 mg) was added into the NMR tube, which was shown in Figure 1c. The ^{1}HNMR and ^{13}CNMR spectra for the mixture solution of **1j**, DBU and CO_2 were acquired after CO_2 (1.2 MPa) was charged into the mixture solution of **1j** (11.6 mg) and DBU (11.0 mg), which were shown in Figure S1a and Figure 2. Afterward, H_2S (0.15 MPa) was continuously introduced into the previous mixture solution of DBU, **1j** and CO_2, then ^{1}HNMR spectra was acquired, which was shown in Figure S1b.



Figures S1. ^{1}HNMR spectra of intermediate **A** (a), **A** and H_2S mixture (b), **2j** (c) in $(CD_3)_2SO$

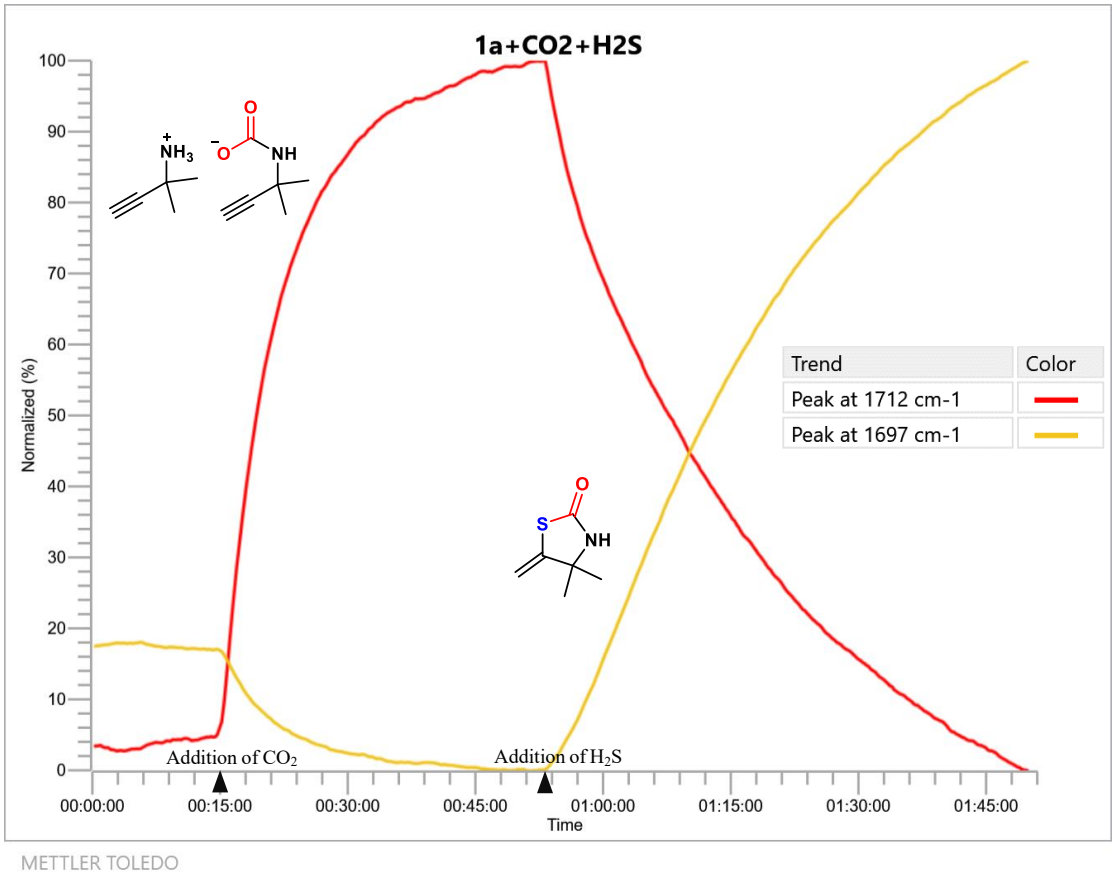
The ^{1}HNMR spectra for the mixture solution of 2-methyl-4-phenylbut-3-yn-2-amine (**1j**) (6.0 mg), DBU (5.7 mg), and H_2S were acquired after H_2S (0.4 MPa) was charged into the mixture solution of **1j** and DBU, which were shown in Figure S2a. Subsequently, the ^{1}HNMR spectra were acquired after CO_2 (1.0 MPa) was charged into the previous mixture solution of DBU, **1j** and H_2S, which were shown in Figure S2b. The ^{1}HNMR experimental results indicated that H_2S could not react with the C≡C bond of **1j** and inactivated the amino group of **1j**.



Figures S2. ^{1}HNMR spectra of **1j**-DBU-H_2S(a), **1j**-DBU-H_2S-CO_2(b), 2**j**(c) in $(CD_3)_2SO$.

3.2 FTIR experiments

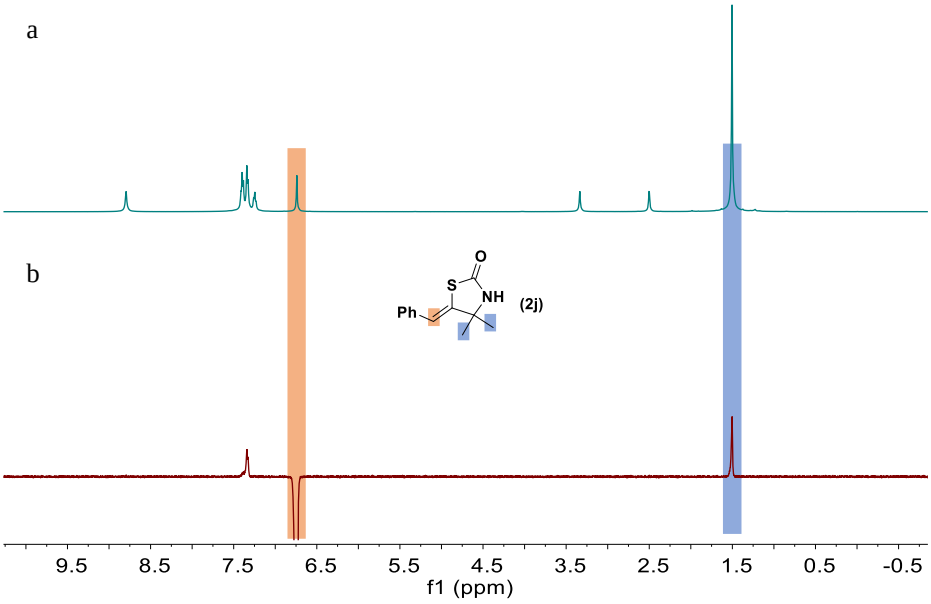
A METTLER TOLEDO ReactIR^{TM} reactor was cleaned and dried for 10 minutes. DMSO (30 mL) was introduced, followed by the 2-methylbut-3-yn-2-amine (**1a**) (0.5 mL). The solution was stirred at 25 ºC and 500 rpm for 5 minutes. IR spectra of the mixture solution were continuously collected in the range of 650-3000 cm^{-1}. The spectra were collected every 15 seconds, each consisted of 44 scans, at 4 cm^{-1} resolution, which was shown in Figure 3 and Figure S3. After 15 minutes, 1.0 MPa CO_2 was introduced into the reactor via an inlet valve. After the mixture reaction solution was detected by in situ IR for 40 minutes, 1.0 MPa H_2S was introduced into the reactor again via an inlet valve. Subsequently, the mixture reaction solution was detected by in situ IR for another 1 h.



Figures S3. The carbonyl signal intensity changes of the carbamate (1697 cm^{-1}) and **2a** (1712 cm^{-1})

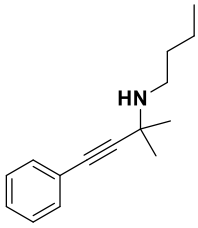
3.3 NOE experiments

As an example, the 10.0 mg (Z)-5-benzylidene-4,4-dimethylthiazolidin-2-one (**2j**) was dissolved in the NMR tube containing DMSO-d_6 (0.4 mL), the ^{1}HNMR was measured at first, and get the NMR spectrum, which was shown in Figure S4a. Subsequently, the NOE irradiation was performed on the H signal peak (6.74 ppm) of the olefin, and then the NOE spectrum was obtained, which was shown in Figure S4b. It can be seen from Figure S4b that the H resonance signal of the methyl group (1.50 ppm) was enhanced, which indicates that the distance was very close in space between the H of olefin and H of methyl. Therefore, the **2j** was the Z geometry configuration.

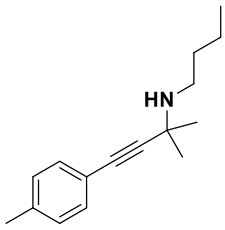


Figures S4. The ^{1}H NMR spectrum of **2j** (a) and the NOE spectrum of **2j** (irradiated 6.74 ppm) (b)

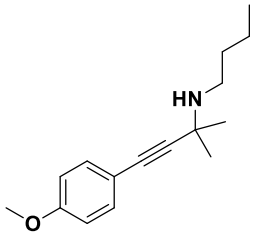
4. Characterization of Substrates and Products



<u>*N*-butyl-2-methyl-4-phenylbut-3-yn-2-amine (1b):</u>[5] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 10/1 – 2/1) to give the product as a yellow oil in 32% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 – 7.36 (m, 2H), 7.28 (dd, *J* = 5.1, 1.9 Hz, 3H), 2.79 (t, *J* = 7.3 Hz, 2H), 1.55 – 1.48 (m, 2H), 1.45 (s, 6H), 1.44 – 1.36 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 131.7, 128.3, 127.9, 123.6, 94.8, 82.1, 50.5, 44.2, 32.8, 29.8, 20.7, 14.1 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{15}H_{21}N$ 216.2; Found 216.3.

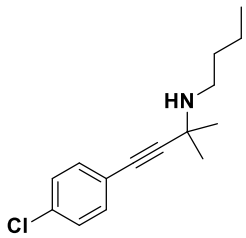


<u>*N*-butyl-2-methyl-4-(p-tolyl)but-3-yn-2-amine (1c):</u>[5] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 10/1 – 2/1) to give the product as a pale yellow oil in 25% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (d, *J* = 7.9 Hz, 2H), 7.09 (d, *J* = 8.1 Hz, 2H), 2.78 (t, *J* = 7.2 Hz, 2H), 2.33 (s, 3H), 1.54 – 1.47 (m, 2H), 1.44 (s, 6H), 1.42 – 1.36 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 137.9, 131.6, 129.1, 120.6, 94.0, 82.1, 50.4, 44.2, 32.8, 29.8, 21.5, 20.7, 14.2 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{16}H_{23}N$ 230.2; Found 230.1.

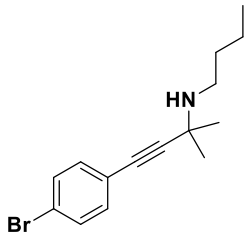


<u>*N*-butyl-4-(4-methoxyphenyl)-2-methylbut-3-yn-2-amine (1d):</u>[5] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 10/1 – 2/1) to give the product as a black oil in 25% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 (d, *J* = 8.4 Hz, 2H), 7.26 (d, *J* = 8.4 Hz, 2H), 2.76 (t, *J* = 7.2 Hz, 2H), 1.56 – 1.48 (m, 2H), 1.44 (s, 6H), 1.43 – 1.35

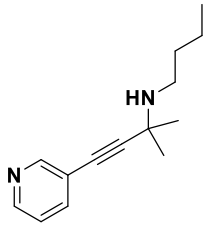
(m, 2H), 0.94 (t, J = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.3, 133.1, 115.7, 113.9, 93.1, 81.9, 55.4, 50.6, 44.2, 32.7, 29.8, 20.7, 14.1 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{16}H_{23}NO$ 246.2; Found 246.3.



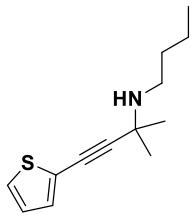
<u>*N*-butyl-4-(4-chlorophenyl)-2-methylbut-3-yn-2-amine (1e):</u>[5] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 10/1 – 2/1) to give the product as a black oil in 10 % yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 (d, J = 8.4 Hz, 2H), 7.26 (d, J = 8.4 Hz, 2H), 2.76 (t, J = 7.2 Hz, 2H), 1.53 – 1.48 (m, 2H), 1.44 (s, 6H), 1.42 – 1.36 (m, 2H), 0.94 (t, J = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 133.8, 130.0, 128.6, 122.1, 95.8, 81.0, 50.4, 44.2, 32.8, 29.7, 20.7, 14.1 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{15}H_{20}ClN$ 250.1; Found 250.0.



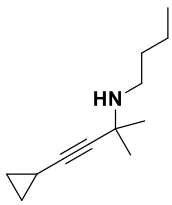
<u>4-(4-bromophenyl)-*N*-butyl-2-methylbut-3-yn-2-amine (1f):</u>[5] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 10/1 – 2/1) to give the product as a yellow oil in 30 % yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 (d, J = 8.5 Hz, 2H), 7.25 (d, J = 8.5 Hz, 2H), 2.76 (t, J = 7.2 Hz, 2H), 1.56 (s, 1H), 1.53 – 1.47 (m, 2H), 1.43 (s, 6H), 1.42 – 1.35 (m, 2H), 0.93 (t, J = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 133.2, 131.6, 122.6, 122.0, 96.0, 81.1, 50.5, 44.2, 32.7, 29.7, 20.7, 14.1 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{15}H_{20}BrN$ 294.1; Found 294.1.



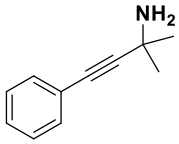
<u>*N*-butyl-2-methyl-4-(pyridin-3-yl)but-3-yn-2-amine (1g):</u> According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 10/1 – 1/2) to give the product as a yellow oil in 10 % yield.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.63 (s, 1H), 8.49 (d, J = 4.1 Hz, 1H), 7.67 (d, J = 7.9 Hz, 1H), 7.24 – 7.18 (m, 1H), 2.76 (t, J = 7.2 Hz, 2H), 1.56 (s, 1H), 1.54 – 1.48 (m, 2H), 1.45 (s, 6H), 1.43 – 1.36 (m, 2H), 0.94 (t, J = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 152.5, 148.3, 138.6, 123.1, 98.5, 78.8, 50.5, 44.2, 32.8, 29.7, 20.7, 14.1 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{14}H_{20}N_2$ 217.2; Found 217.2.



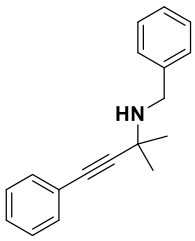
<u>*N*-butyl-2-methyl-4-(thiophen-2-yl)but-3-yn-2-amine (1h):</u> According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 10/1 – 4/1) to give the product as a yellow oil in 40 % yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.21 – 7.18 (m, 1H), 7.14 – 7.11 (m, 1H), 6.96 – 6.91 (m, 1H), 2.75 (t, J = 7.2 Hz, 2H), 1.52 – 1.48 (m, 2H), 1.43 (s, 6H), 1.42 – 1.35 (m, 2H), 0.93 (t, J = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 131.4, 127.0, 126.4, 123.7, 98.7, 75.2, 50.7, 44.2, 32.8, 29.6, 20.7, 14.1 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{13}H_{19}NS$ 222.1; Found 222.2.



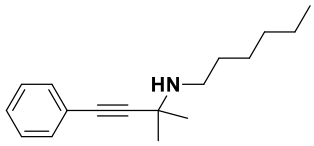
<u>*N*-butyl-4-cyclopropyl-2-methylbut-3-yn-2-amine (1i):</u>[5] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 4/1) to give the product as a yellow oil in 16 % yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.72 (t, *J* = 6.6 Hz, 2H), 1.57 – 1.48 (m, 2H), 1.37 (s, 6H), 1.29 – 1.22 (m, 2H), 0.95 – 0.91 (m, 3H), 0.81 – 0.76 (m, 2H), 0.76 – 0.71 (m, 3H), 0.64 – 0.59 (m, 2H) ppm; MS(ESI) m/z: $[M+Na]^+$ Calcd for $C_{11}H_{13}NNa$ 202.2; Found 202.3.



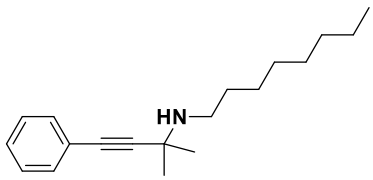
<u>2-methyl-4-phenylbut-3-yn-2-amine (1j):</u>[5] According to general procedure, the crude residue was purified by flash chromatography (CH_2Cl_2/CH_3OH = 30/1 – 5/1) to give the product as a yellow solid in 53 % yield. m.p. = 171–174 ºC; ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.55 (s, 2H), 7.47 – 7.37 (m, 5H), 1.62 (s, 6H) ppm; ^{13}C NMR (125 MHz, DMSO-d_6) δ 131.4, 129.2, 128.8, 121.2, 90.2, 83.3, 47.4, 27.8 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{11}H_{13}N$ 160.1; Found 160.2.



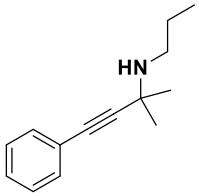
<u>*N*-benzyl-2-methyl-4-phenylbut-3-yn-2-amine (1k):</u>[5] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 10/1 – 2/1) to give the product as a black oil in 33% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 – 7.42 (m, 2H), 7.39 (d, *J* = 7.0 Hz, 2H), 7.35 – 7.28 (m, 5H), 7.24 (d, *J* = 7.6 Hz, 1H), 3.96 (s, 2H), 1.51 (s, 6H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 140.8, 131.8, 128.6(3), 128.6(1), 128.4, 128.0, 127.1, 123.6, 94.6, 82.6, 50.9, 49.3, 29.8 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{18}H_{19}N$ 250.2; Found 250.1.



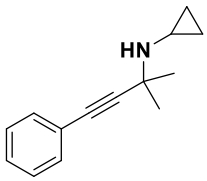
<u>*N*-(2-methyl-4-phenylbut-3-yn-2-yl)hexan-1-amine (1l):</u> According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 10/1 – 2/1) to give the product as a yellow oil in 22% yield.^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 – 7.38 (m, 2H), 7.31 – 7.26 (m, 3H), 2.77 (t, *J* = 7.3 Hz, 2H), 1.55 – 1.48 (m, 2H), 1.44 (s, 6H), 1.40 – 1.29 (m, 7H), 0.88 (t, *J* = 6.7 Hz, 3H) ppm; ^{13}CNMR (125 MHz, $CDCl_3$) δ 131.7, 128.3, 127.9, 123.7, 94.9, 82.1, 50.4, 44.6, 31.9, 30.7, 29.8, 27.3, 22.8, 14.2 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{17}H_{25}N$ 244.2; Found 244.3.



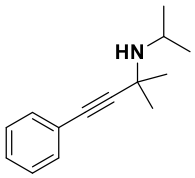
<u>*N*-(2-methyl-4-phenylbut-3-yn-2-yl)octan-1-amine (1m):</u> According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 10/1 – 2/1) to give the product as a yellow oil in 25% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 – 7.37 (m, 2H), 7.31 – 7.26 (m, 3H), 2.77 (t, *J* = 7.3 Hz, 2H), 1.55 – 1.48 (m, 2H), 1.44 (s, 6H), 1.39 – 1.25 (m, 10H), 0.87 (t, *J* = 6.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 131.7, 128.3, 127.9, 123.7, 94.8, 82.1, 50.5, 44.6, 32.0, 30.7, 29.8, 29.7, 29.4, 27.6, 22.8, 14.2 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{19}H_{29}N$ 272.2; Found 272.3.



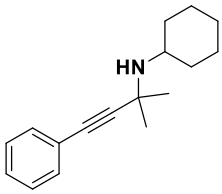
2-methyl-4-phenyl-*N*-propylbut-3-yn-2-amine (1n): According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 10/1 – 2/1) to give the product as a yellow oil in 11% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 – 7.37 (m, 2H), 7.31 – 7.26 (m, 3H), 2.75 (t, *J* = 7.4 Hz, 2H), 1.65 – 1.51 (m, 3H), 1.45 (s, 6H), 0.97 (t, *J* = 7.4 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 131.7, 128.3, 127.9, 123.6, 94.7, 82.1, 50.5, 46.5, 29.8, 23.8, 12.1 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{14}H_{19}N$ 202.2; Found 202.0.



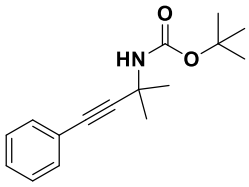
***N*-(2-methyl-4-phenylbut-3-yn-2-yl)cyclopropanamine (1o):**[5] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 10/1 – 4/1) to give the product as a yellow oil in 11% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 – 7.40 (m, 2H), 7.32 – 7.26 (m, 3H), 2.47 – 2.42 (m, 1H), 1.96 (s, 1H), 1.47 (s, 6H), 0.55 – 0.51 (m, 2H), 0.43 – 0.39 (m, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 131.7, 128.3, 127.9, 123.7, 95.4, 81.9, 51.4, 30.0, 26.4, 6.7 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{14}H_{17}N$ 200.1; Found 200.2.



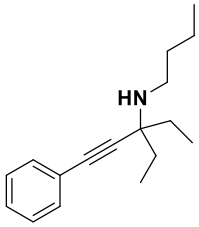
***N*-isopropyl-2-methyl-4-phenylbut-3-yn-2-amine (1p):** According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 10/1 – 4/1) to give the product as a yellow oil in 11% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 – 7.34 (m, 2H), 7.32 – 7.26 (m, 3H), 3.30 – 3.21 (m, 1H), 1.47 (s, 6H), 1.19 (d, *J* = 6.4 Hz, 6H) ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{14}H_{19}N$ 202.2; Found 202.3.



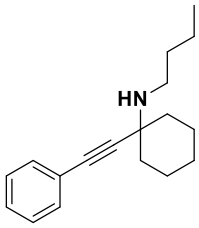
***N*-(2-methyl-4-phenylbut-3-yn-2-yl)cyclohexanamine (1q):**[5] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 10/1 – 4/1) to give the product as a yellow oil in 10% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 – 7.35 (m, 2H), 7.32 – 7.27 (m, 3H), 2.83 – 2.76 (m, 1H), 2.00 – 1.93 (m, 2H), 1.77 – 170 (m, 2H), 1.63 – 1.57 (m, 1H), 1.44 (s, 6H), 1.38 – 1.28 (m, 2H), 1.22 – 1.08 (m, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 131.6, 128.4, 127.8, 123.8, 95.7, 81.4, 53.6, 50.5, 36.4, 30.9, 25.9(2), 25.8(8) ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{17}H_{23}N$ 242.2; Found 242.2.



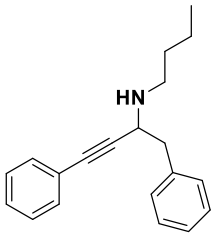
***tert*-butyl (2-methyl-4-phenylbut-3-yn-2-yl)carbamate (1r):** The crude residue was purified by flash chromatography (PE/EA = 4/1) to give the product as a white solid in 83% yield. m.p. = 90–93 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 – 7.38 (m, 2H), 7.30 – 7.26 (m, 3H), 4.79 (s, 1H), 1.66 (s, 6H), 1.47 (s, 9H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 146.9, 131.8, 128.3, 128.1, 123.3, 93.2, 85.3, 48.0, 28.6, 27.6 ppm; MS(ESI) m/z: $[M+Na]^+$ Calcd for $C_{16}H_{21}NO_2Na$ 282.1; Found 282.1.



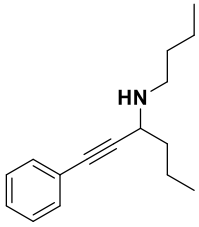
<u>*N*-butyl-3-ethyl-1-phenylpent-1-yn-3-amine (1s):</u> According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 10/1 – 4/1) to give the product as a yellow oil in 16% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 – 7.38 (m, 2H), 7.31 – 7.25 (m, 3H), 2.71 (t, *J* = 7.2 Hz, 2H), 1.75 – 1.62 (m, 4H), 1.54 – 1.47 (m, 2H), 1.44 – 1.36 (m, 2H), 1.00 (t, *J* = 7.4 Hz, 6H), 0.94 (t, *J* = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 131.8, 128.3, 127.8, 123.9, 93.6, 84.0, 57.9, 43.3, 32.8, 30.8, 20.8, 14.2, 8.5 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{17}H_{25}N$ 244.2; Found 244.0.



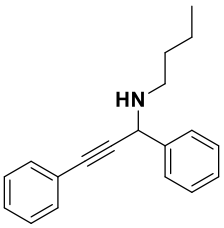
<u>*N*-butyl-1-(phenylethynyl)cyclohexan-1-amine (1t):</u>[5] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 10/1 – 2/1) to give the product as a pale yellow oil in 63% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 – 7.40 (m, 2H), 7.31 – 7.25 (m, 3H), 2.80 (t, *J* = 7.2 Hz, 2H), 1.94 (d, *J* = 12.5 Hz, 2H), 1.72 – 1.62 (m, 5H), 1.55 – 1.31 (m, 7H), 1.24 (s, 1H), 0.94 (t, *J* = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 131.7, 128.3, 127.8, 123.9, 93.8, 84.6, 55.2, 43.1, 38.4, 32.9, 26.1, 23.2, 20.7, 14.2 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{18}H_{25}N$ 256.2; Found 256.0.



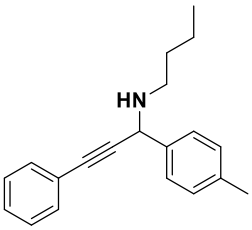
<u>*N*-butyl-1,4-diphenylbut-3-yn-2-amine (1u):</u> According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 10/1 – 4/1) to give the product as a yellow oil in 13% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 – 7.16 (m, 10H), 3.84 (t, *J* = 6.6 Hz, 1H), 3.10 – 2.97 (m, 2H), 2.96 – 2.89 (m, 1H), 2.70 – 2.62 (m, 1H), 1.55 – 1.41 (m, 3H), 1.39 – 1.29 (m, 2H), 0.91 (t, *J* = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 137.8, 131.6, 129.7, 128.2(4), 128.1(9), 127.9, 126.6, 123.3, 90.4, 84.6, 52.1, 47.3, 42.2, 32.1, 20.5, 14.0 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{20}H_{23}N$ 278.2; Found 278.1.



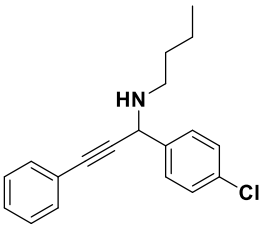
<u>*N*-butyl-1-phenylhex-1-yn-3-amine (1v):</u> According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 10/1 – 4/1) to give the product as a pale yellow oil in 44% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 – 7.39 (m, 2H), 7.32 – 7.25 (m, 3H), 3.60 (t, *J* = 6.2 Hz, 1H), 2.93 (q, *J* = 9.2, 8.5 Hz, 1H), 2.67 (q, *J* = 9.9, 8.8 Hz, 1H), 1.77 – 1.63 (m, 2H), 1.62 – 1.45 (m, 4H), 1.45 – 1.31 (m, 3H), 0.95 (dt, *J* = 17.7, 7.3 Hz, 6H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 131.7, 128.3, 127.9, 123.6, 91.4, 83.7, 50.8, 47.5, 38.4, 32.3, 20.6, 19.5, 14.1, 14.0 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{16}H_{23}N$ 230.2; Found 230.0.



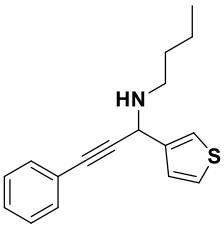
<u>*N*-(1,3-diphenylprop-2-yn-1-yl)butan-1-amine (1w):</u>[5] According to general procedure, the crude residue was purified by flash chromatography (hexanes/CH_2Cl_2 = 10/1 – 2/1) to give the product as a yellow oil in 52% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 (d, *J* = 7.8 Hz, 2H), 7.49 – 7.44 (m, 2H), 7.37 (t, *J* = 7.5 Hz, 2H), 7.33 – 7.27 (m, 4H), 4.80 (s, 1H), 2.90 – 2.81 (m, 1H), 2.76 – 2.68 (m, 1H), 1.57 – 1.48 (m, 2H), 1.46 (s, 1H), 1.42 – 1.34 (m, 2H), 0.92 (t, *J* = 7.6 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 140.8, 131.8, 128.6, 128.4, 128.2, 127.8, 127.7, 123.4, 89.8, 85.4, 54.9, 47.2, 32.3, 20.6, 14.1 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{19}H_{21}N$ 264.2; Found:264.1.



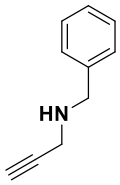
<u>*N*-(3-phenyl-1-(p-tolyl)prop-2-yn-1-yl)butan-1-amine (1x):</u> According to general procedure, the crude residue was purified by flash chromatography (hexanes/CH_2Cl_2 = 10/1 – 2/1) to give the product as a yellow oil in 71% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 (d, *J* = 6.6 Hz, 4H), 7.33 – 7.28 (m, 3H), 7.18 (d, *J* = 7.9 Hz, 2H), 4.77 (s, 1H), 2.88 – 2.68 (m, 2H), 2.36 (s, 3H), 1.57 (s, 1H), 1.56 – 1.48 (m, 2H), 1.43 – 1.34 (m, 2H), 0.93 (t, *J* = 7.3, 1.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 137.9, 137.5, 131.8, 129.3, 128.3, 128.2, 127.6, 123.4, 90.0, 85.2, 54.6, 47.2, 32.2, 21.3, 20.7, 14.1 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{20}H_{23}N$ 278.2; Found 278.3.



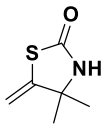
<u>*N*-(1-(4-chlorophenyl)-3-phenylprop-2-yn-1-yl)butan-1-amine (1y):</u> According to general procedure, the crude residue was purified by flash chromatography (hexanes/CH_2Cl_2 = 10/1 – 2/1) to give the product as a yellow oil in 27% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54 (d, *J* = 8.3 Hz, 2H), 7.48 –7.44 (m, 2H), 7.35 (s, 1H), 7.34 – 7.29 (m, 4H), 4.78 (s, 1H), 2.86 – 2.67 (m, 2H), 1.56 – 1.50 (m, 2H), 1.49 (s, 1H), 1.43 – 1.34 (m, 2H), 0.93 (t, *J* = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 139.3, 133.5, 131.8, 129.1, 128.7, 128.4(3), 128.3(8), 123.1, 89.2, 85.7, 54.2, 47.1, 32.3, 20.6, 14.1 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{19}H_{20}ClN$ 298.1; Found 298.2.



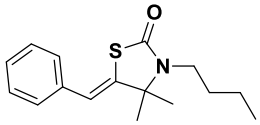
<u>*N*-(3-phenyl-1-(thiophen-3-yl)prop-2-yn-1-yl)butan-1-amine (1z):</u> According to general procedure, the crude residue was purified by flash chromatography (hexanes/CH_2Cl_2 = 10/1 – 1/2) to give the product as a yellow oil in 10% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 – 7.45 (m, 2H), 7.43 – 7.40 (m, 1H), 7.35 – 7.29 (m, 4H), 7.27 – 7.24 (m, 1H), 4.88 (s, 1H), 2.91 – 2.71 (m, 2H), 1.58 – 1.48 (m, 3H), 1.44 – 1.36 (m, 2H), 0.93 (t, *J* = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 142.0, 131.8, 128.4, 128.2, 127.2, 126.0, 123.2, 122.4, 89.6, 84.5, 50.5, 46.9, 32.2, 20.6, 14.1 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{17}H_{19}NS$ 270.1; Found 270.2.



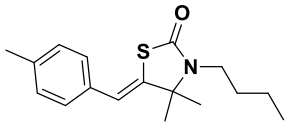
<u>*N*-benzylprop-2-yn-1-amine (1ab):</u>[6] The crude residue was purified by flash chromatography (PE/EA = 10/1 – 4/1) to give the product as a yellow oil in 51% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 – 7.29 (m, 4H), 7.29 – 7.21 (m, 1H), 3.89 (s, 2H), 3.43 (d, *J* = 2.4 Hz, 2H), 2.26 (t, *J* = 2.4 Hz, 1H), 1.59 (s, 1H) ppm; ^{13}C NMR (126 MHz, $CDCl_3$) δ 139.5, 128.6, 128.5, 127.3, 82.2, 71.7, 52.4, 37.5 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{10}H_{11}N$ 145.1; Found 146.3.



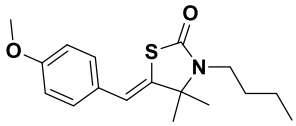
<u>4,4-dimethyl-5-methylenethiazolidin-2-one (2a):</u>[6] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 2/1) to give the product as a yellow solid (232 mg, 81%). m.p. = 93–96 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.32 (s, 1H), 5.19 (d, *J* = 2.2 Hz, 1H), 5.11 – 5.06 (m, 1H), 1.50 (s, 6H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.7, 149.3, 104.9, 62.7, 29.9 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for C_6H_9NOS 144.0478; Found 144.0474.



<u>(*Z*)-5-benzylidene-3-butyl-4,4-dimethylthiazolidin-2-one (2b):</u>[5] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 2/1) to give the product as a yellow oil (478 mg, 83 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 – 7.31 (m, 4H), 7.25 – 7.21 (m, 1H), 6.53 (s, 1H), 3.30 – 3.24 (m, 2H), 1.69 – 1.60 (m, 2H), 1.56 (s, 6H), 1.41 – 1.32 (m, 2H), 0.95 (t, *J* = 7.4 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.9, 139.6, 136.2, 128.7, 128.2, 127.3, 119.0, 68.1, 42.5, 31.7, 28.4, 20.6, 13.9 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{16}H_{21}NOS$ 276.1417; Found 276.1416.

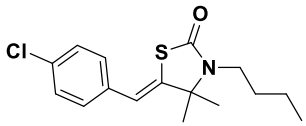


<u>(*Z*)-3-butyl-4,4-dimethyl-5-(4-methylbenzylidene)thiazolidin-2-one (2c):</u>[5] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 2/1) to give the product as a brown solid (534 mg, 92 %). m.p. = 60–63 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.23 (d, *J* = 8.2 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 6.50 (s, 1H), 3.29 – 3.23 (m, 2H), 2.34 (s, 3H), 1.68 – 1.59 (m, 2H), 1.55 (s, 6H), 1.41 – 1.31 (m, 2H), 0.95 (t, *J* = 7.4 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.1, 138.3, 137.1, 133.3, 129.4, 128.1, 119.0, 68.0, 42.5, 31.7, 28.3, 21.3, 20.5, 13.9 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{17}H_{23}NOS$ 290.1573; Found 290.1568.

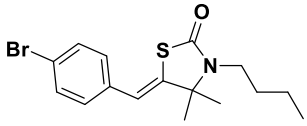


<u>(*Z*)-3-butyl-5-(4-methoxybenzylidene)-4,4-dimethylthiazolidin-2-one (2d):</u>[5] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 2/1) to give the product as a brown solid (494 mg, 81 %). m.p. = 61–64 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27 (d, *J* = 10.5 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 6.47 (s, 1H), 3.82 (s, 3H), 3.29 – 3.22 (m, 2H), 1.68 – 1.60 (m, 2H), 1.54 (s, 6H), 1.40 – 1.31 (m, 2H), 0.95 (t, *J* = 7.4 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.2, 158.7, 136.9, 129.5, 128.9, 118.6, 114.2, 68.0, 55.4, 42.5, 31.7, 28.3, 20.5, 13.9 pp

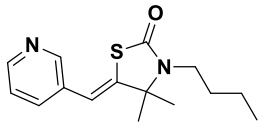
m; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{17}H_{23}NO_2S$ 306.1522; Found 306.1516.



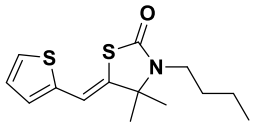
(*Z*)-3-butyl-5-(4-chlorobenzylidene)-4,4-dimethylthiazolidin-2-one (2e):[5] According to general procedure, the crude r-esidue was purified by flash chromatography (PE/EA = 2/1) to give the product as a brown solid (513 mg, 83 %). m.p. = 63–66 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 (d, *J* = 8.5 Hz, 2H), 7.26 (d, *J* = 8.7 Hz, 2H), 6.47 (s, 1H), 3.30 – 3.23 (m, 2H), 1.68 – 1.60 (m, 2H), 1.55 (s, 6H), 1.42 – 1.31 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.5, 140.5, 134.7, 132.8, 129.4, 128.9, 117.8, 68.1, 42.6, 31.7, 28.3, 20.5, 13.9 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{16}H_{20}ClNOS$ 310.1027; Found 310.1021.



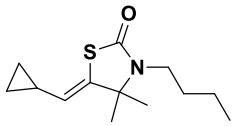
(*Z*)-5-(4-bromobenzylidene)-3-butyl-4,4-dimethylthiazolidin-2-one (2f): According to general procedure, the crude r-esidue was purified by flash chromatography (PE/EA = 2/1) to give the product as a yellow oil (526 mg, 75 %).^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 (d, *J* = 8.5 Hz, 2H), 7.20 (d, *J* = 8.6 Hz, 2H), 6.45 (s, 1H), 3.29 – 3.24 (m, 2H), 1.68 – 1.60 (m, 2H), 1.55 (s, 6H), 1.40 – 1.32 (m, 2H), 0.95 (td, *J* = 7.4, 1.1 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.5, 140.7, 135.1, 131.9, 129.7, 121.0, 117.9, 68.1, 42.6, 31.7, 28.3, 20.6, 13.9 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{16}H_{20}BrNOS$ 354.1; Found 354.1.



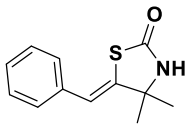
(*Z*)-3-butyl-4,4-dimethyl-5-(pyridin-3-ylmethylene)thiazolidin-2-one (2g): According to general procedure, the crude res-idue was purified by flash chromatography (PE/EA = 1/3) to give the product as a yellow oil (437 mg, 79 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.52 (d, *J* = 55.4 Hz, 2H), 7.70 (d, *J* = 7.5 Hz, 1H), 7.32 (s, 1H), 6.49 (s, 1H), 3.31 – 3.24 (m, 2H), 1.66 – 1.63 (m, 2H), 1.58 (s, 6H), 1.41 – 1.32 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.1, 150.1, 147.9, 142.9, 134.2, 115.4, 68.3, 42.7, 31.7, 28.4, 20.5, 13.9 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{15}H_{22}N_2OS$ 277.1369; Found 277.1376.



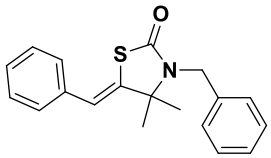
(*Z*)-3-butyl-4,4-dimethyl-5-(thiophen-2-ylmethylene)thiazolidin-2-one (2h): According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 4/1) to give the product as a yellow solid (459 mg, 82 %). m.p. = 80–83 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 (d, *J* = 5.0 Hz, 1H), 7.07 – 7.04 (m, 1H), 7.02 (d, *J* = 3.1 Hz, 1H), 6.74 (s, 1H), 3.29 – 3.23 (m, 2H), 1.66 – 1.61 (m, 2H), 1.54 (s, 6H), 1.40 – 1.31 (m, 2H), 0.95 (t, *J* = 7.4 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.4, 140.2, 137.9, 127.5, 126.6, 126.0, 112.0, 67.7, 42.7, 31.7, 28.3, 20.5, 13.9 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{14}H_{19}NOS_2$ 282.0981; Found 282.0981.



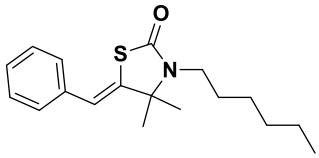
(*Z*)-3-butyl-5-(cyclopropylmethylene)-4,4-dimethylthiazolidin-2-one (2i):[5] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 4/1) to give the product as a yellow oil (191 mg, 40 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.99 (d, *J* = 8.8 Hz, 1H), 3.22 – 3.14 (m, 2H), 1.63 – 1.55 (m, 2H), 1.39 (s, 6H), 1.37 – 1.28 (m, 3H), 1.23 – 1.16 (m, 1H), 0.92 (t, *J* = 7.4 Hz, 3H), 0.85 – 0.78 (m, 2H), 0.44 – 0.39 (m, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.5, 136.9, 122.9, 66.4, 42.3, 31.8, 28.0, 20.5, 13.9, 13.1, 7.3 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{13}H_{21}NOS$ 240.1; Found 240.2.



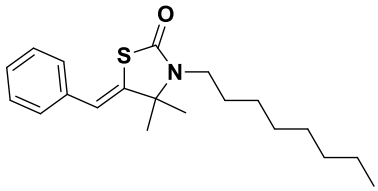
(*Z*)-5-benzylidene-4,4-dimethylthiazolidin-2-one (2j):[5] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 1/2) to give the product as a yellow solid (381 mg, 87 %). m.p. = 118–120 °C; ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.79 (s, 1H), 7.43 – 7.33 (m, 4H), 7.25 (t, *J* = 7.3 Hz, 1H), 6.74 (s, 1H), 1.50 (s, 6H) ppm; ^{13}C NMR (125 MHz, DMSO-d_6) δ 166.1, 140.8, 136.0, 128.6, 127.7, 126.9, 118.7, 63.5, 29.7 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{12}H_{13}NOS$ 220.0791; Found 220.0788.



(*Z*)-3-benzyl-5-benzylidene-4,4-dimethylthiazolidin-2-one (2k):[5] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 2/1) to give the product as a yellow oil (552 mg, 89 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40-7.33 (m, 4H), 7.32-7.29 (m, 4H), 7.27 – 7.23 (m, 2H), 6.53 (s, 1H), 4.62 (s, 2H), 1.49 (s, 6H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.1, 139.1, 138.1, 136.0, 128.8, 128.7, 128.2, 127.5, 127.4, 127.3, 119.3, 68.4, 45.3, 28.5 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{19}H_{19}NOS$ 310.1260; Found 310.1250.

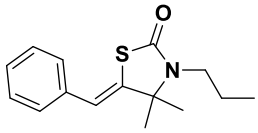


(*Z*)-5-benzylidene-3-hexyl-4,4-dimethylthiazolidin-2-one (2l): According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 4/1) to give the product as a yellow oil (516 mg, 85 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 – 7.32 (m, 4H), 7.26 – 7.21 (m, 1H), 6.53 (s, 1H), 3.29 – 3.22 (m, 2H), 1.69 – 1.62 (m, 2H), 1.56 (s, 6H), 1.38 – 1.28 (m, 6H), 0.89 (t, *J* = 6.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.9, 139.5, 136.2, 128.7, 128.2, 127.2, 119.0, 68.1, 42.7, 31.6, 29.6, 28.4, 27.0, 22.72, 14.1 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{18}H_{25}NOS$ 304.1730; Found 304.1734.

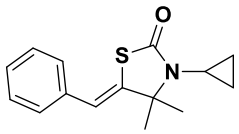


(*Z*)-5-benzylidene-4,4-dimethyl-3-octylthiazolidin-2-one (2m): According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 2/1) to give the product as a yellow oil (572 mg, 86 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 – 7.31 (m, 4H), 7.26 – 7.21 (m, 1H), 6.53 (s, 1H), 3.28 – 3.23 (m, 2H), 1.70 – 1.62 (m, 2H), 1.56 (s, 6H),

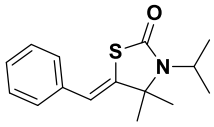
1.34 – 1.23 (m, 10H), 0.88 (t, J = 6.7 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.9, 139.5, 136.2, 128.7, 128.2, 127.3, 119.0, 68.1, 42.8, 31.9, 29.7, 29.3(9), 29.3(7), 28.4, 27.3, 22.8, 14.2 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{20}H_{29}NOS$ 332.2043; Found 332.2049.



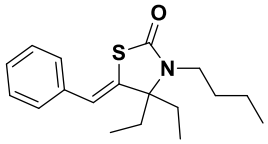
(*Z*)-5-benzylidene-4,4-dimethyl-3-propylthiazolidin-2-one (2n): According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 2/1) to give the product as a brown solid (492 mg, 94 %). m.p. = 102–105 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) 1 δ 7.41 – 7.29 (m, 4H), 7.25 – 7.21 (m, 1H), 6.53 (s, 1H), 3.27 – 3.16 (m, 2H), 1.74 – 1.63 (m, 2H), 1.56 (s, 6H), 0.94 (t, J = 7.4 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.9, 139.3, 136.0, 128.6, 128.0, 127.1, 118.9, 67.9, 44.2, 28.2, 22.7, 11.5 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{15}H_{19}NOS$ 262.1260; Found 262.1257.



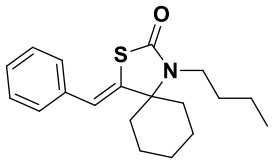
(*Z*)-5-benzylidene-3-cyclopropyl-4,4-dimethylthiazolidin-2-one (2o):[6] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 4/1) to give the product as a yellow solid (373 mg, 72 %). m.p. = 93–96 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 – 7.31 (m, 4H), 7.25 – 7.21 (m, 1H), 6.54 (s, 1H), 2.39 – 2.33 (m, 1H), 1.67 (s, 6H), 0.99 – 0.94 (m, 2H), 0.93 – 0.88 (m, 2H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.8, 138.9, 136.1, 128.7, 128.2, 127.3, 119.0, 69.6, 28.3, 24.3, 6.4 ppm; MS(ESI) m/z: $[M+H]^+$ Calcd for $C_{15}H_{17}NOS$ 260.1; Found 260.2.



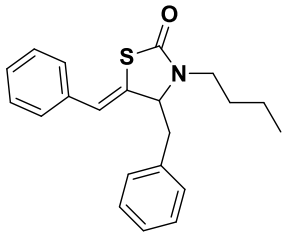
(*Z*)-5-benzylidene-3-isopropyl-4,4-dimethylthiazolidin-2-one (2p): According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 2/1) to give the product as a yellow solid (240 mg, 46 %). m.p. = 137–140 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 – 7.31 (m, 4H), 7.25 – 7.20 (m, 1H), 6.46 (s, 1H), 3.58 – 3.47 (m, 1H), 1.56 (s, 6H), 1.49 (d, J = 6.8 Hz, 6H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.6, 139.8, 136.3, 128.7, 128.2, 127.1, 118.8, 69.1, 47.7, 28.3, 20.5 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{15}H_{19}NOS$ 262.1260; Found 262.1256.



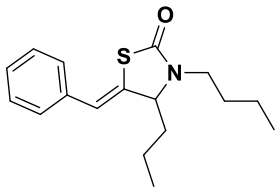
(*Z*)-5-benzylidene-3-butyl-4,4-diethylthiazolidin-2-one (2s): According to general procedure, the crude residue was purified by flash chromatography (CH_2Cl_2/MeOH = 200/1) to give the product as a yellow solid (485 mg, 80 %). m.p. = 55–58 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 – 7.31 (m, 4H), 7.25 – 7.21 (m, 1H), 6.39 (s, 1H), 3.21 – 3.14 (m, 2H), 1.95 – 1.83 (m, 2H), 1.78 – 1.69 (m, 2H), 1.69 – 1.63 (m, 2H), 1.41 – 1.32 (m, 2H), 0.96 (t, J = 7.4 Hz, 3H), 0.86 (t, J = 7.2 Hz, 6H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.8, 136.4, 136.3, 128.8, 128.1, 127.1, 118.2, 75.9, 42.6, 34.1, 31.0, 20.8, 13.9, 7.8 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{18}H_{25}NOS$ 304.1730; Found 304.1725.



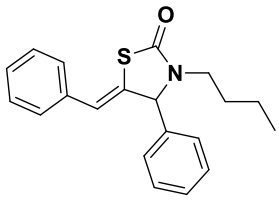
(*Z*)-4-benzylidene-1-butyl-3-thia-1-azaspiro[4.5]decan-2-one (2t):[5] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 2/1) to give the product as a yellow solid (578 mg, 92 %). m.p. = 90–93 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 – 7.30 (m, 4H), 7.28 – 7.22 (m, 1H), 6.96 (s, 1H), 3.29 – 3.21 (m, 2H), 2.07 (d, *J* = 10.4 Hz, 2H), 1.87 – 1.72 (m, 7H), 1.63 – 1.50 (m, 2H), 1.40 – 1.25 (m, 3H), 0.94 (t, *J* = 7.4 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.1, 139.6, 136.3, 128.6, 128.5, 127.5, 122.7, 69.7, 42.4, 33.7, 32.2, 24.7, 22.8, 20.5, 13.9 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{19}H_{25}NOS$ 316.1730; Found 316.1722.



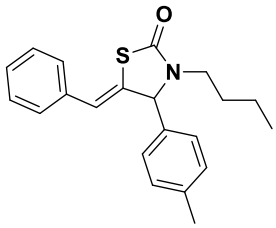
(*Z*)-4-benzyl-5-benzylidene-3-butylthiazolidin-2-one (2u): According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 2/1) to give the product as a red oil (668 mg, 99 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 (t, *J* = 7.6 Hz, 2H), 7.29 – 7.20 (m, 4H), 7.19 – 7.11 (m, 4H), 6.07 (s, 1H), 4.69 – 4.49 (m, 1H), 3.92 (dt, *J* = 14.5, 8.0 Hz, 1H), 3.13 (dd, *J* = 13.6, 4.1 Hz, 1H), 3.10 – 2.98 (m, 2H), 1.69 – 1.55 (m, 2H), 1.41 – 1.29 (m, 2H), 0.95 (t, *J* = 7.4 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.1, 135.4, 135.2, 131.0, 129.9, 128.6, 128.5, 127.9, 127.3, 127.1, 122.4, 66.6, 42.5, 40.5, 29.6, 20.0, 13.7 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{21}H_{23}NOS$ 338.1573; Found 338.1542.



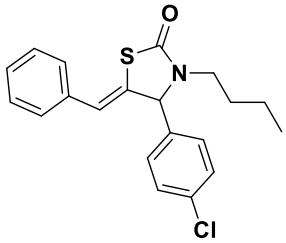
(*Z*)-5-benzylidene-3-butyl-4-propylthiazolidin-2-one (2v): According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 2/1) to give the product as a yellow oil (522 mg, 90 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 (t, *J* = 7.7 Hz, 2H), 7.31 (d, *J* = 7.3 Hz, 2H), 7.23 (d, *J* = 7.3 Hz, 1H), 6.49 (s, 1H), 4.56 (s, 1H), 3.85 – 3.74 (m, 1H), 3.05 – 2.93 (m, 1H), 1.97 – 1.87 (m, 1H), 1.79 – 1.68 (m, 1H), 1.66 – 1.49 (m, 2H), 1.47 – 1.30 (m, 4H), 0.95 (t, *J* = 7.4 Hz, 6H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.1, 135.8, 132.5, 128.8, 128.1, 127.3, 121.1, 65.2, 42.3, 36.4, 29.6, 20.1, 16.1, 14.1, 13.9 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{17}H_{23}NOS$ 290.1573; Found 290.1558.



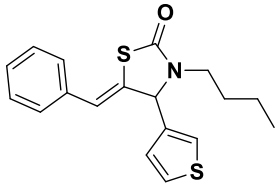
(*Z*)-5-benzylidene-3-butyl-4-phenylthiazolidin-2-one (2w):[5] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 3/1) to give the product as a yellow solid (589 mg, 91 %). m.p. = 83–86 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 – 7.37 (m, 5H), 7.35 – 7.30 (m, 2H), 7.25 – 7.19 (m, 3H), 6.31 (s, 1H), 5.48 (s, 1H), 3.76 – 3.65 (m, 1H), 2.77 – 2.65 (m, 1H), 1.53 – 1.42 (m, 2H), 1.33 – 1.23 (m, 2H), 0.88 (t, *J* = 7.4 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.0, 139.4, 135.7, 132.4, 129.4, 129.1, 128.7, 128.1, 127.4, 123.3, 69.9, 42.9, 29.2, 20.0, 13.8 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{20}H_{21}NOS$ 324.1417; Found 324.1409.



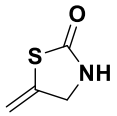
(*Z*)-5-benzylidene-3-hexyl-4-(p-tolyl)thiazolidin-2-one (2x): According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 4/1) to give the product as a yellow solid (634 mg, 94 %). m.p. = 83–86 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 (t, *J* = 7.7 Hz, 2H), 7.25 (d, *J* = 3.1 Hz, 2H), 7.24 – 7.17 (m, 5H), 6.28 (s, 1H), 5.43 (s, 1H), 3.73 – 3.65 (m, 1H), 2.74 – 2.66 (m, 1H), 2.37 (s, 3H), 1.51 – 1.43 (m, 2H), 1.33 – 1.21 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.0, 139.0, 136.4, 135.8, 132.7, 130.0, 128.7, 128.1, 127.3(3), 127.3(1), 123.1, 69.7, 42.8, 29.2, 21.4, 20.1, 13.8 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{21}H_{23}NOS$ 338.1573; Found 338.1578.



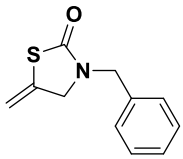
(*Z*)-5-benzylidene-3-butyl-4-(4-chlorophenyl)thiazolidin-2-one (2y): According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 4/1) to give the product as a yellow solid (471 mg, 66 %). m.p. = 71–74 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 (d, *J* = 8.3 Hz, 2H), 7.33 (d, *J* = 8.2 Hz, 4H), 7.23 (d, *J* = 7.6 Hz, 3H), 6.28 (s, 1H), 5.45 (s, 1H), 3.71 (dt, *J* = 15.4, 7.9 Hz, 1H), 2.73 – 2.66 (m, 1H), 1.51 – 1.42 (m, 2H), 1.34 – 1.21 (m, 2H), 0.89 (t, *J* = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.0, 138.0, 135.5, 135.1, 131.9, 129.7, 128.7, 128.7, 128.1, 127.6, 123.6, 69.1, 42.9, 29.2, 20.0, 13.8 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{20}H_{20}ClNOS$ 358.1027; Found 358.1031.



(*Z*)-5-benzylidene-3-butyl-4-(thiophen-3-yl)thiazolidin-2-one (2z): According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 4/1) to give the product as a yellow solid (544 mg, 83 %). m.p. = 109–112 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 – 7.31 (m, 4H), 7.22 (dd, *J* = 13.3, 5.9 Hz, 2H), 7.08 (d, *J* = 6.1 Hz, 1H), 6.34 (s, 1H), 5.61 (s, 1H), 3.71 – 3.63 (m, 1H), 2.84 – 2.76 (m, 1H), 1.53 – 1.40 (m, 2H), 1.34 – 1.22 (m, 3H), 0.89 (t, *J* = 7.3 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.6, 140.2, 135.7, 131.5, 128.7, 128.1(3), 128.1(2), 127.9, 127.4, 125.8, 124.0, 123.2, 65.4, 42.9, 29.3, 20.1, 13.8 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for $C_{18}H_{19}NOS_2$ 330.0981; Found 330.0981.



5-methylenethiazolidin-2-one (2aa):[6] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 1/2) to give the product as a yellow solid (55 mg, 24 %). m.p. = 111–114 ºC; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.53 (s, 1H), 5.23 (q, *J* = 2.2 Hz, 1H), 5.18 – 5.11 (m, 1H), 4.31 (s, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) δ 173.1, 138.8, 106.9, 49.4 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for C_4H_5NOS 116.0165; Found 116.0167.



3-benzyl-5-methylenethiazolidin-2-one (2ab):[6] According to general procedure, the crude residue was purified by flash chromatography (PE/EA = 5/1) to give the product as a yellow oil (144 mg, 35 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 – 7.34 (m, 2H), 7.33 – 7.29 (m, 1H), 7.27 (d, *J* = 7.9 Hz, 2H), 5.17 – 5.14 (m, 1H), 5.14 – 5.11 (m, 1H), 4.53 (s, 2H), 4.17 – 4.13 (m, 2H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.1, 135.5, 135.4, 129.0, 128.3, 128.2, 106.6, 53.9, 48.3 ppm; HRMS(ESI) m/z: $[M+H]^+$ Calcd for C_4H_5NOS: 206.1; Found 206.0.

5. References

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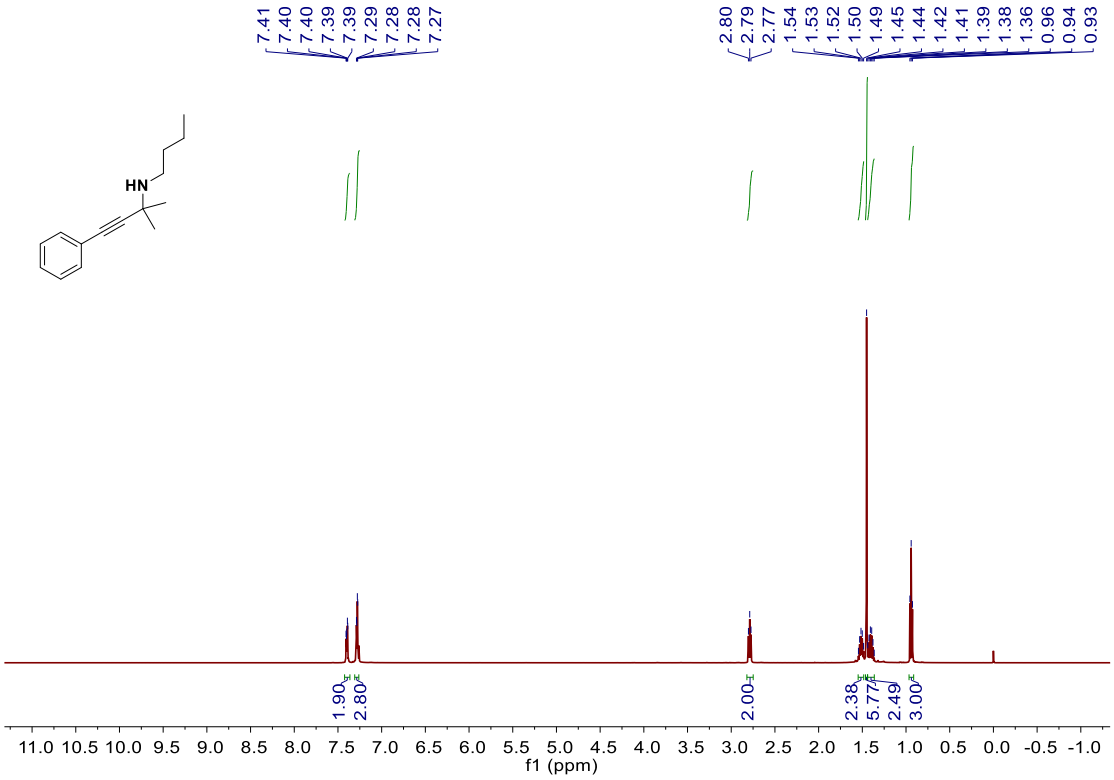
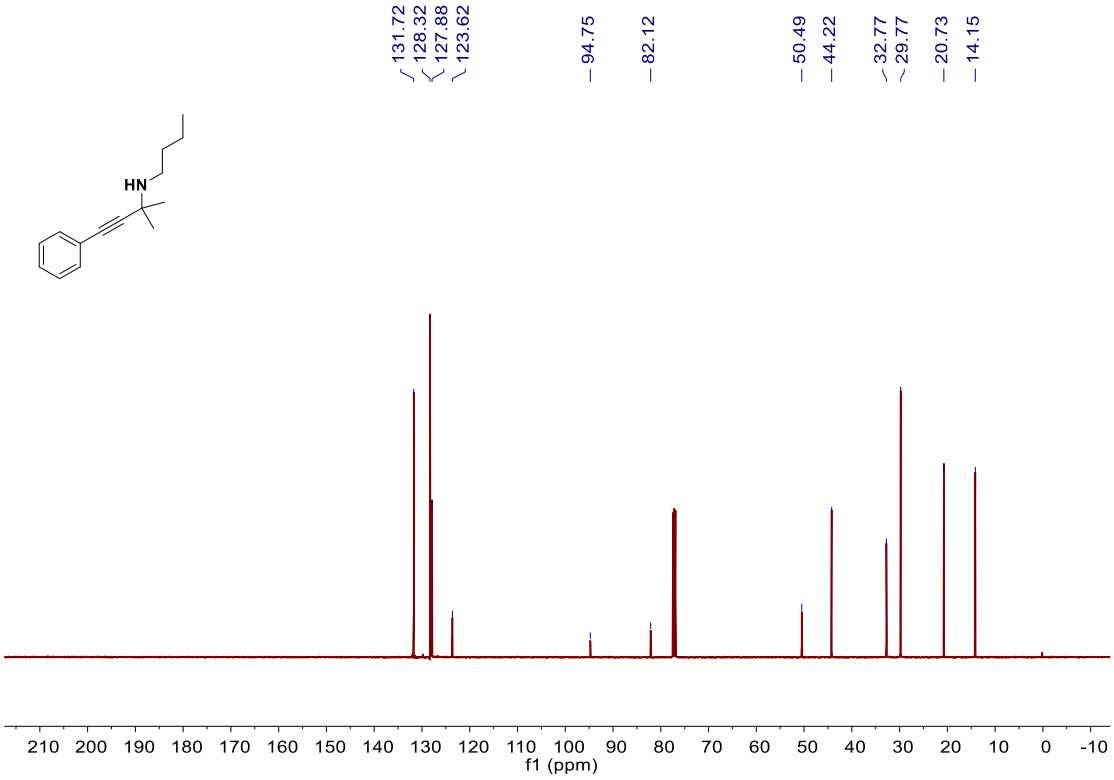
(6) Zhou, H.; Zhang, R.; Mu, S.; Zhang, H.; Lu, X.-B. Organocatalytic Cyclization of COS and Propargylic Derivatives to Value-Added Heterocyclic Compounds. *ChemCatChem* **2019**, *11*, 5728−5732.

6. NMR Spectra

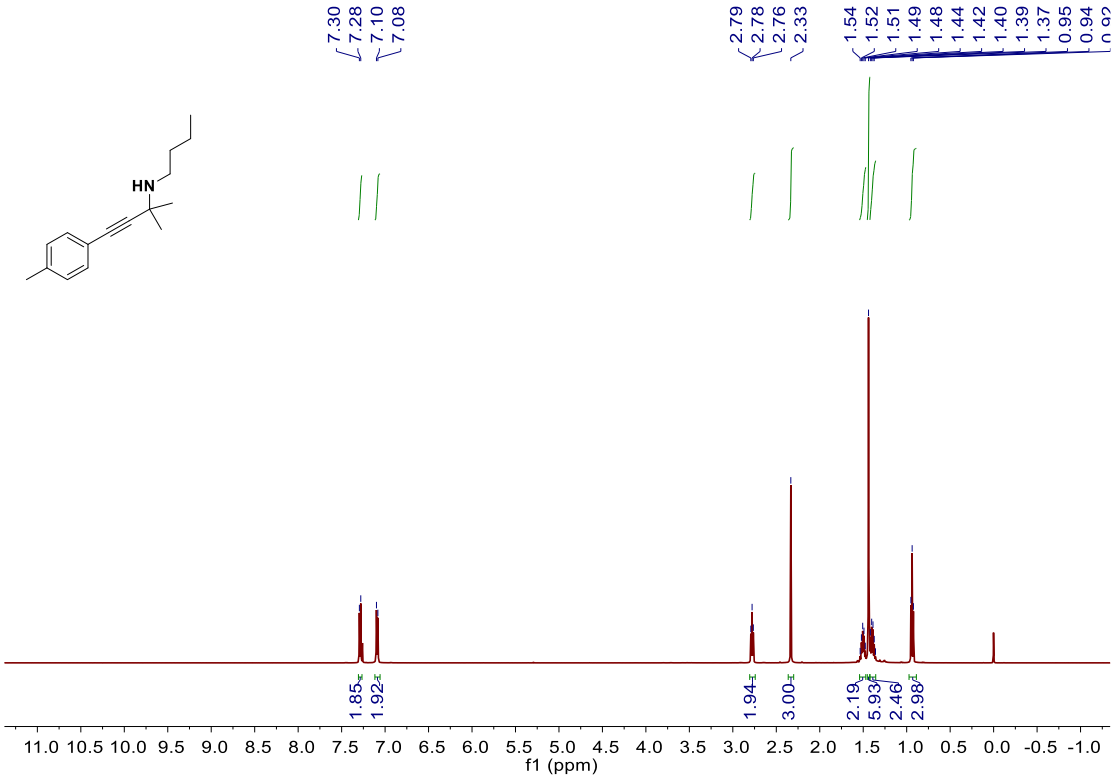
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-butyl-2-methyl-4-phenylbut-3-yn-2-amine (1b)



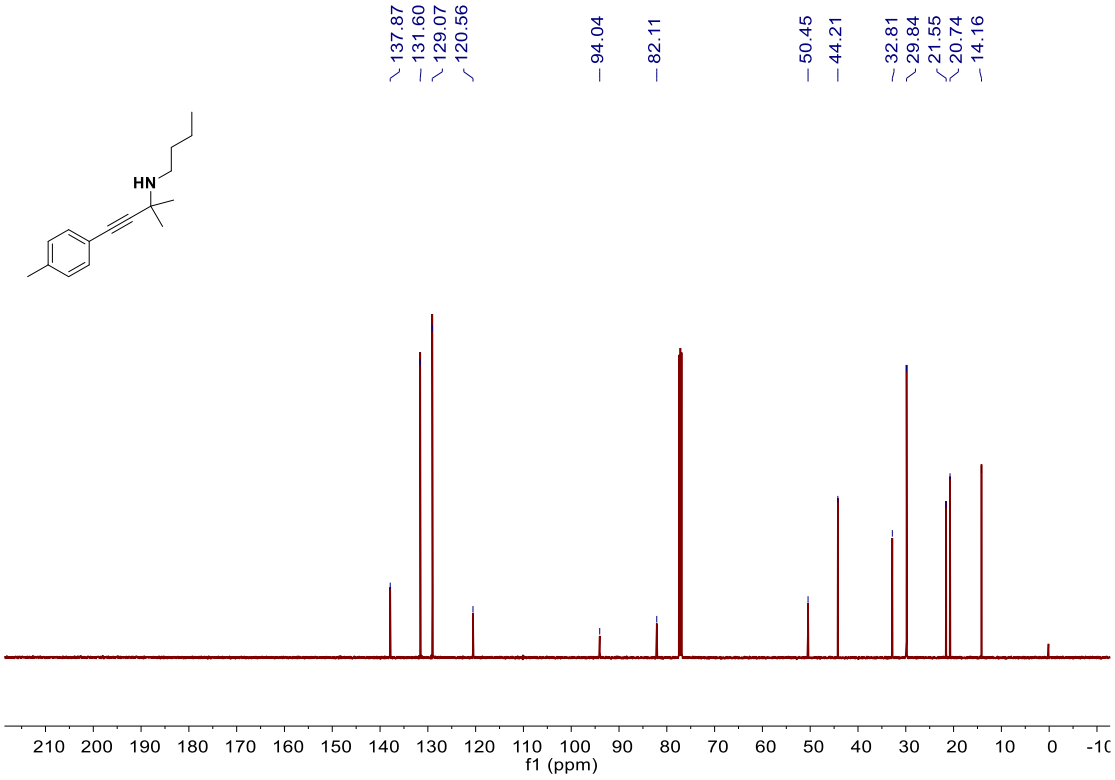
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-butyl-2-methyl-4-phenylbut-3-yn-2-amine (1b)



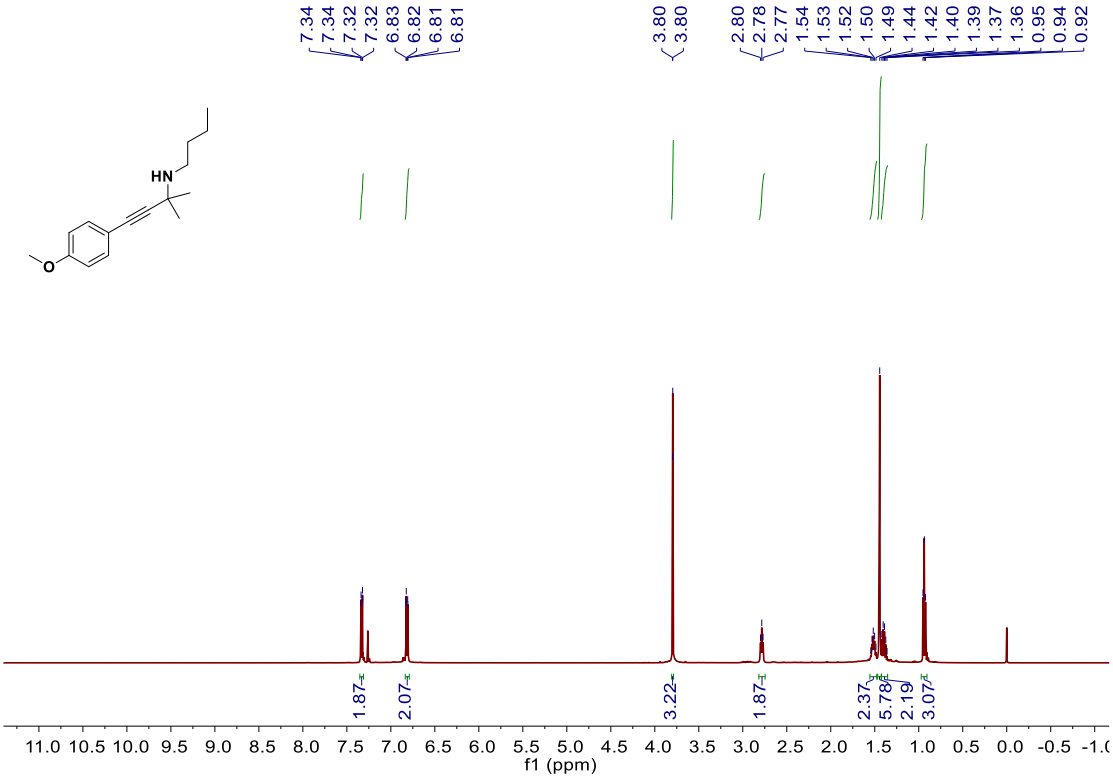
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-butyl-2-methyl-4-(p-tolyl)but-3-yn-2-amine (1c)



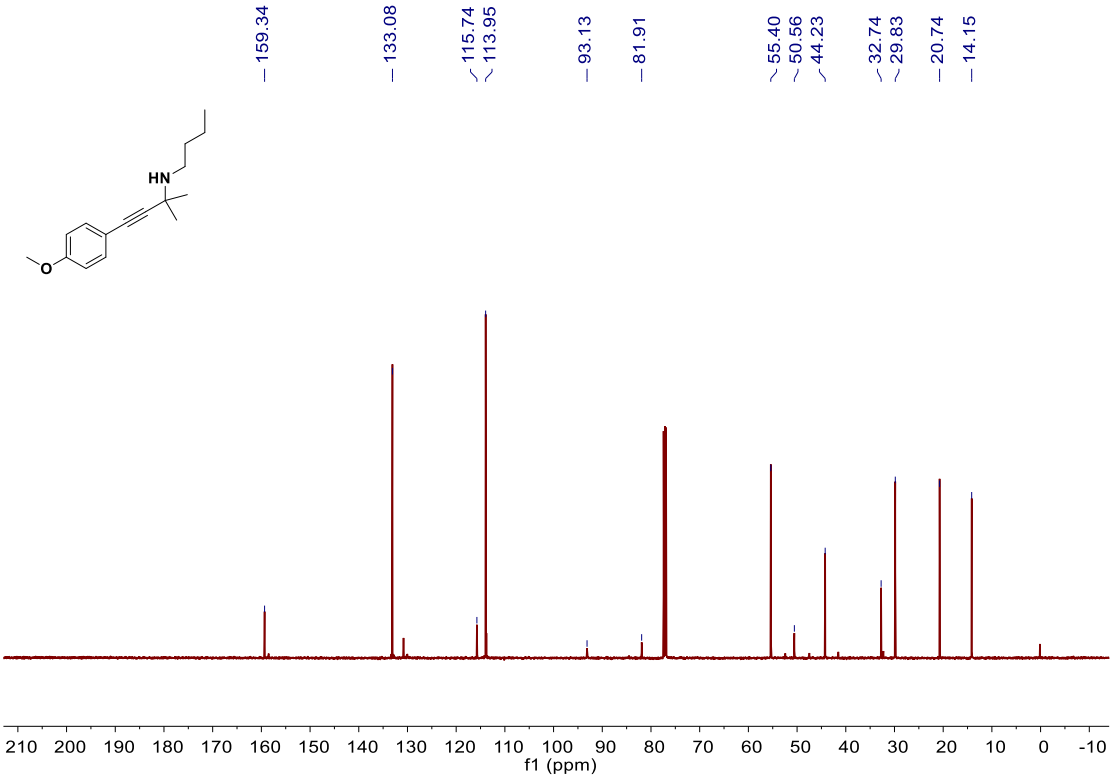
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-butyl-2-methyl-4-(p-tolyl)but-3-yn-2-amine (1c)



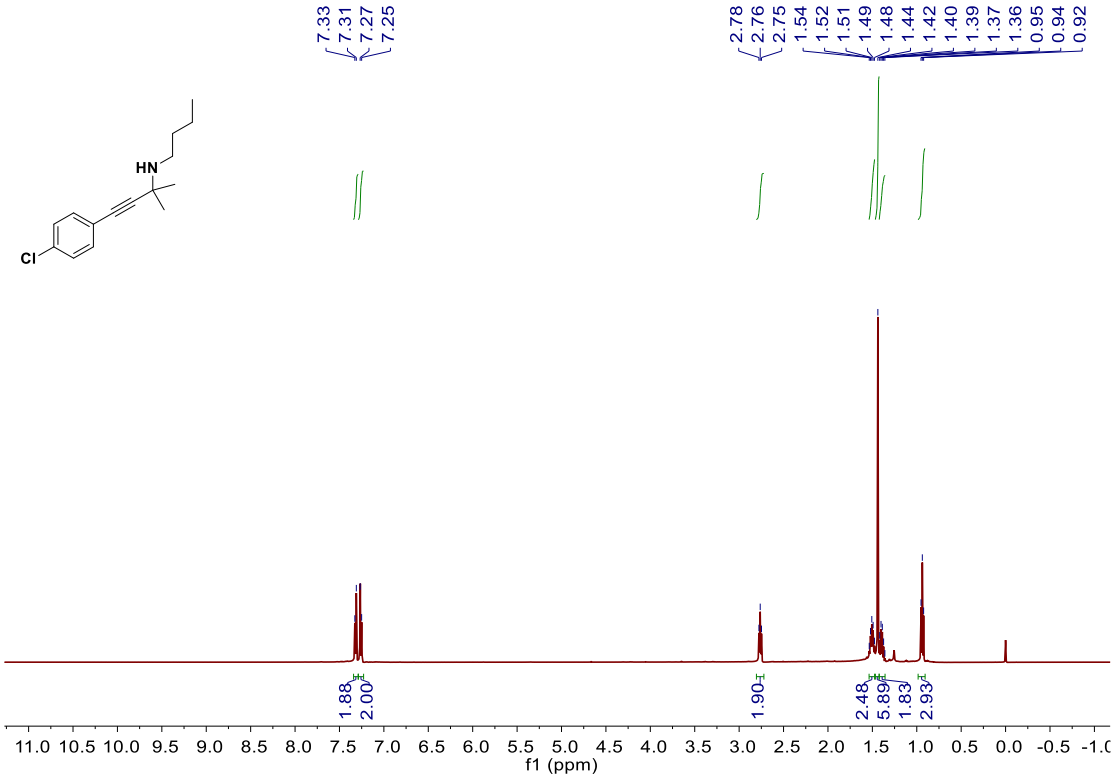
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-butyl-4-(4-methoxyphenyl)-2-methylbut-3-yn-2-amine (1d)



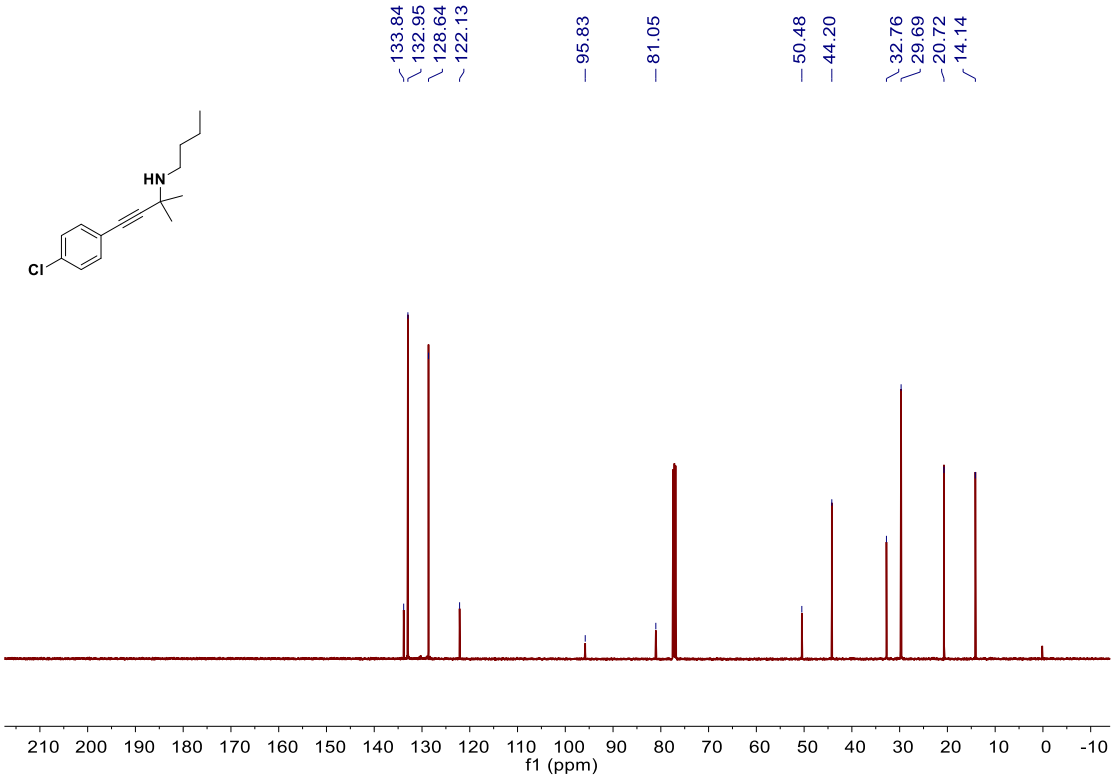
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-butyl-4-(4-methoxyphenyl)-2-methylbut-3-yn-2-amine (1d)



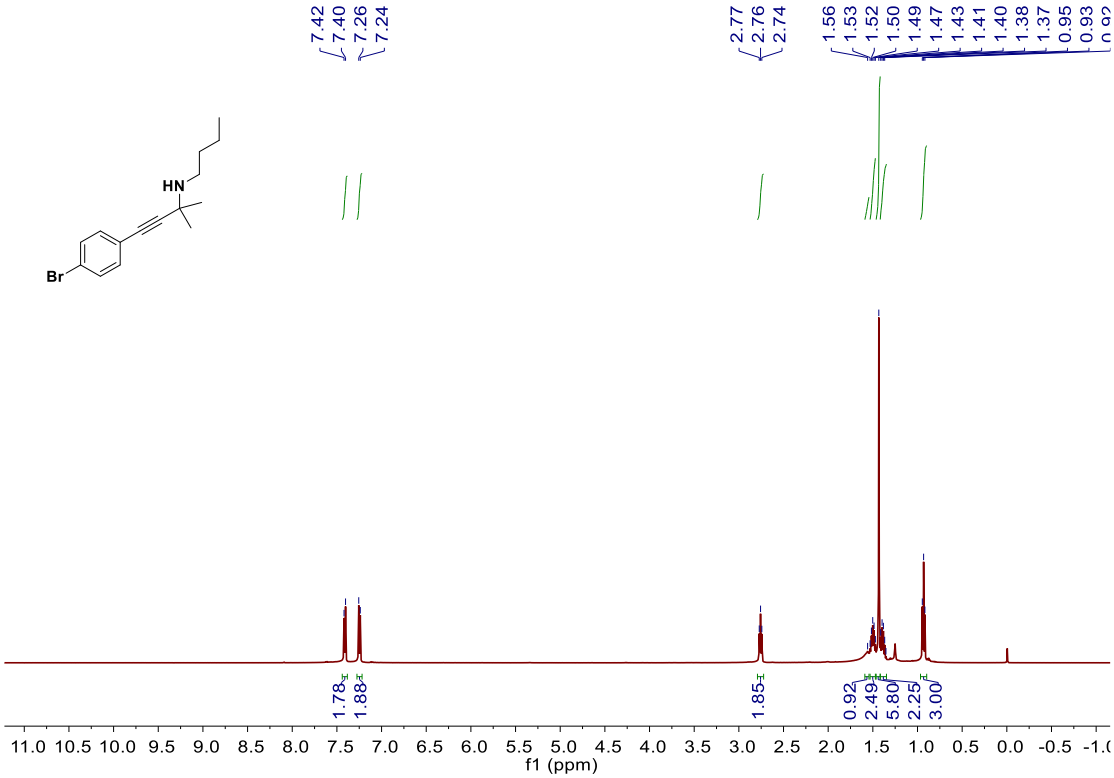
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-butyl-4-(4-chlorophenyl)-2-methylbut-3-yn-2-amine (1e)



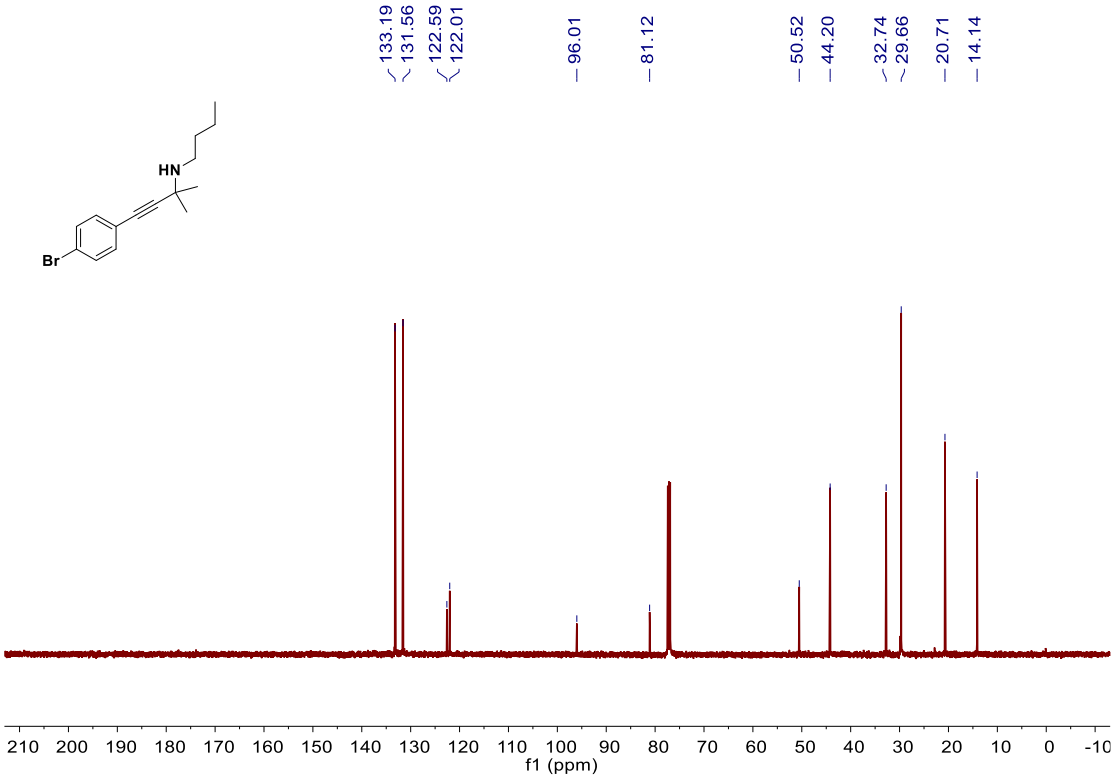
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-butyl-4-(4-chlorophenyl)-2-methylbut-3-yn-2-amine (1e)



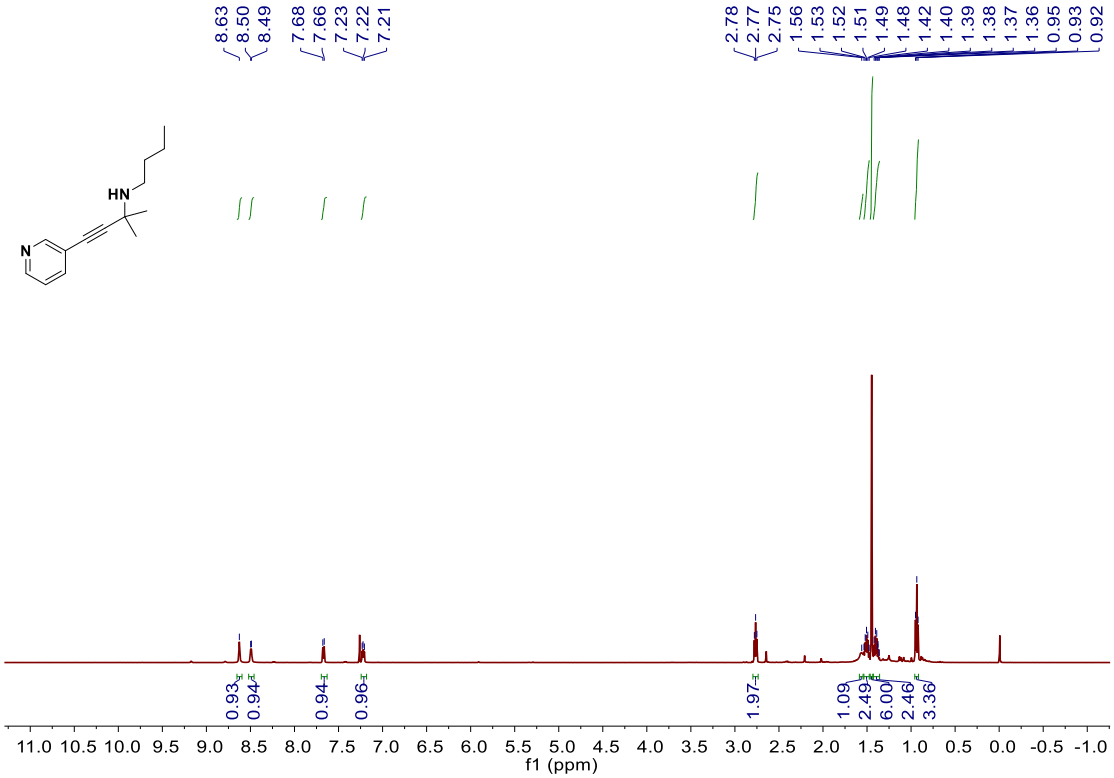
^{1}H NMR ($CDCl_3$, 500 MHz) of <u>4-(4-bromophenyl)-*N*-butyl-2-methylbut-3-yn-2-amine (1f)</u>



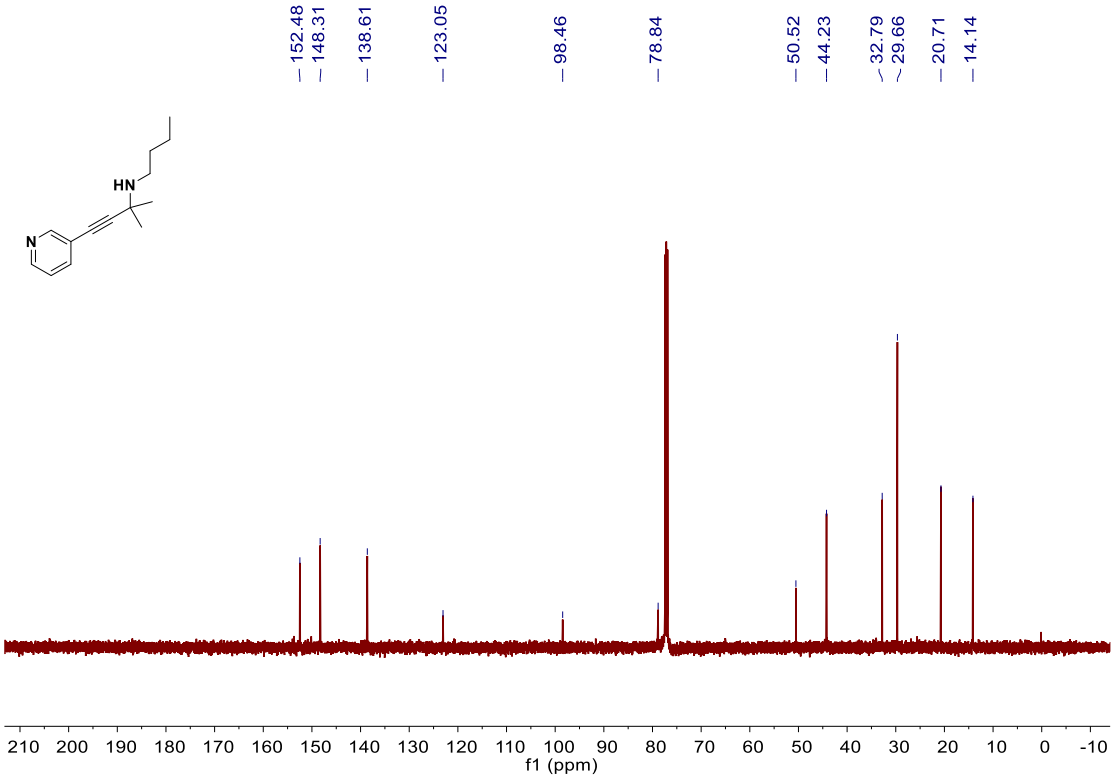
^{13}C NMR ($CDCl_3$, 125 MHz) of <u>4-(4-bromophenyl)-*N*-butyl-2-methylbut-3-yn-2-amine (1f)</u>



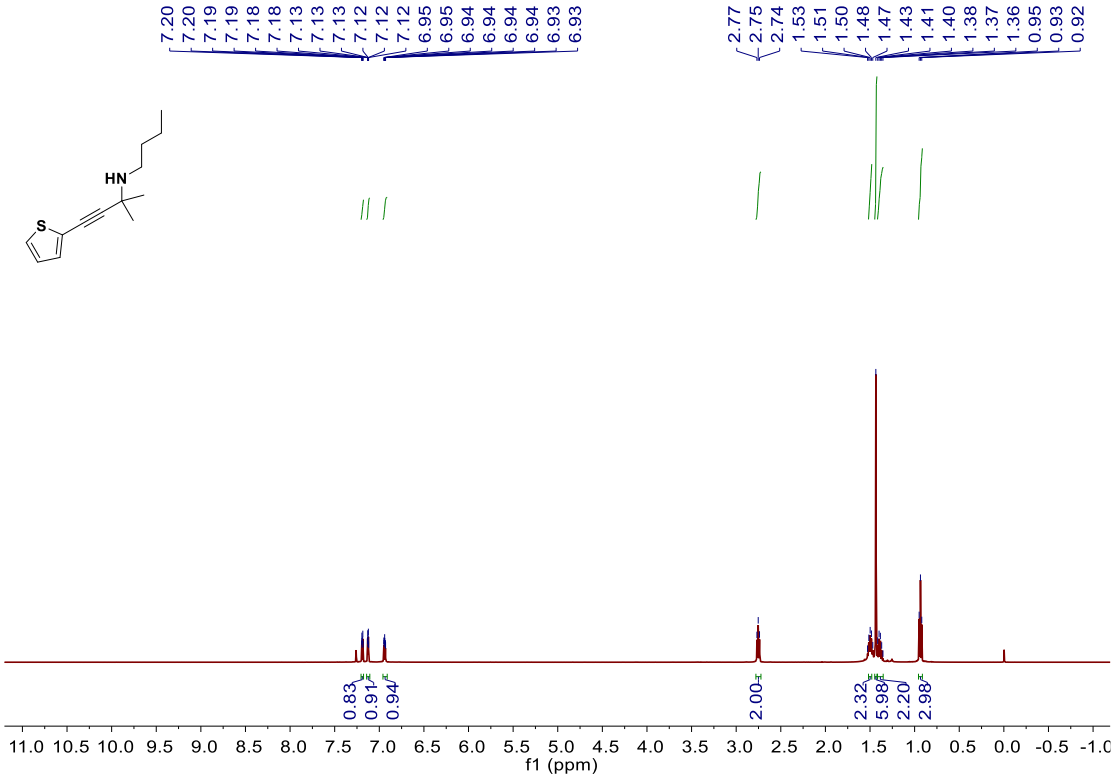
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-butyl-2-methyl-4-(pyridin-3-yl)but-3-yn-2-amine (1g)



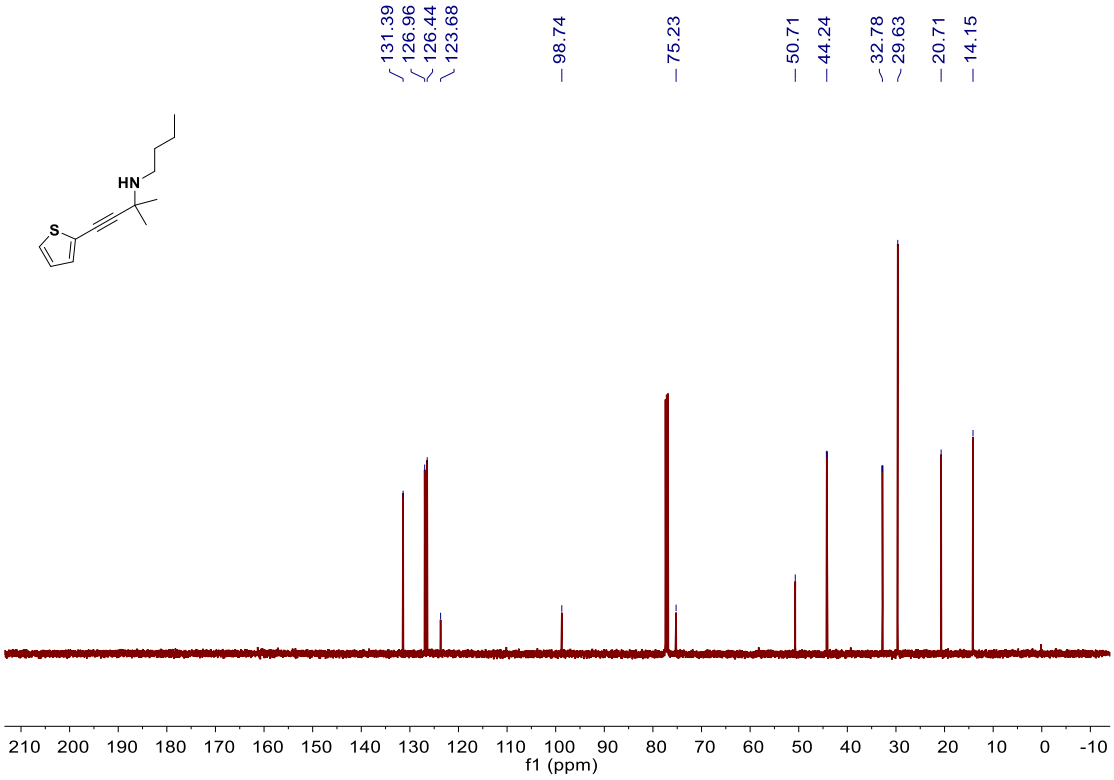
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-butyl-2-methyl-4-(pyridin-3-yl)but-3-yn-2-amine (1g)



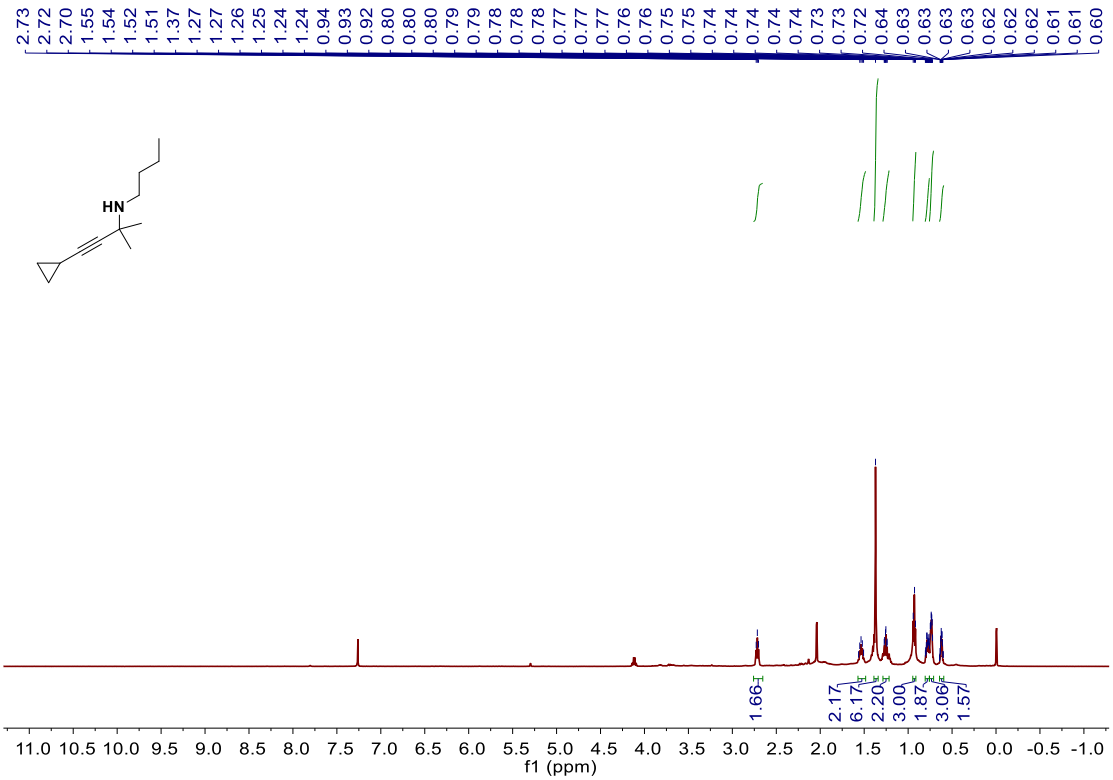
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-butyl-2-methyl-4-(thiophen-2-yl)but-3-yn-2-amine (1h)



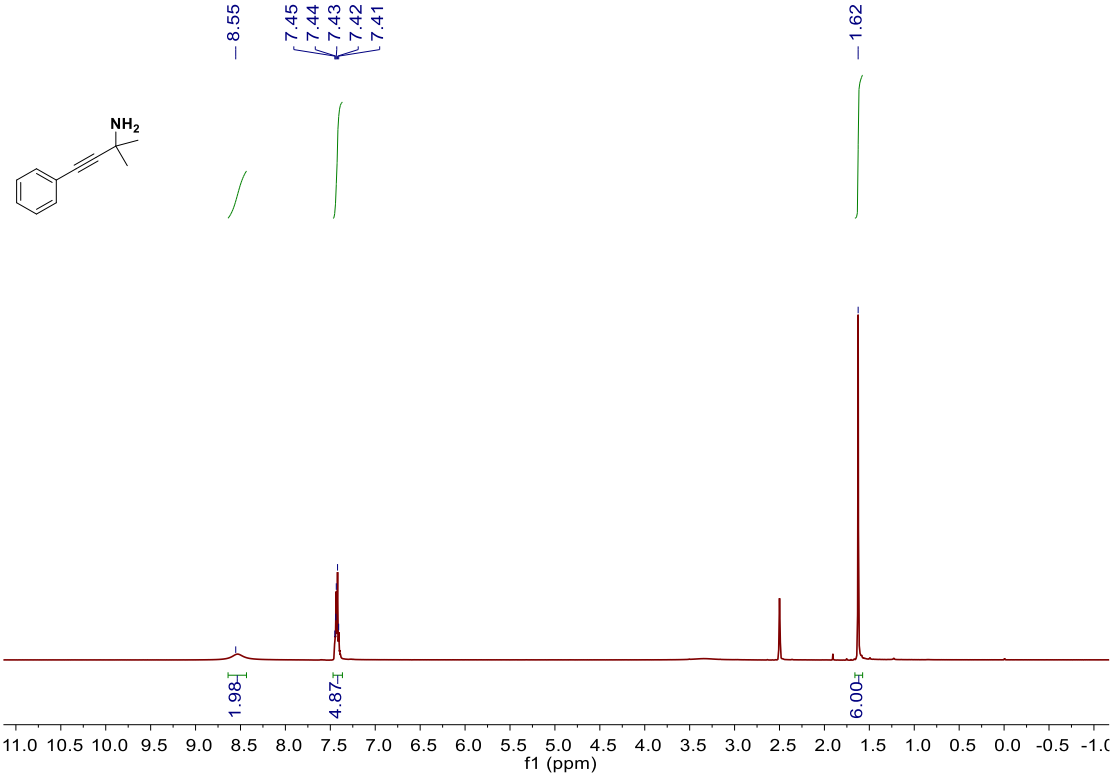
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-butyl-2-methyl-4-(thiophen-2-yl)but-3-yn-2-amine (1h)



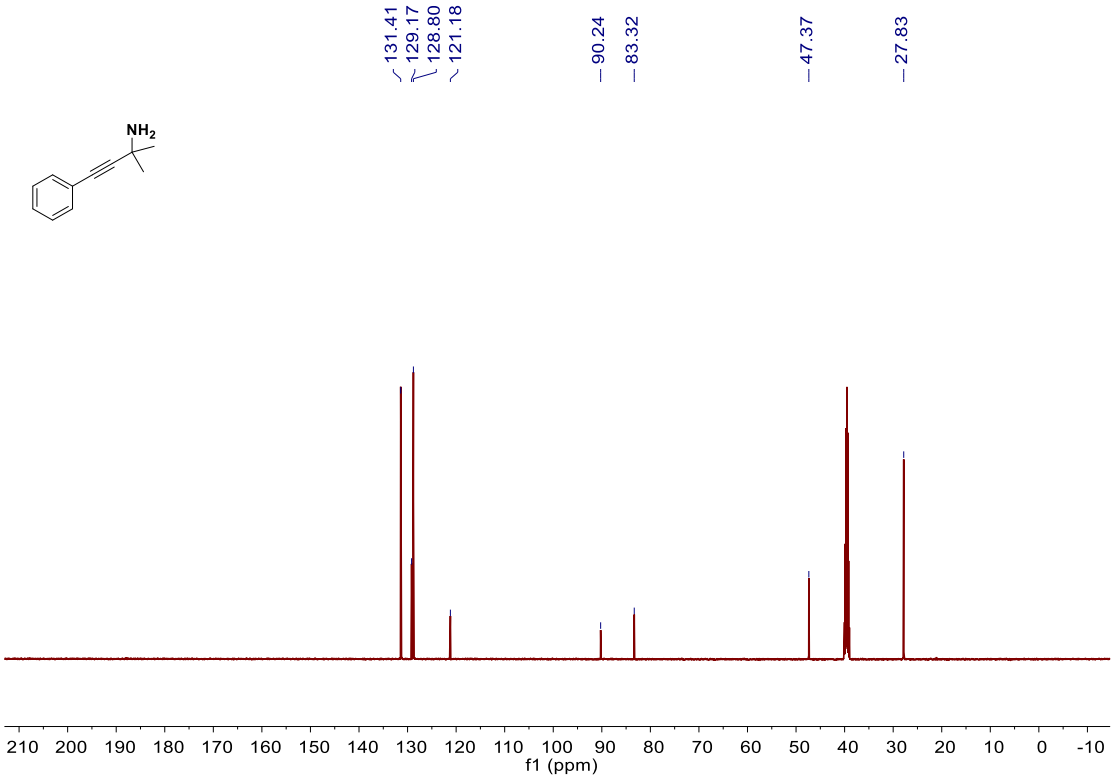
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-butyl-4-cyclopropyl-2-methylbut-3-yn-2-amine (1i)



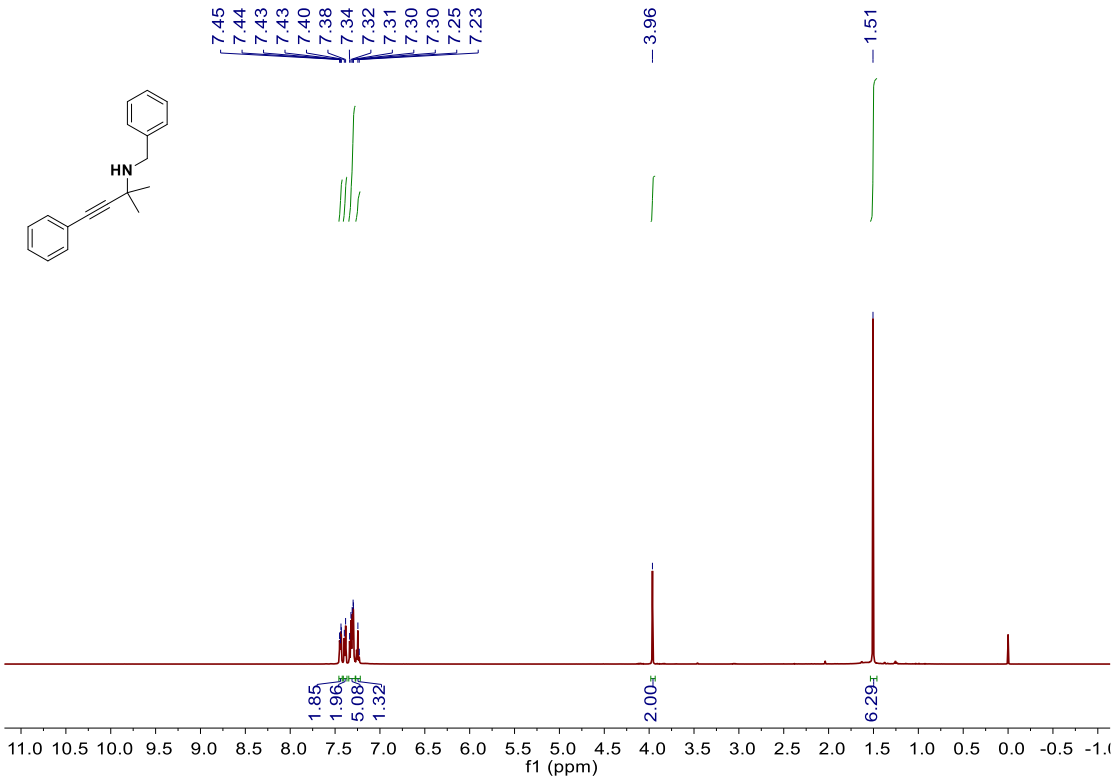
^{1}H NMR (DMSO-d_6, 500 MHz) of 2-methyl-4-phenylbut-3-yn-2-amine (1j)



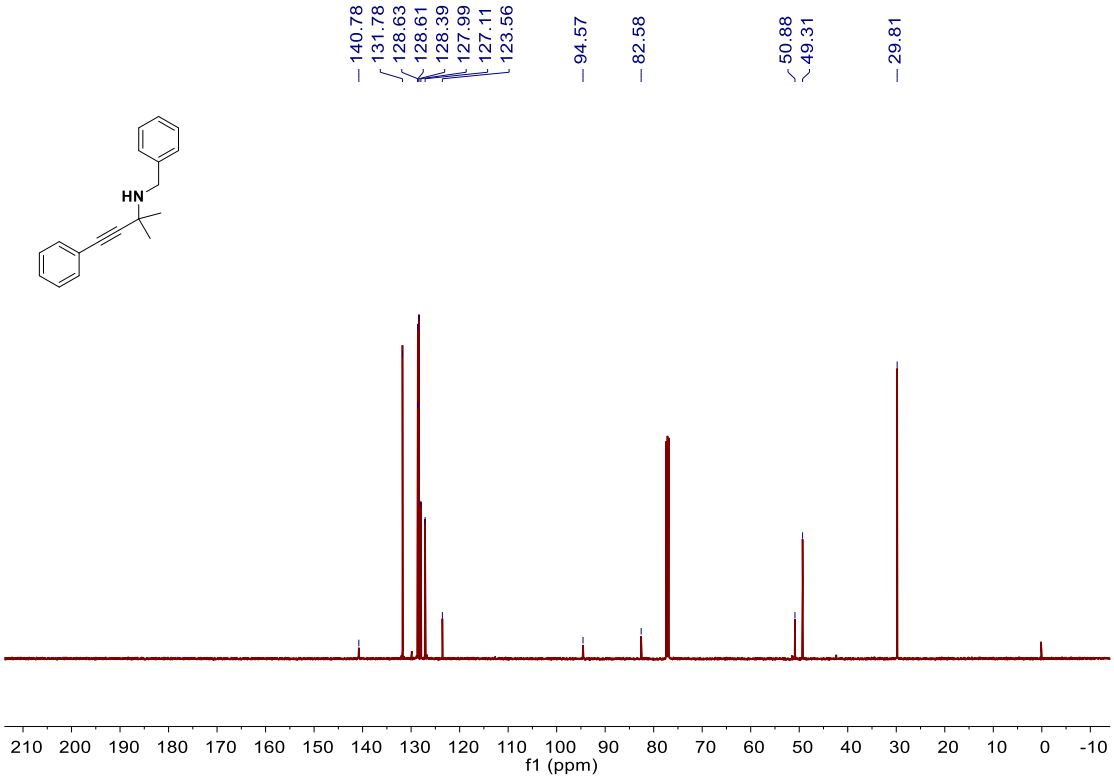
^{13}C NMR (DMSO-d_6, 125 MHz) of <u>2-methyl-4-phenylbut-3-yn-2-amine (1j)</u>



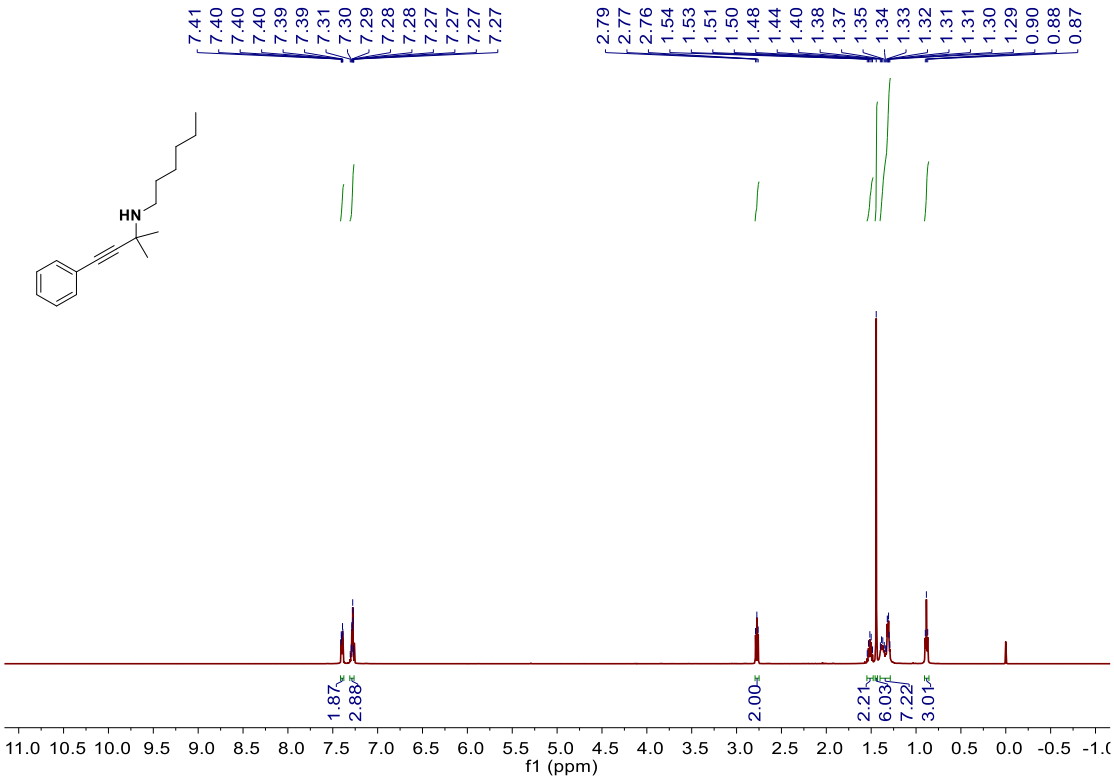
^{1}H NMR ($CDCl_3$, 500 MHz) of <u>*N*-benzyl-2-methyl-4-phenylbut-3-yn-2-amine (1k)</u>



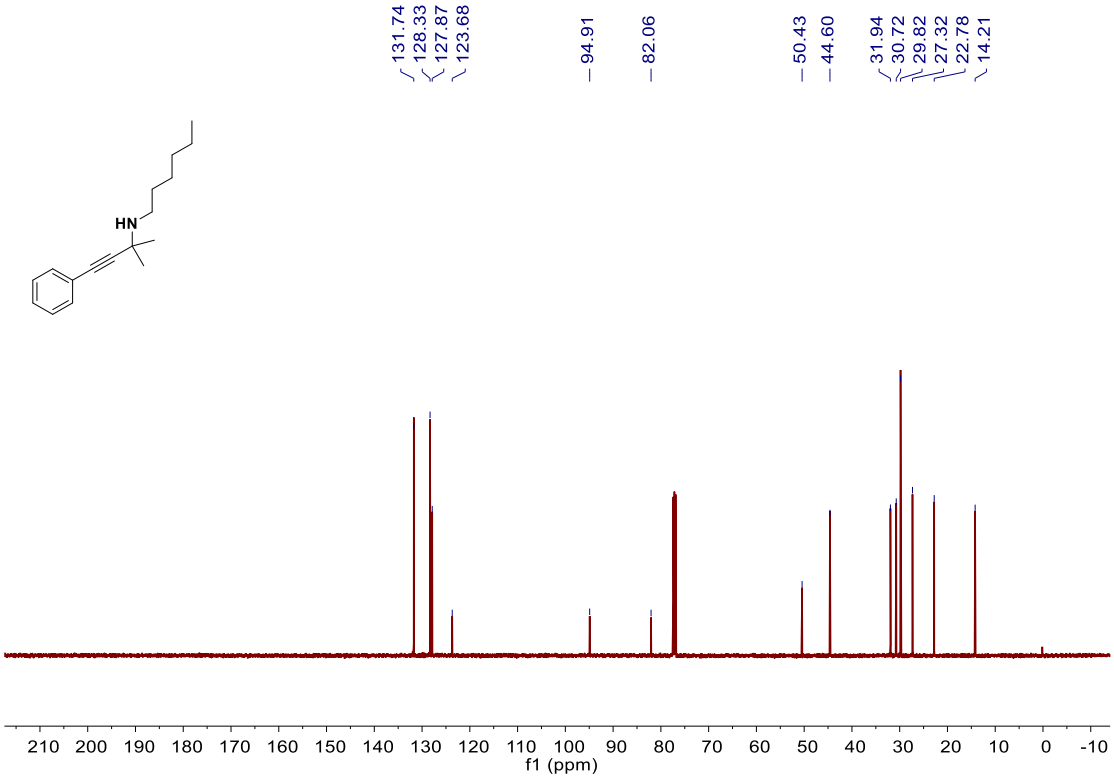
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-benzyl-2-methyl-4-phenylbut-3-yn-2-amine (1k)



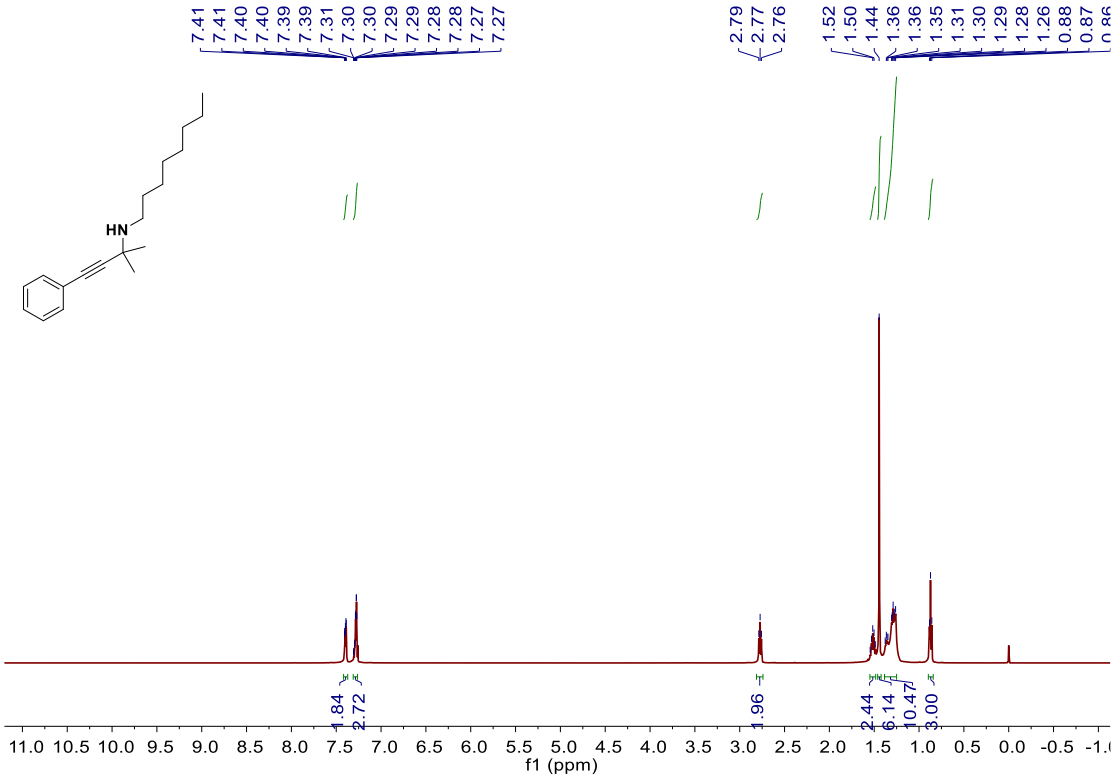
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-(2-methyl-4-phenylbut-3-yn-2-yl)hexan-1-amine (1l)



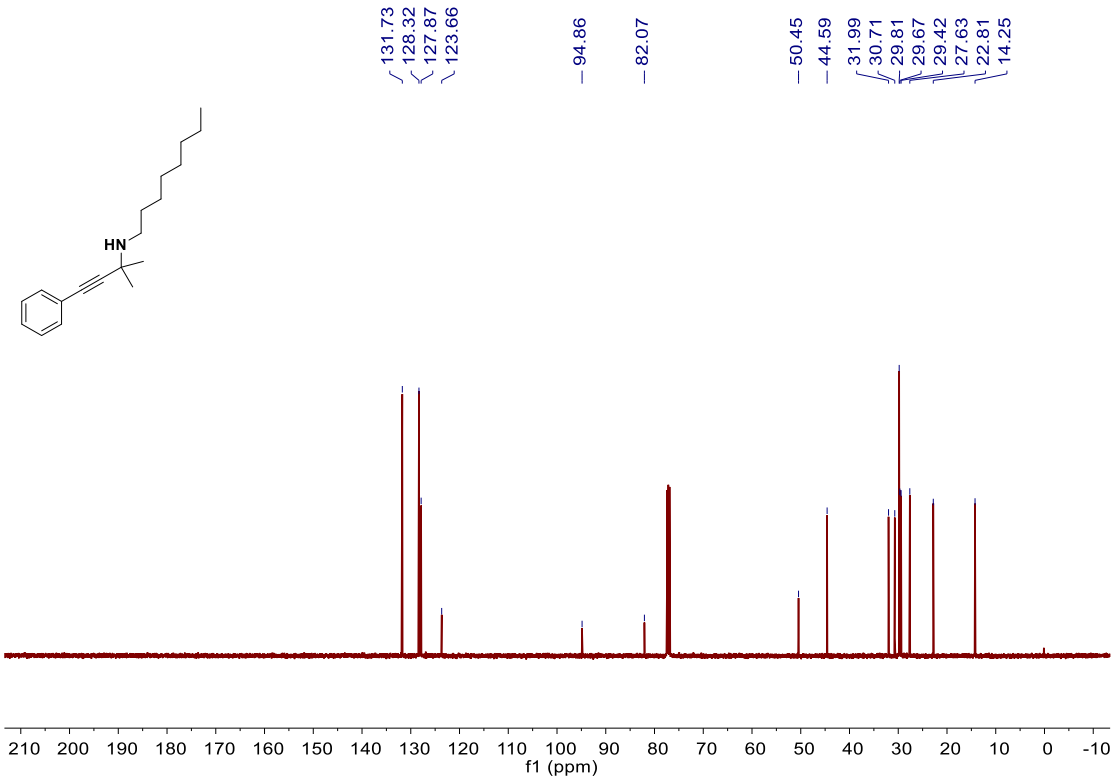
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-(2-methyl-4-phenylbut-3-yn-2-yl)hexan-1-amine (1l)



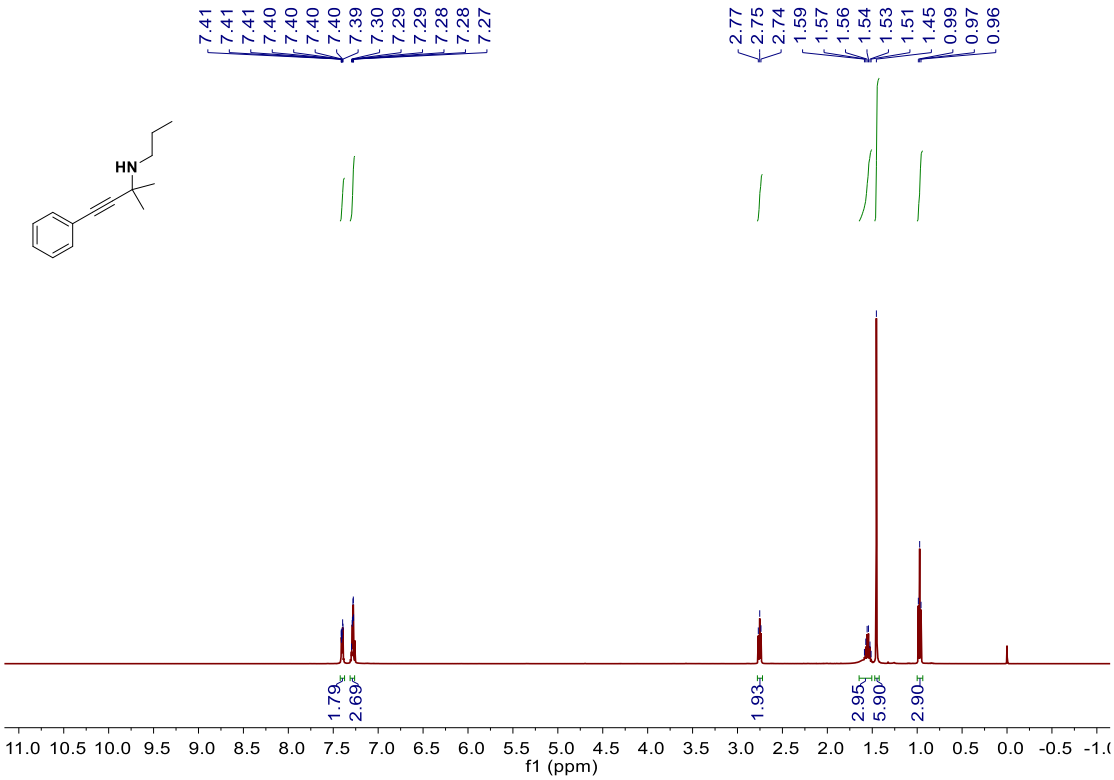
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-(2-methyl-4-phenylbut-3-yn-2-yl)octan-1-amine (1m)



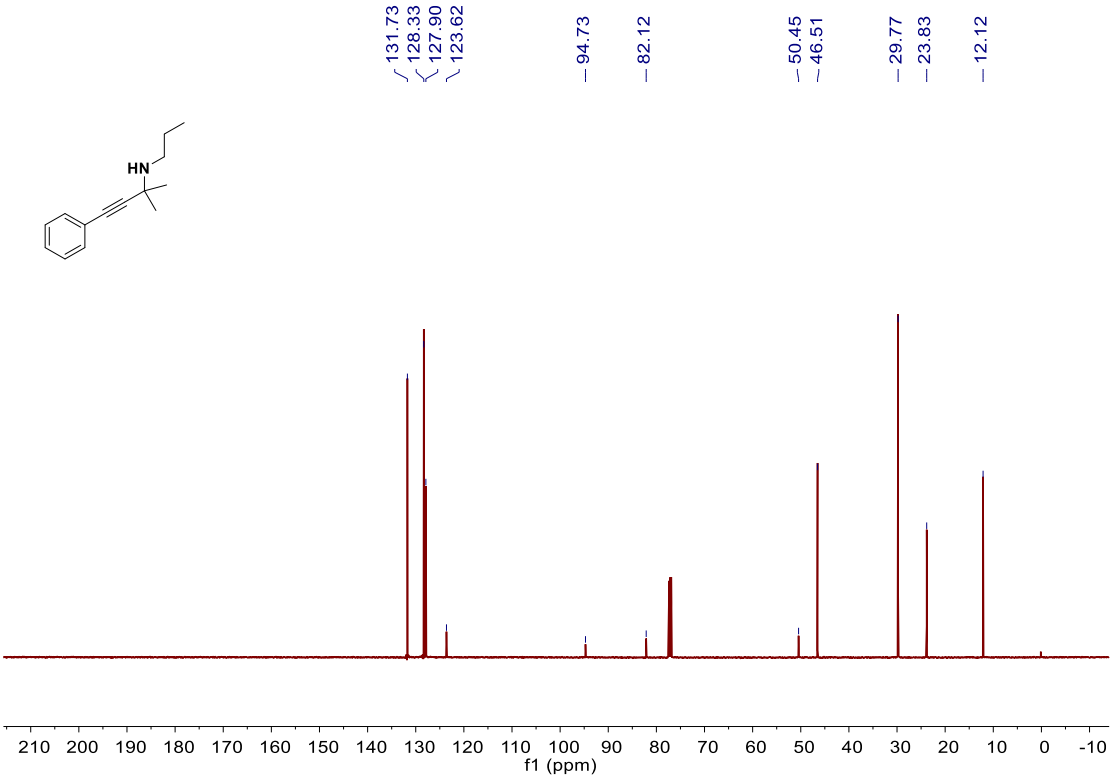
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-(2-methyl-4-phenylbut-3-yn-2-yl)octan-1-amine (1m)



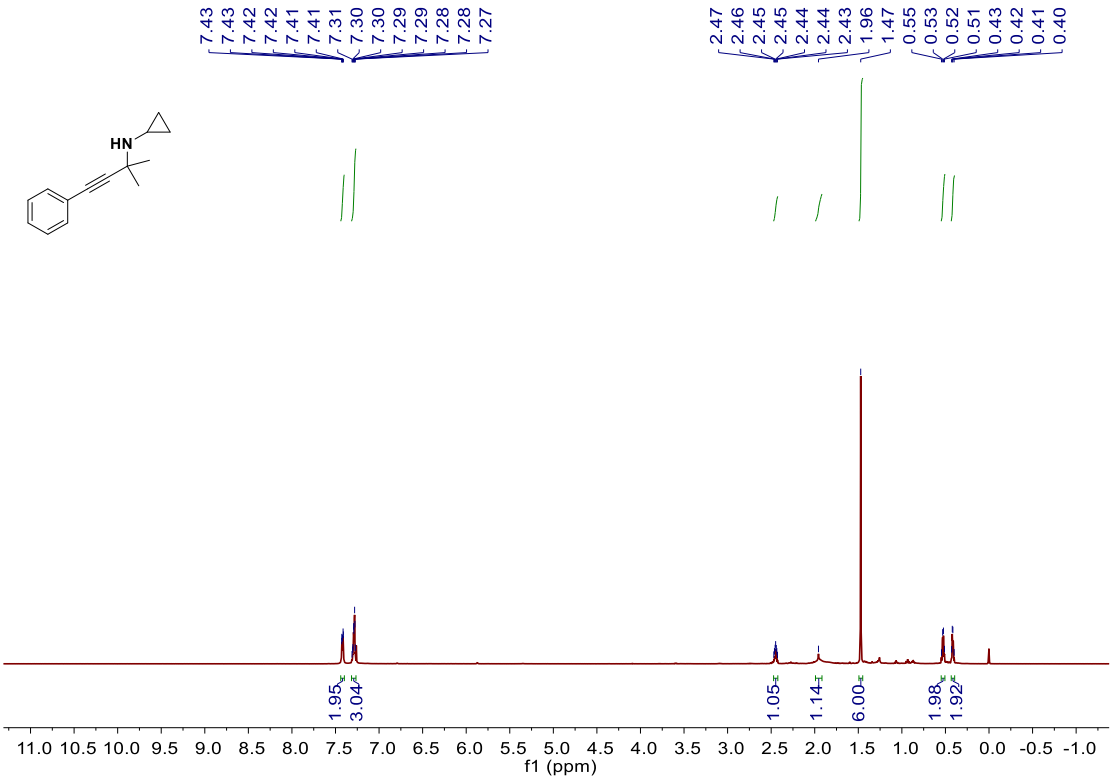
^{1}H NMR ($CDCl_3$, 500 MHz) of 2-methyl-4-phenyl-*N*-propylbut-3-yn-2-amine (1n)



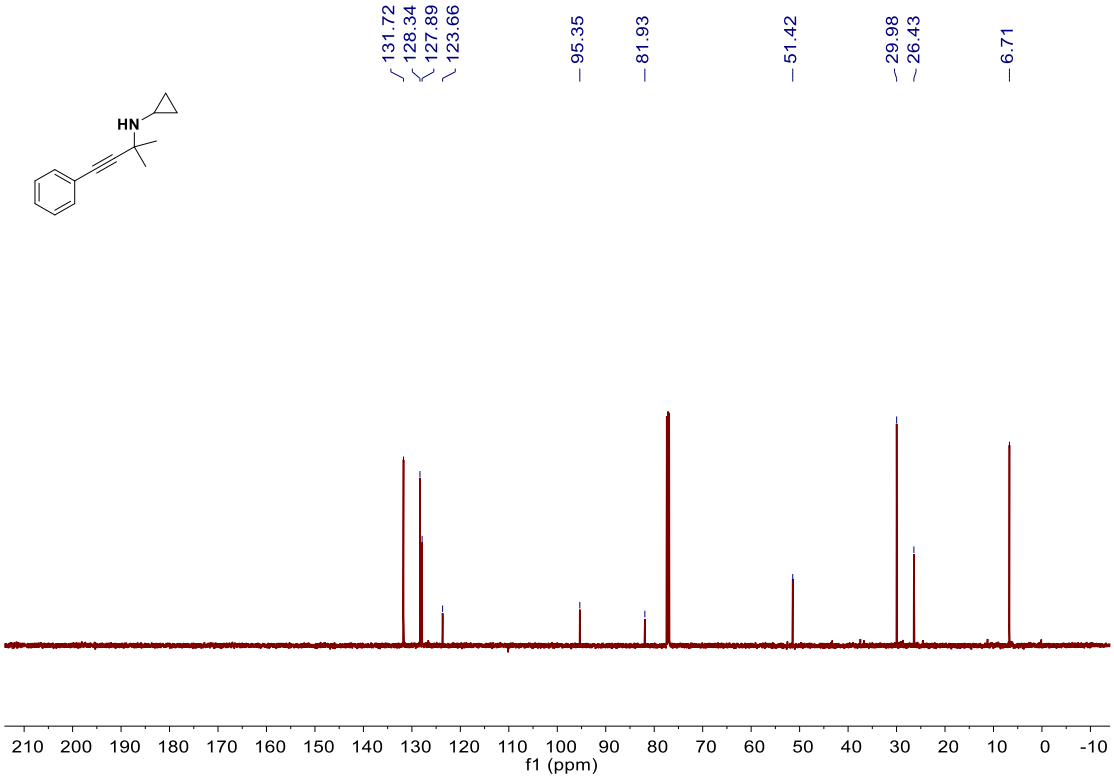
^{13}C NMR ($CDCl_3$, 125 MHz) of 2-methyl-4-phenyl-*N*-propylbut-3-yn-2-amine (1n)



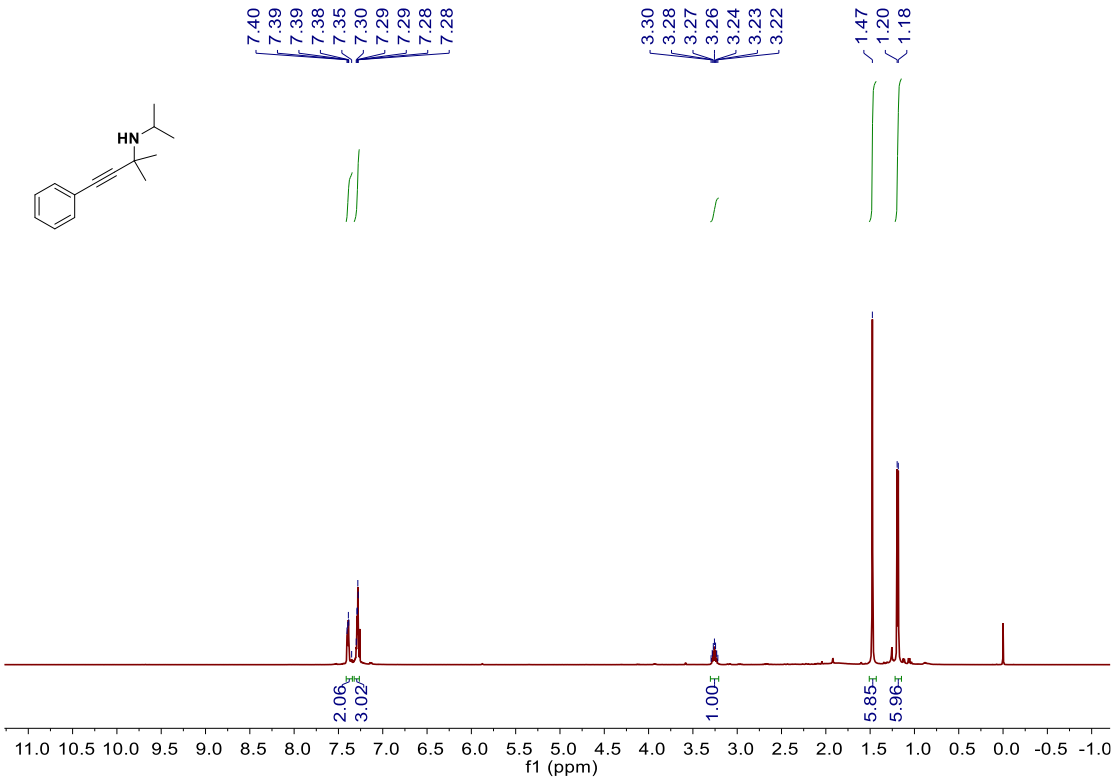
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-(2-methyl-4-phenylbut-3-yn-2-yl)cyclopropanamine (1o)



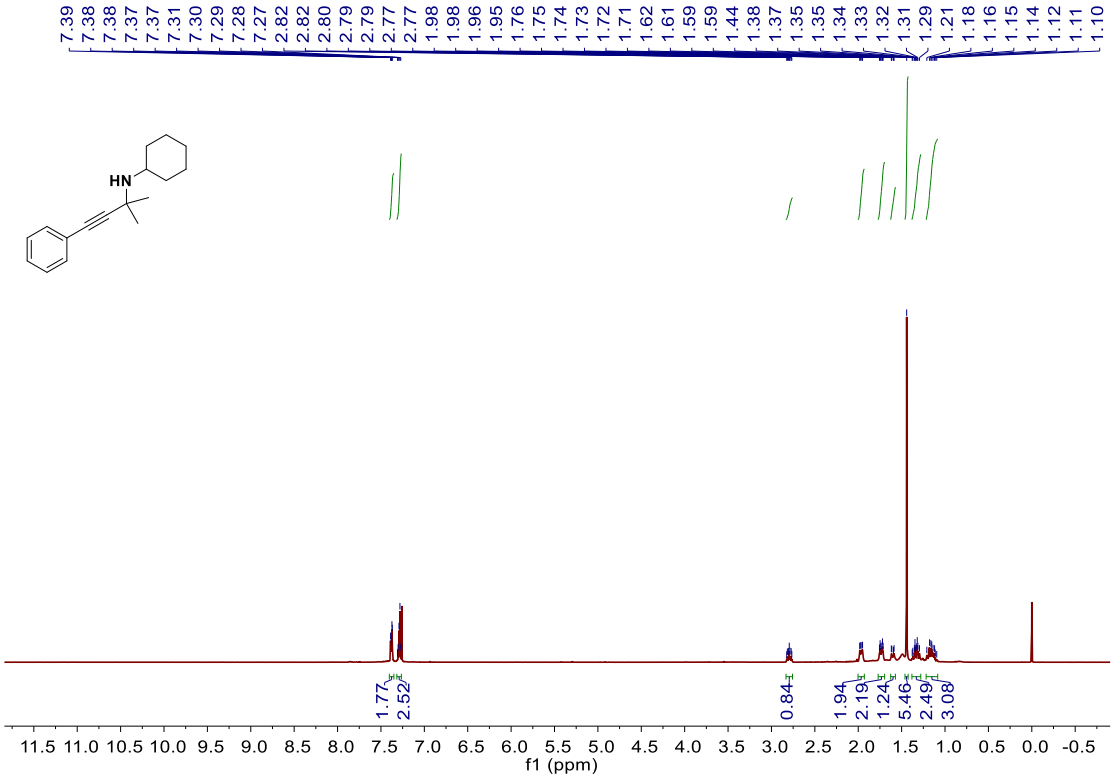
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-(2-methyl-4-phenylbut-3-yn-2-yl)cyclopropanamine (1o)



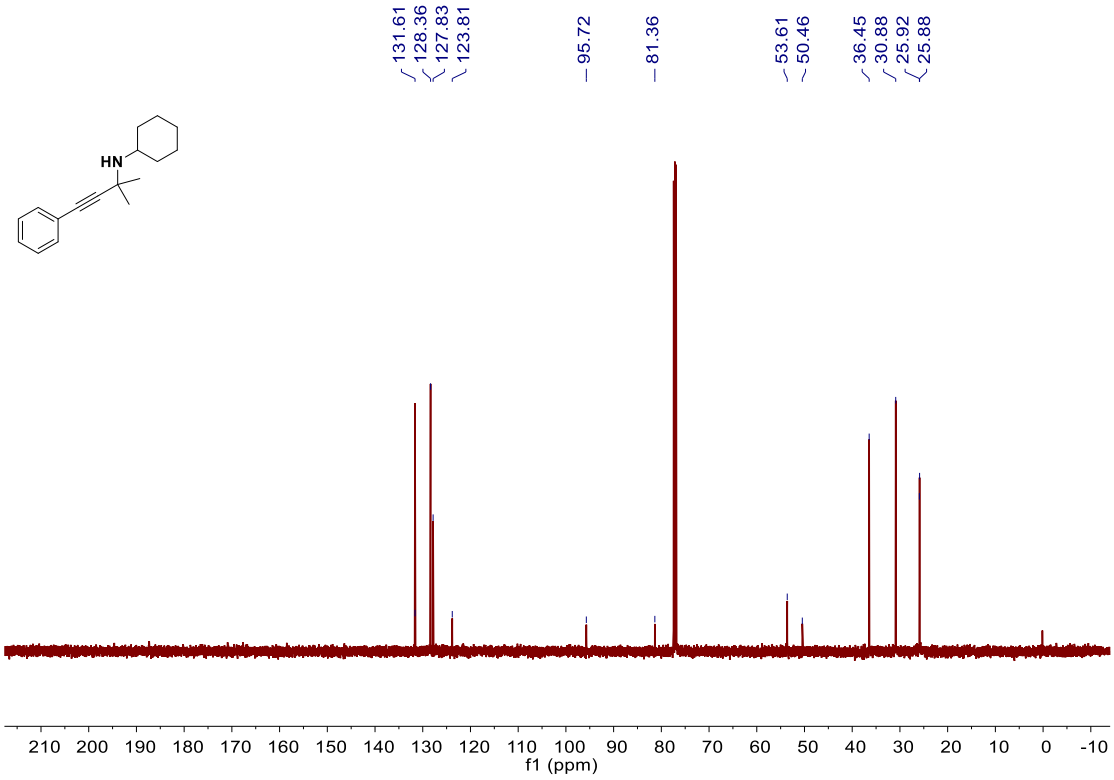
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-isopropyl-2-methyl-4-phenylbut-3-yn-2-amine (1p)



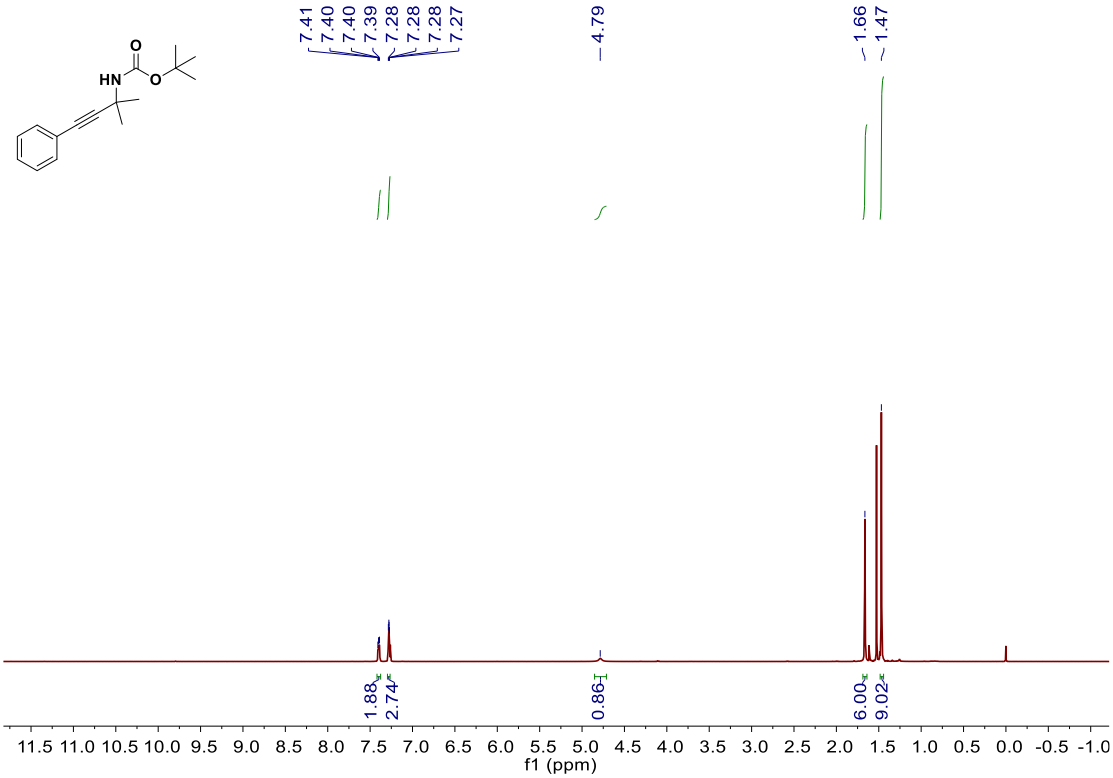
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-(2-methyl-4-phenylbut-3-yn-2-yl)cyclohexanamine (1q)



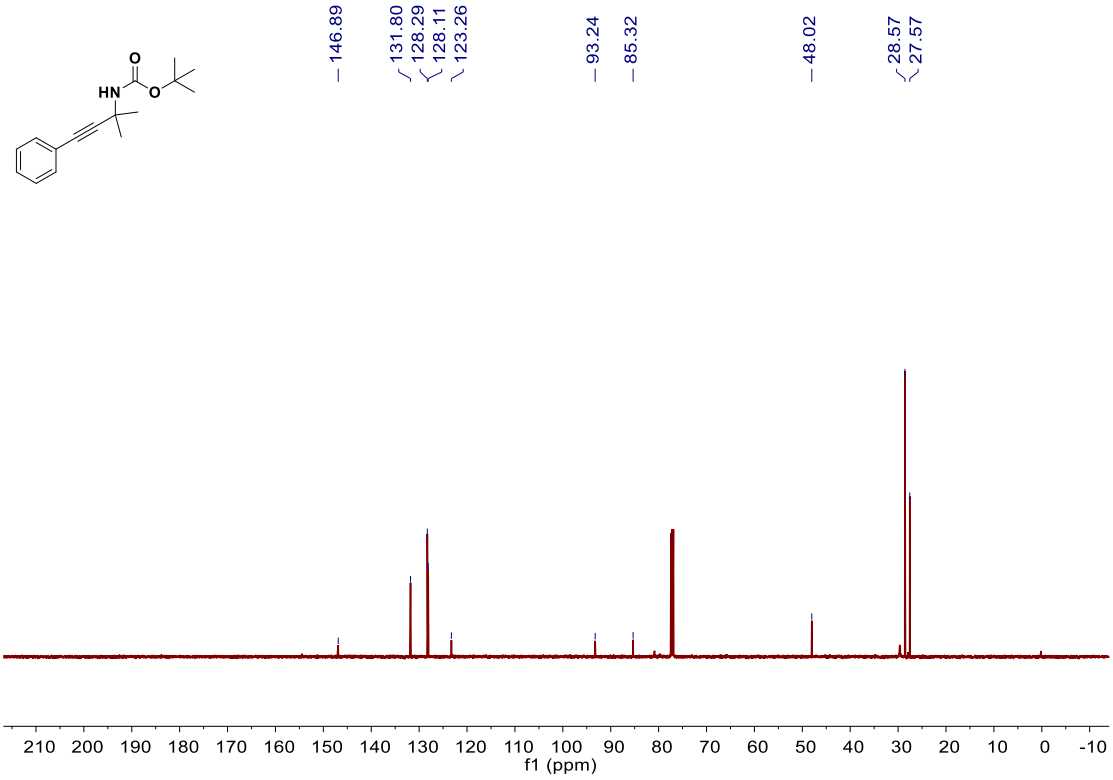
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-(2-methyl-4-phenylbut-3-yn-2-yl)cyclohexanamine (1q)



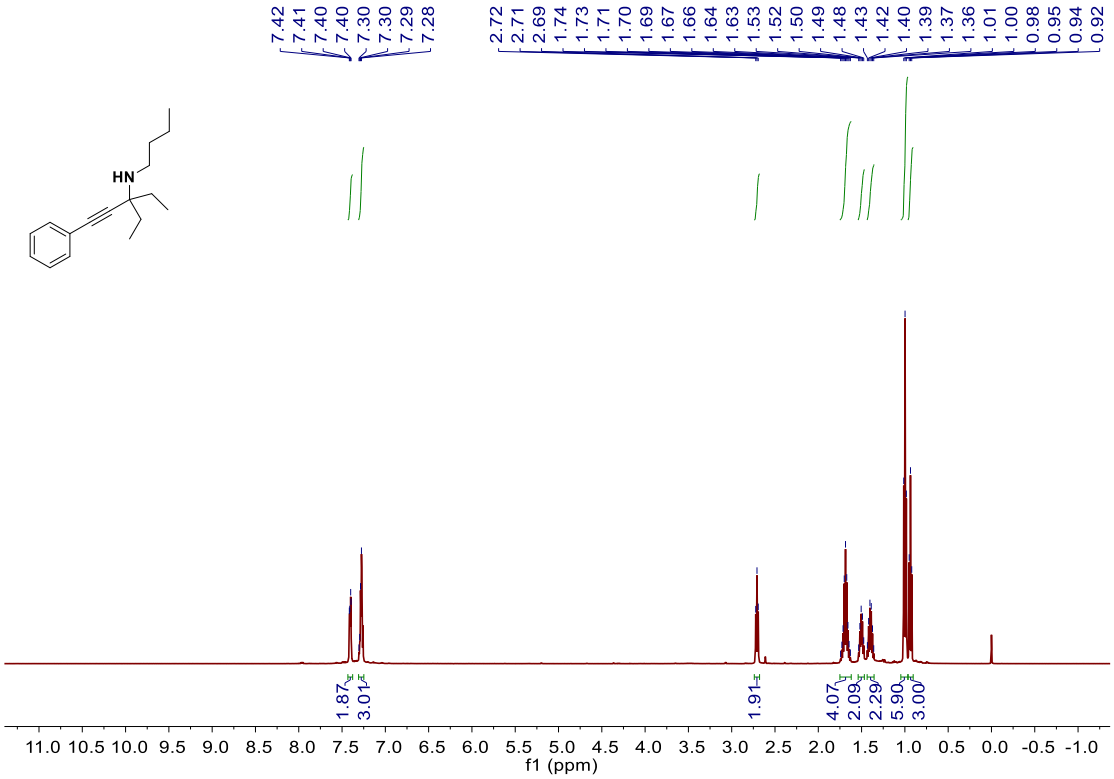
^{1}H NMR ($CDCl_3$, 500 MHz) of *tert*-butyl (2-methyl-4-phenylbut-3-yn-2-yl)carbamate (1r)



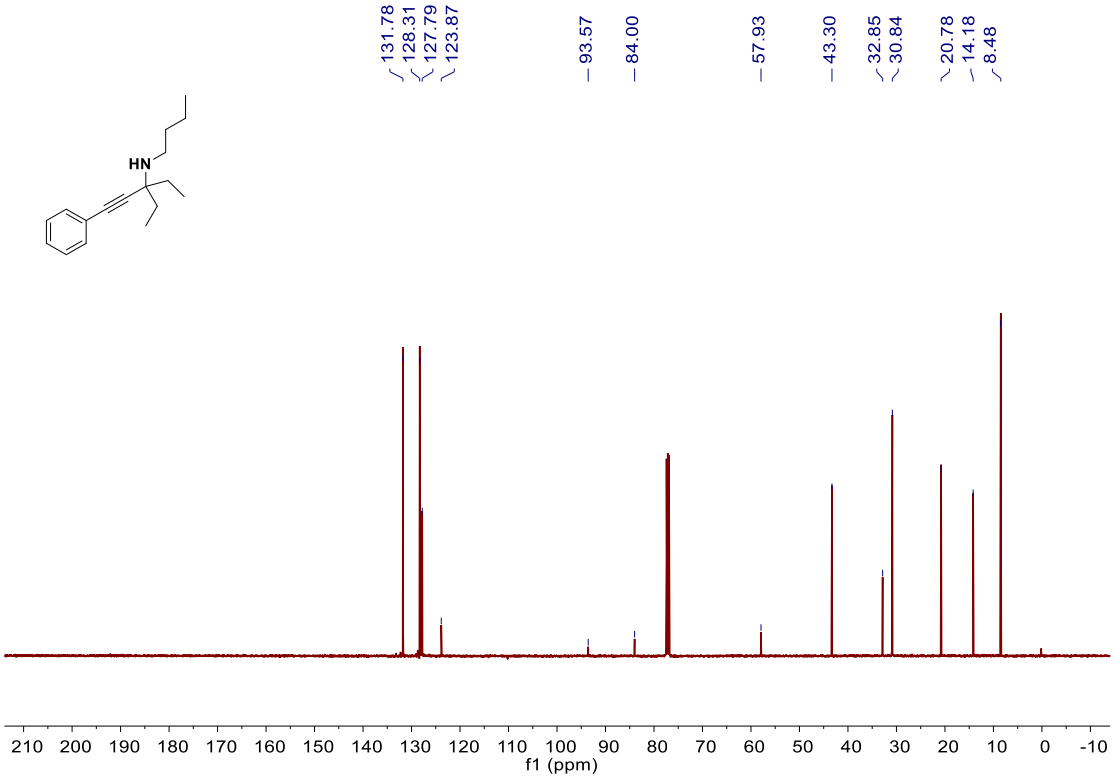
^{13}C NMR ($CDCl_3$, 125 MHz) of *tert*-butyl (2-methyl-4-phenylbut-3-yn-2-yl)carbamate (1r)



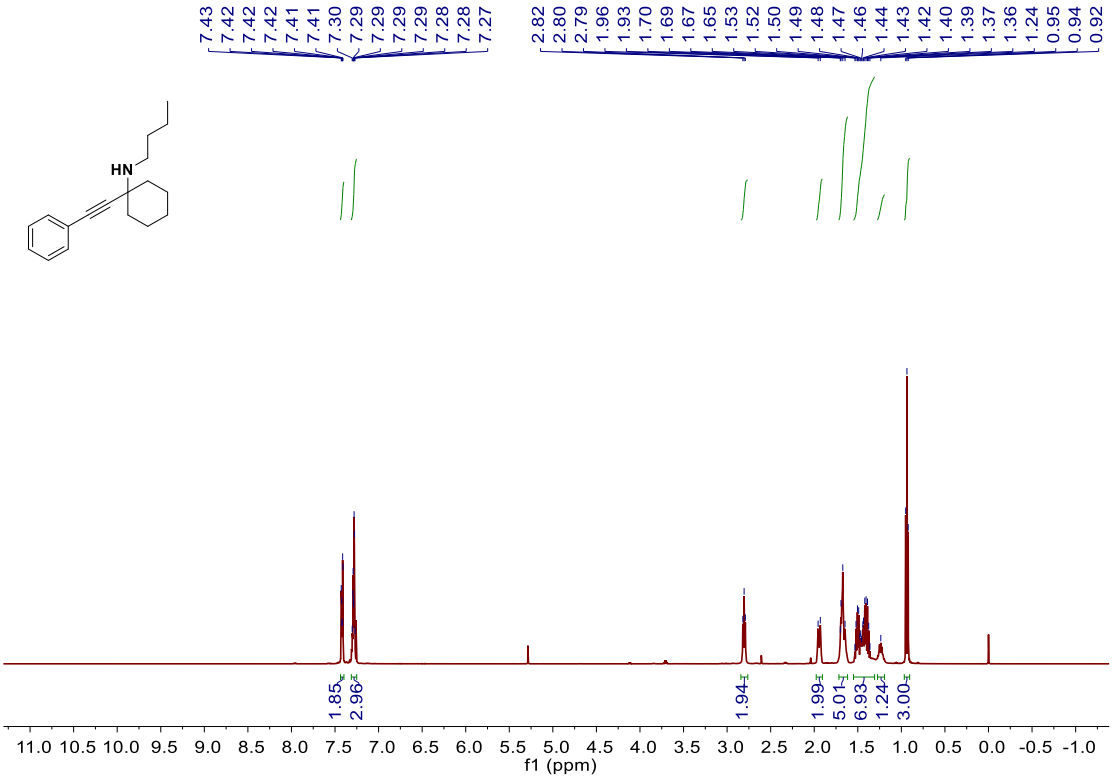
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-butyl-3-ethyl-1-phenylpent-1-yn-3-amine (1s)



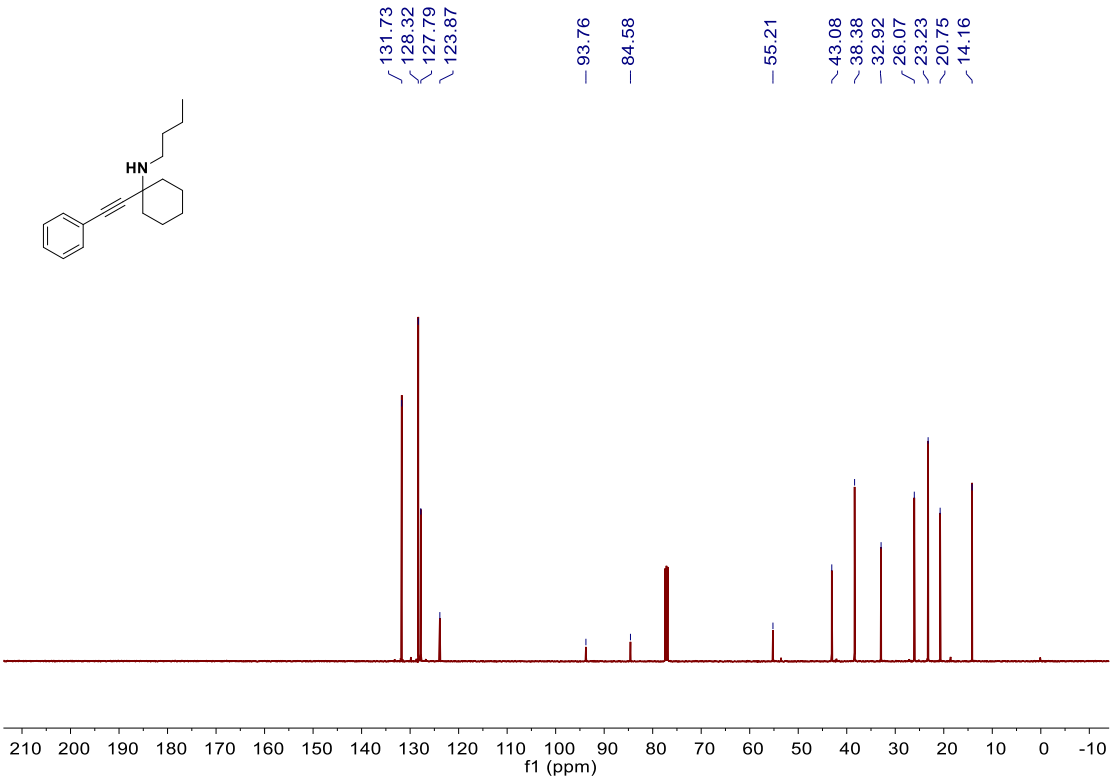
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-butyl-3-ethyl-1-phenylpent-1-yn-3-amine (1s)



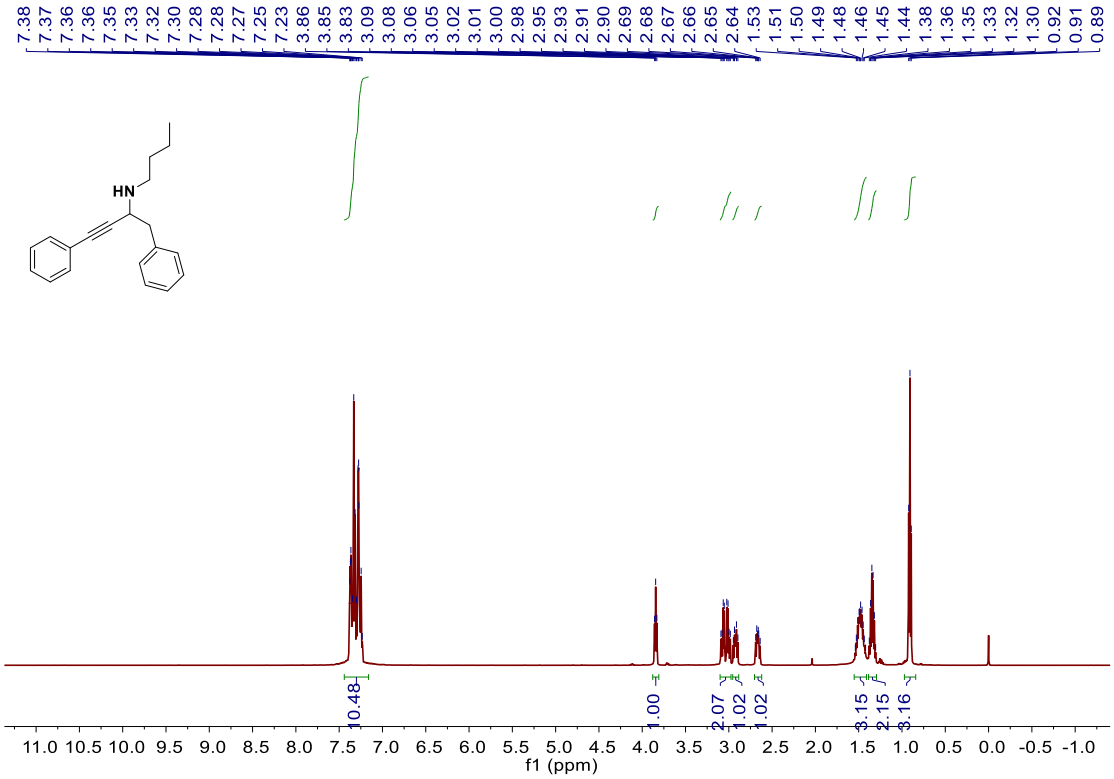
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-butyl-1-(phenylethynyl)cyclohexan-1-amine (1t)



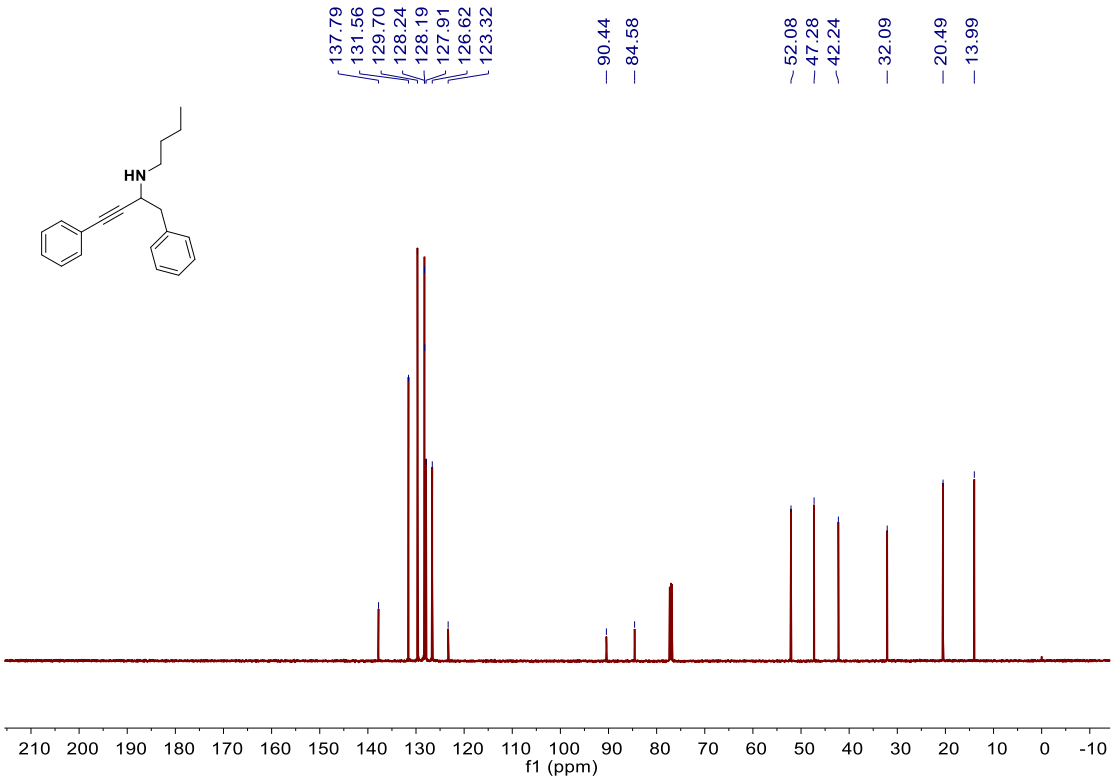
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-butyl-1-(phenylethynyl)cyclohexan-1-amine (1t)



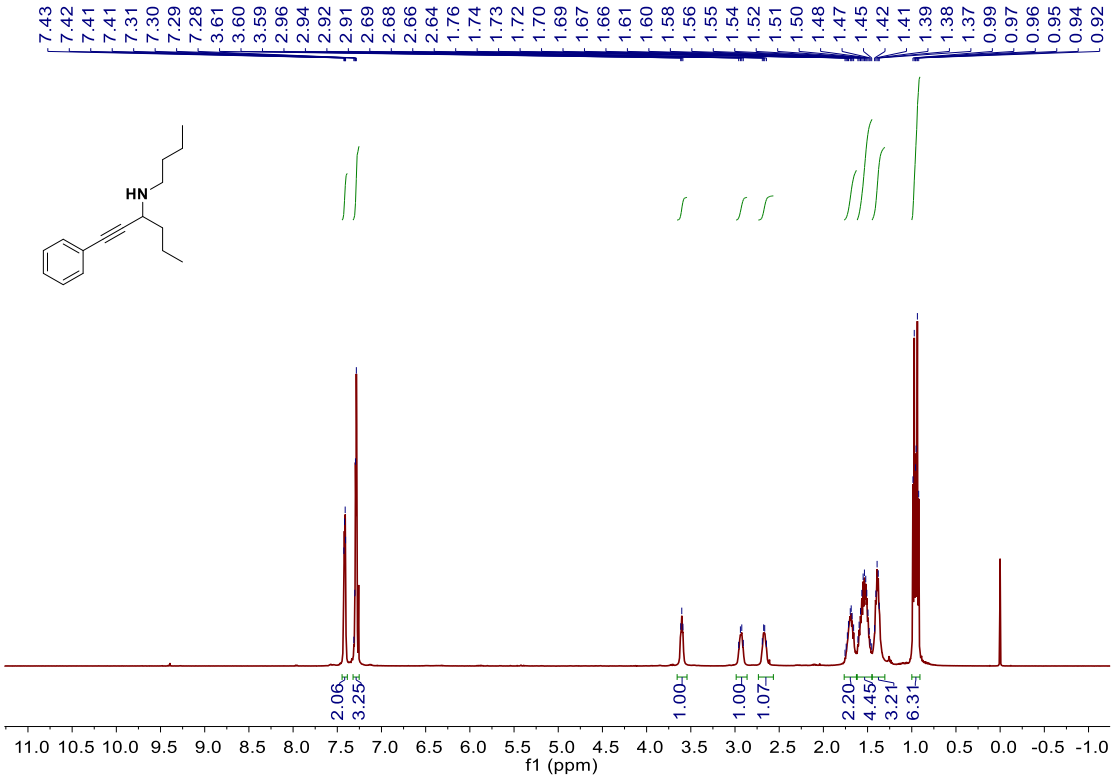
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-butyl-1,4-diphenylbut-3-yn-2-amine (1u)



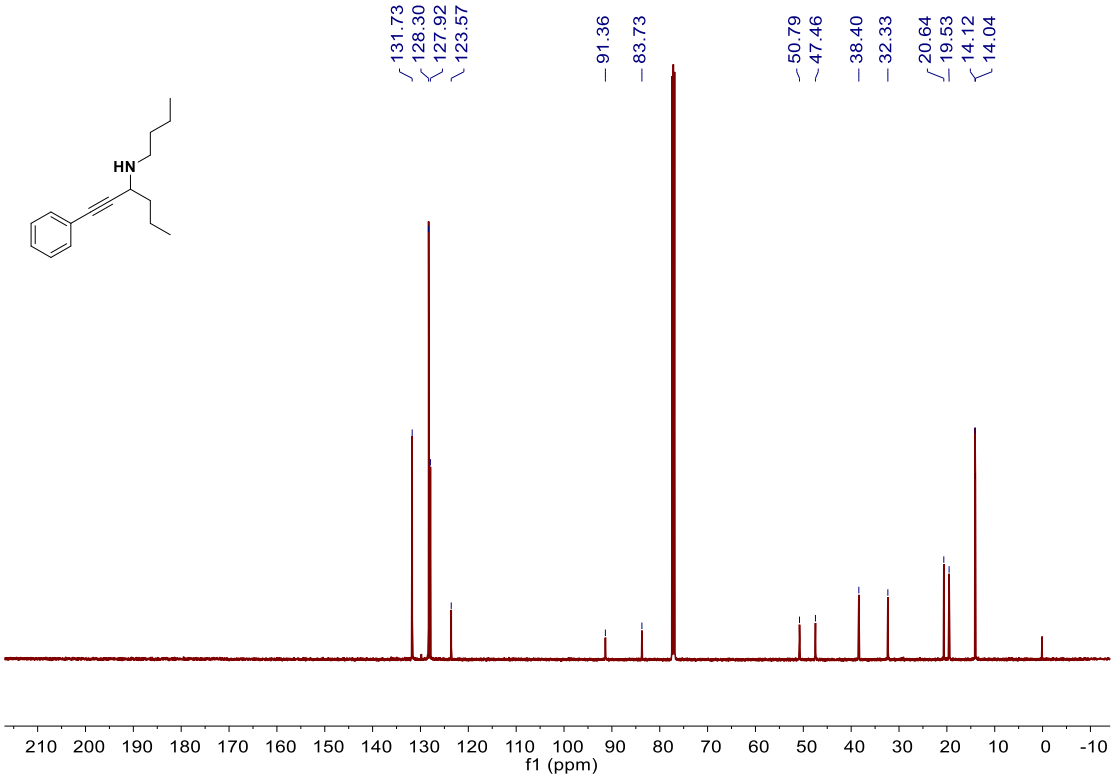
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-butyl-1,4-diphenylbut-3-yn-2-amine (1u)



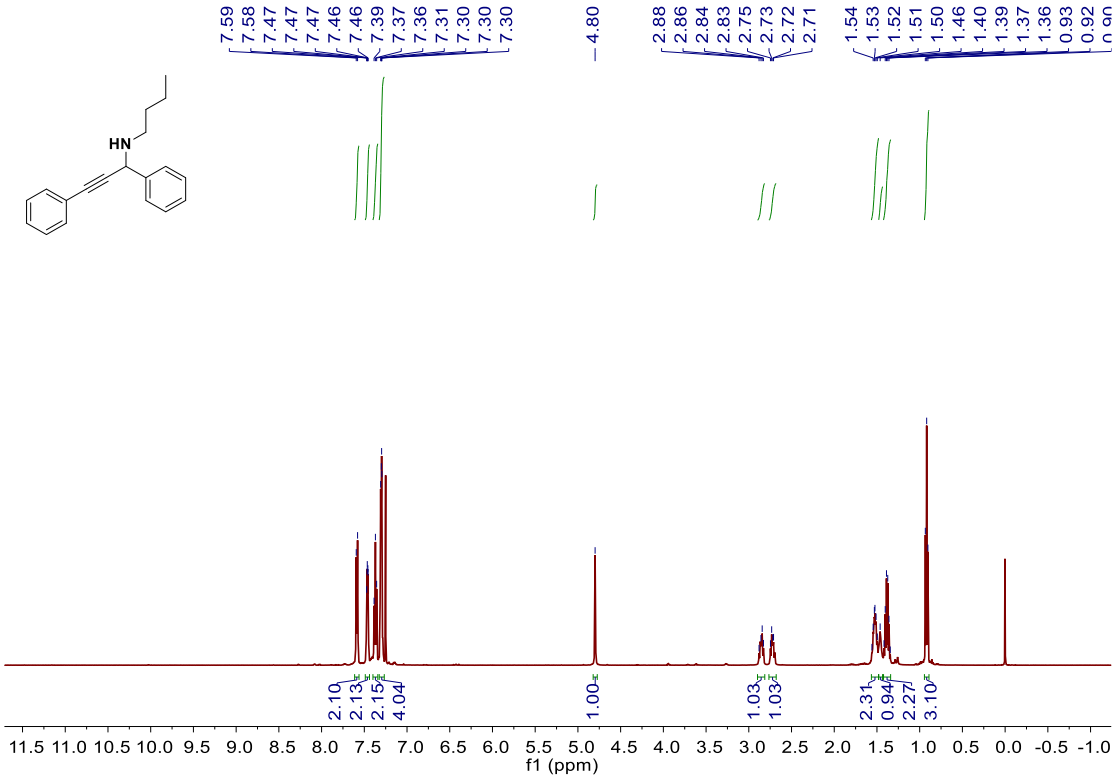
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-butyl-1-phenylhex-1-yn-3-amine (1v)



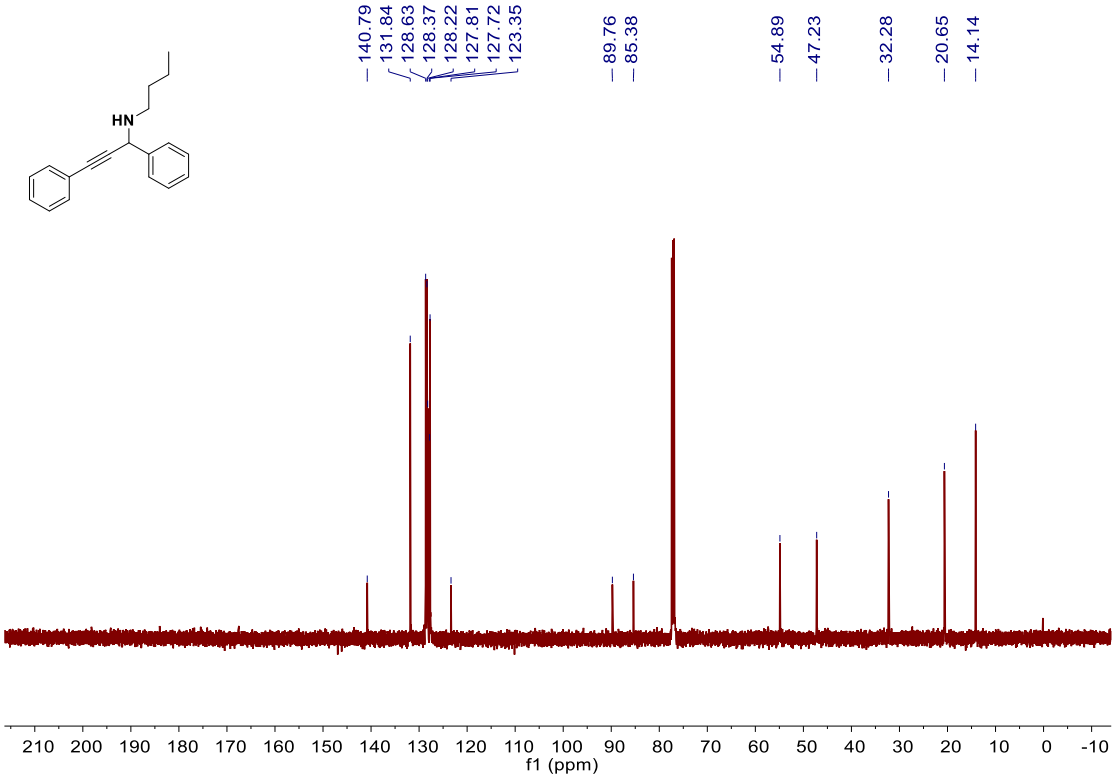
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-butyl-1-phenylhex-1-yn-3-amine (1v)



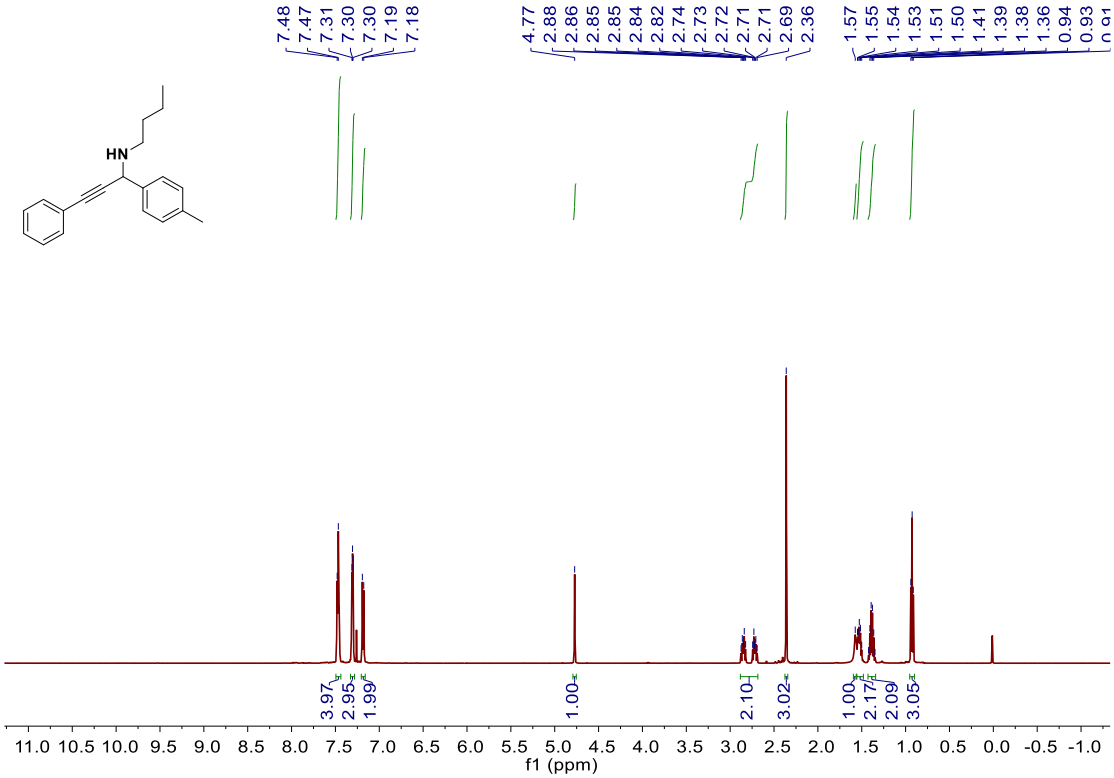
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-(1,3-diphenylprop-2-yn-1-yl)butan-1-amine (1w)



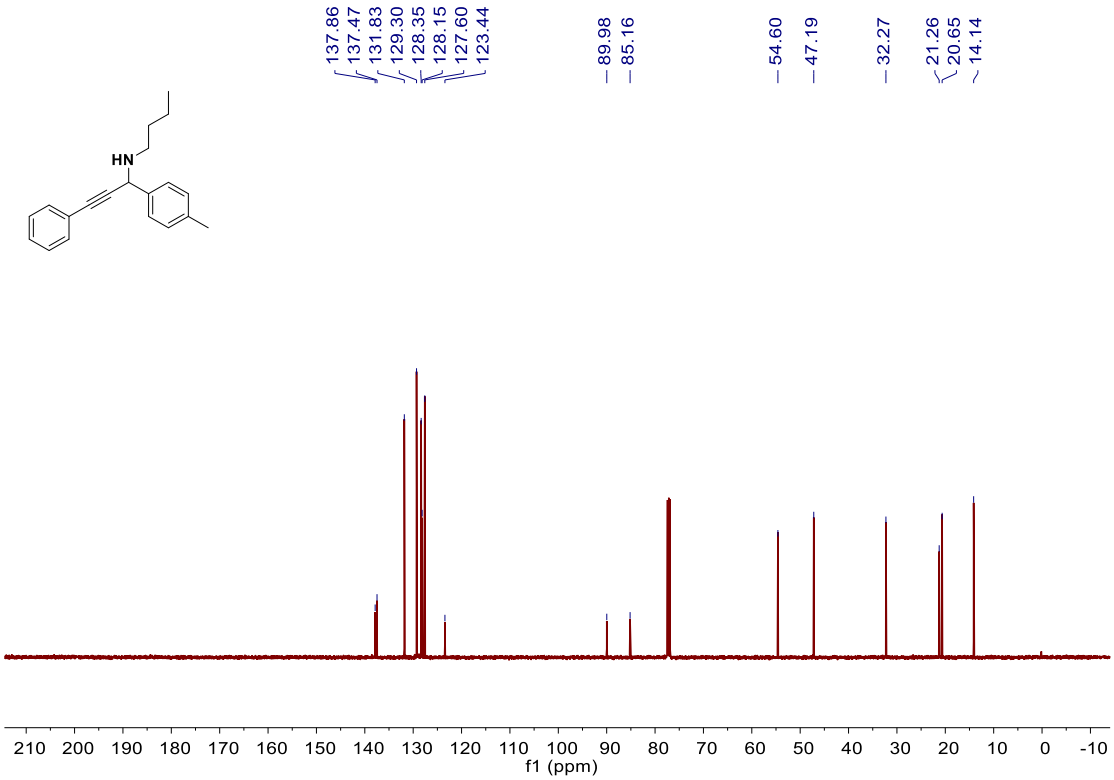
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-(1,3-diphenylprop-2-yn-1-yl)butan-1-amine (1w)



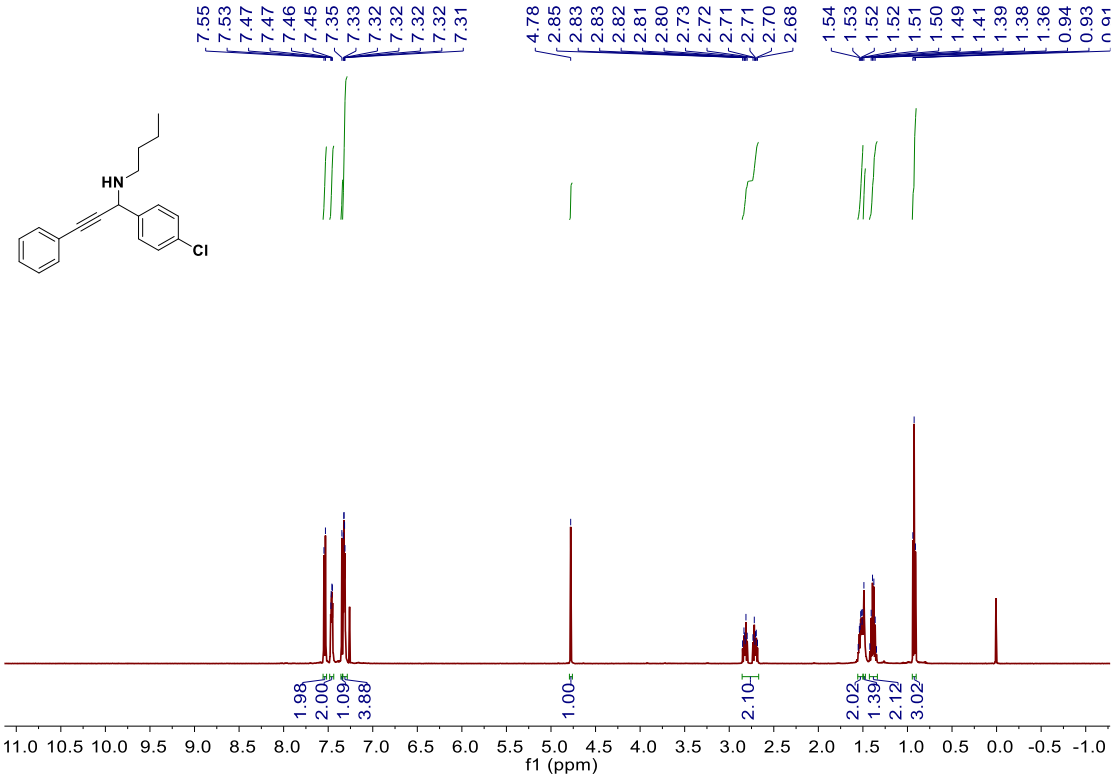
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-(3-phenyl-1-(p-tolyl)prop-2-yn-1-yl)butan-1-amine (1x)



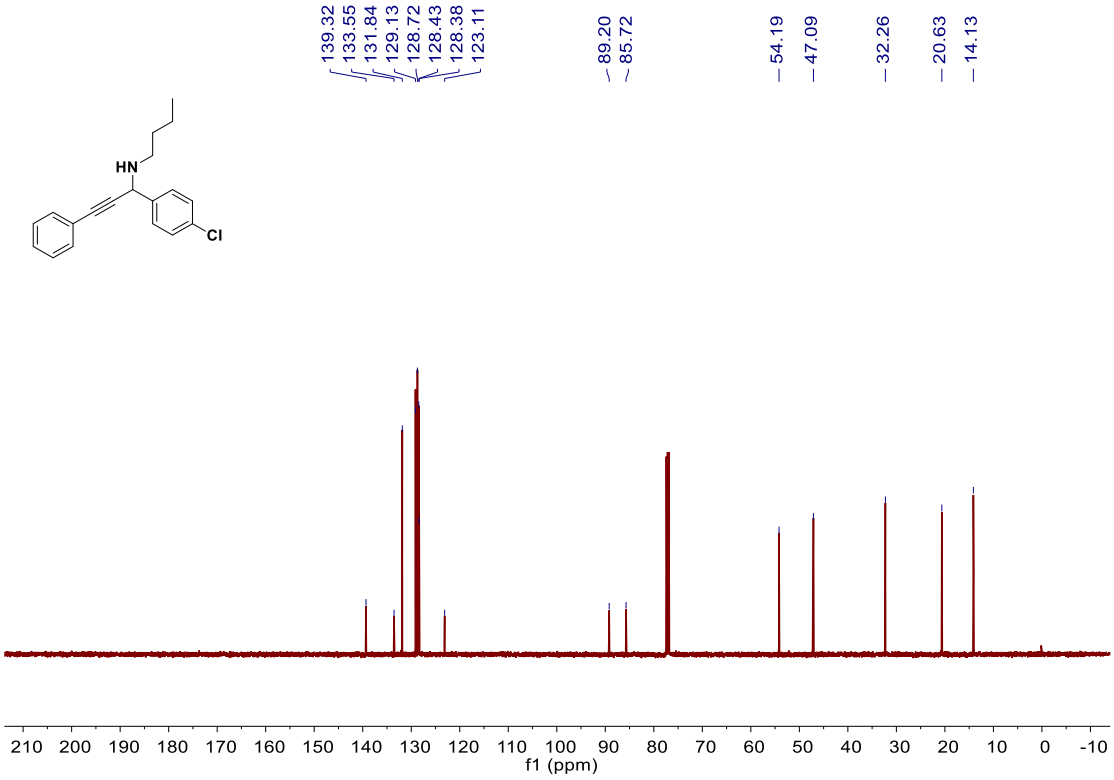
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-(3-phenyl-1-(p-tolyl)prop-2-yn-1-yl)butan-1-amine (1x)



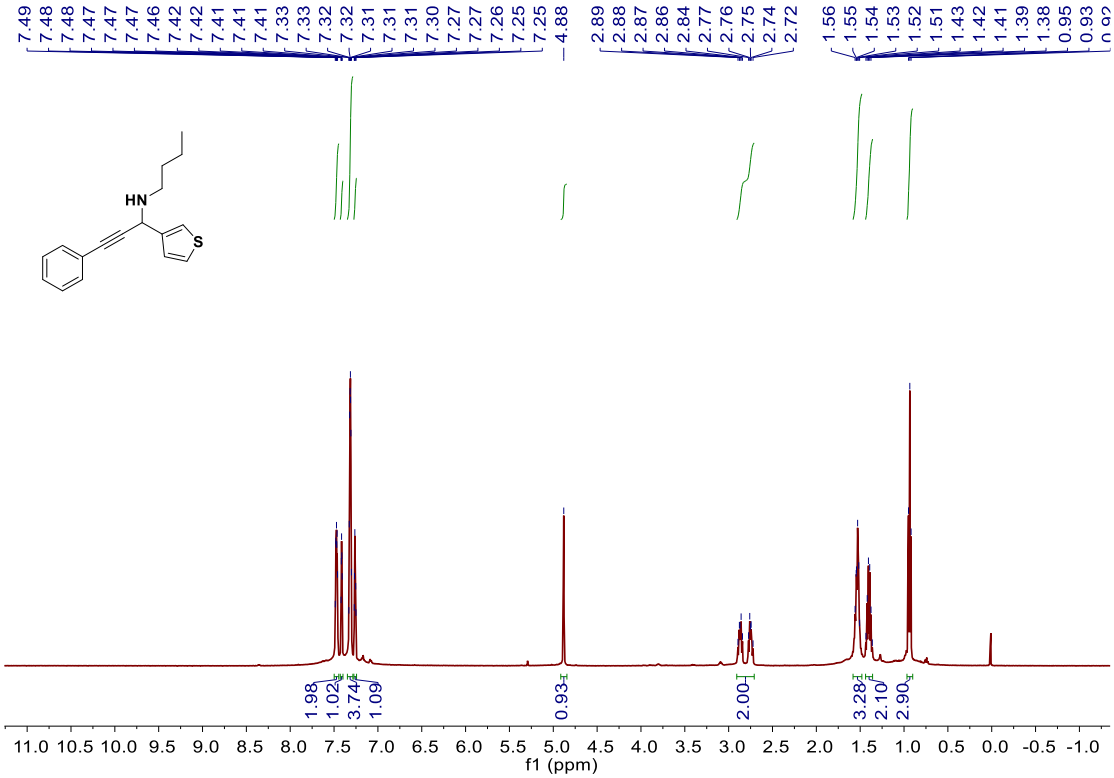
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-(1-(4-chlorophenyl)-3-phenylprop-2-yn-1-yl)butan-1-amine (1y)



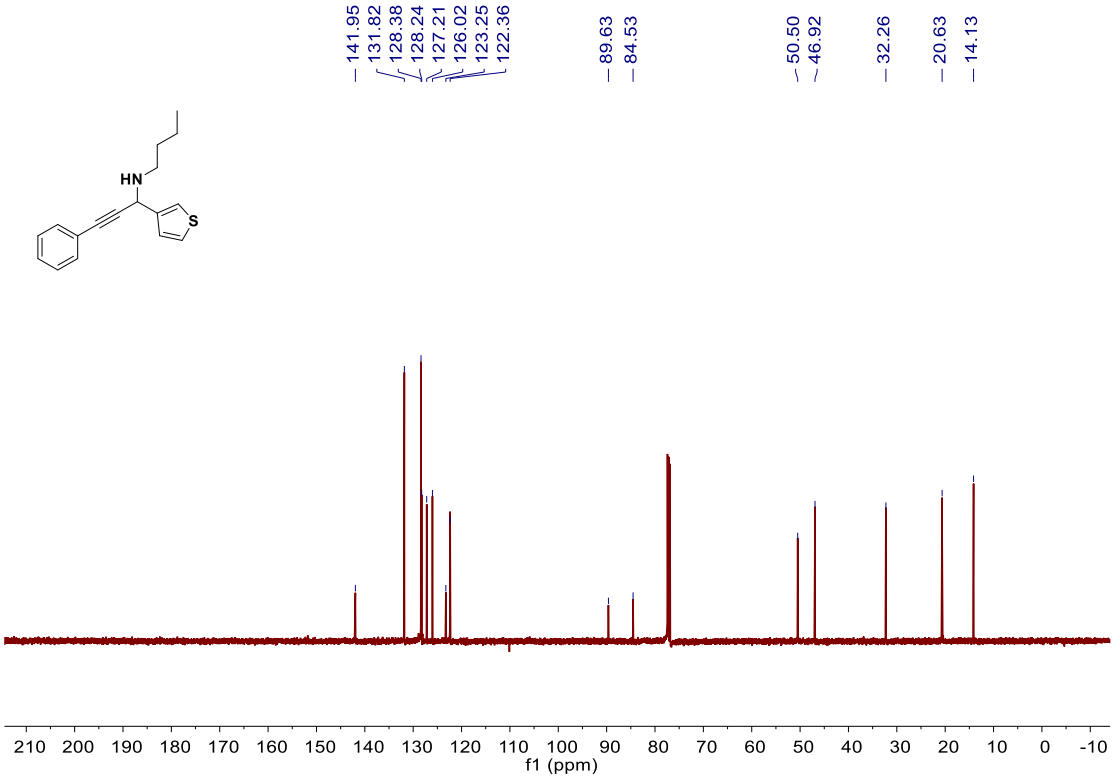
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-(1-(4-chlorophenyl)-3-phenylprop-2-yn-1-yl)butan-1-amine (1y)



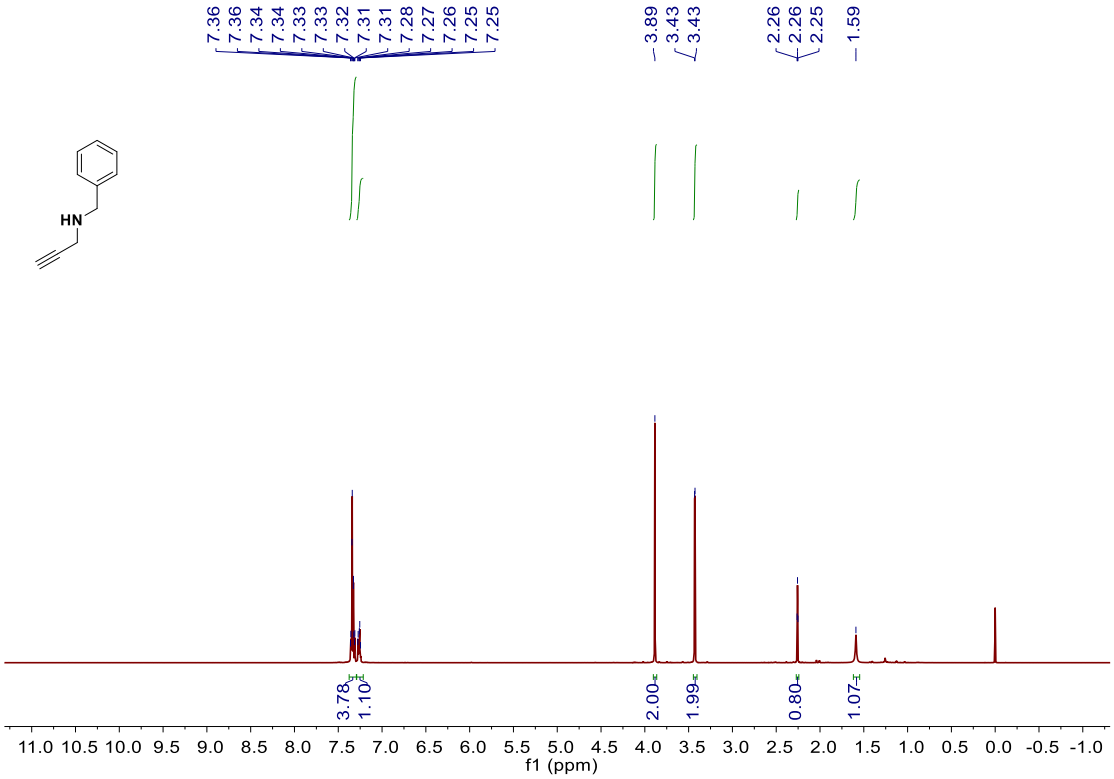
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-(3-phenyl-1-(thiophen-3-yl)prop-2-yn-1-yl)butan-1-amine (1z)



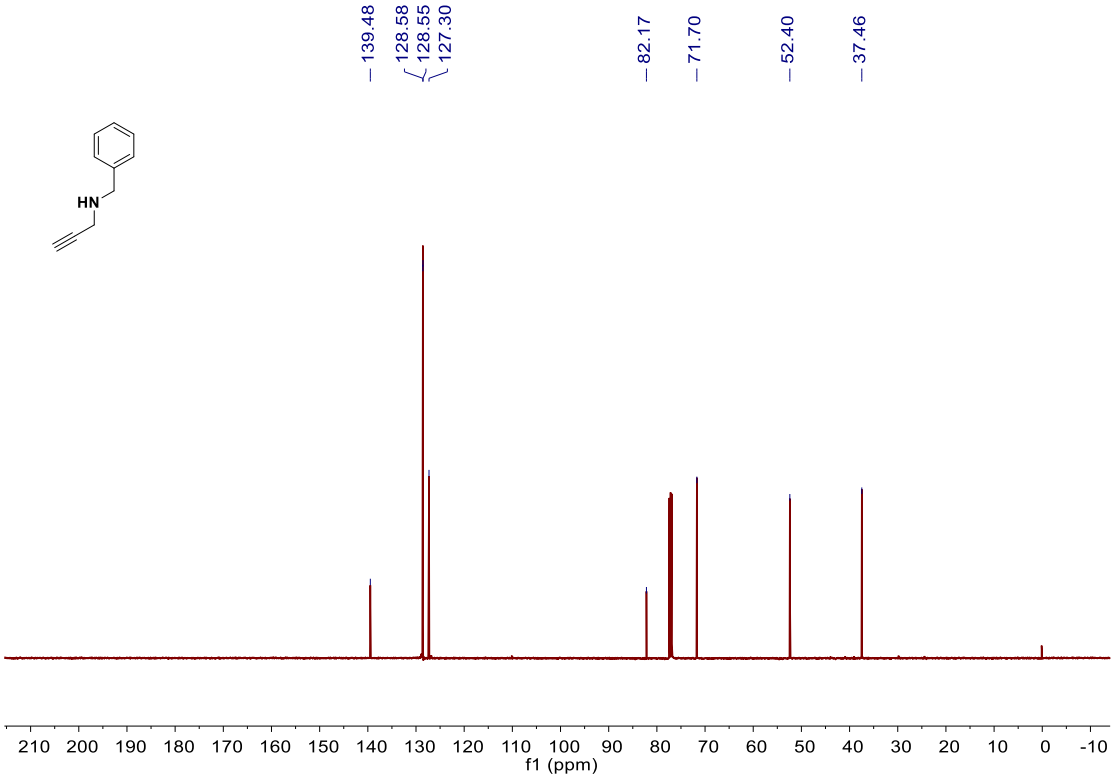
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-(3-phenyl-1-(thiophen-3-yl)prop-2-yn-1-yl)butan-1-amine (1z)



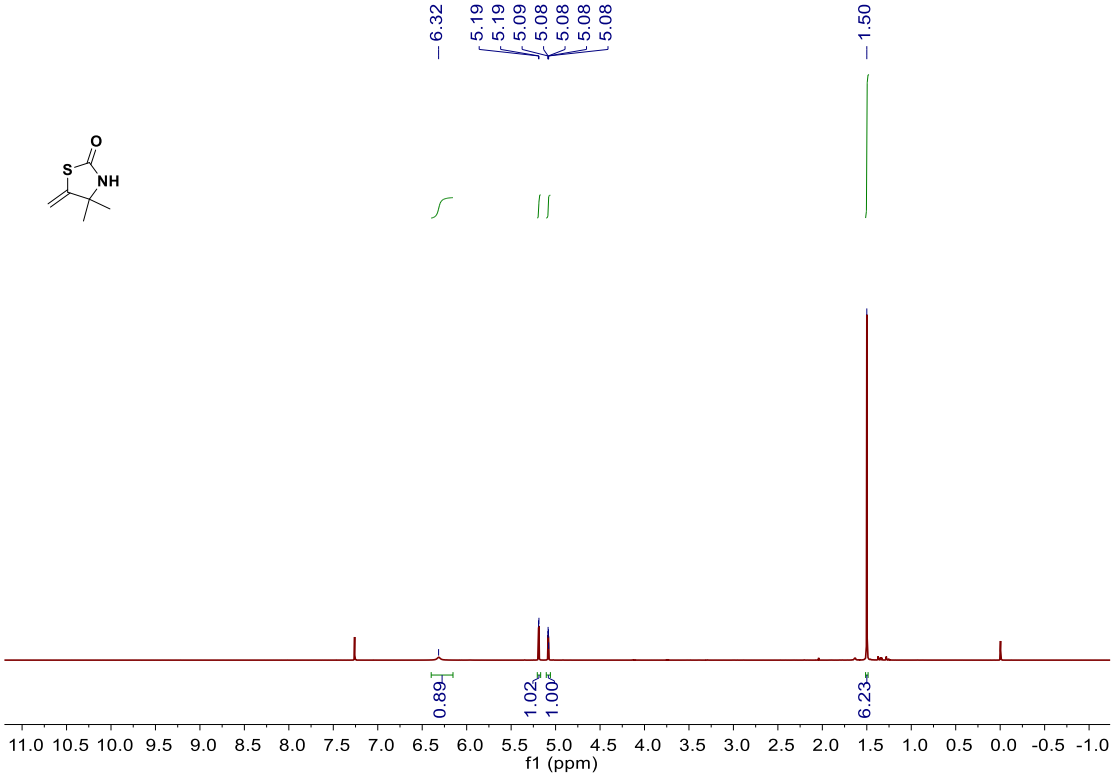
^{1}H NMR ($CDCl_3$, 500 MHz) of *N*-benzylprop-2-yn-1-amine (1ab)



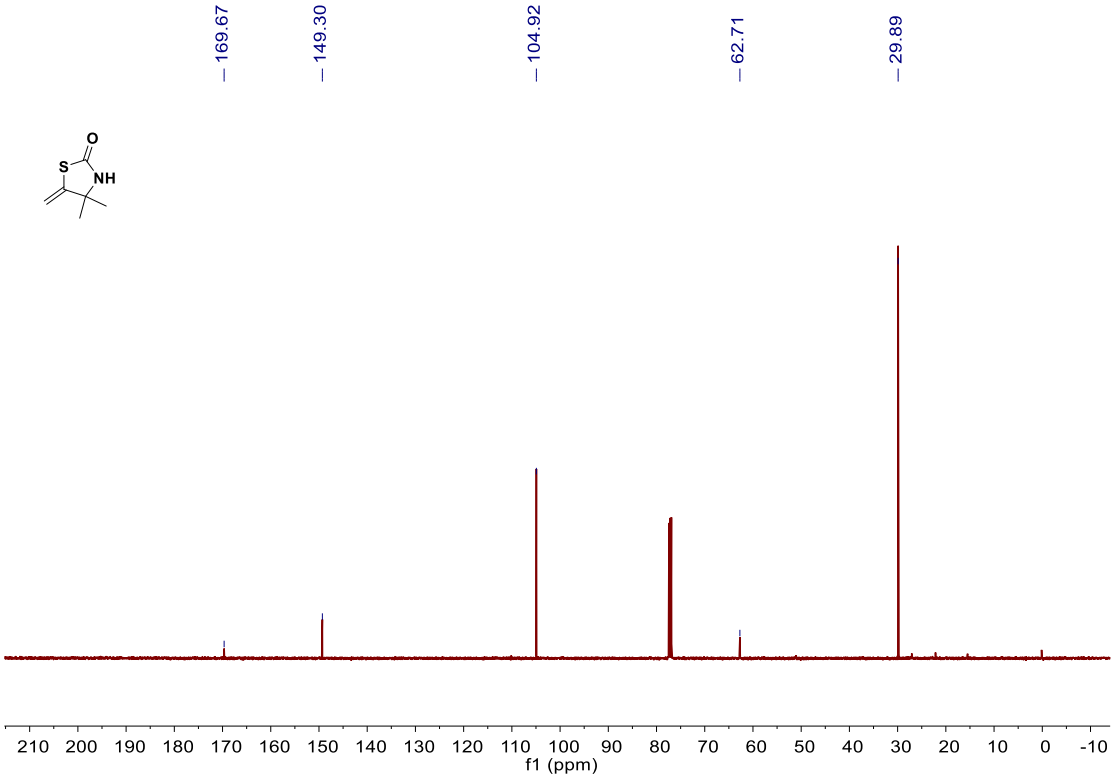
^{13}C NMR ($CDCl_3$, 125 MHz) of *N*-benzylprop-2-yn-1-amine (1ab)



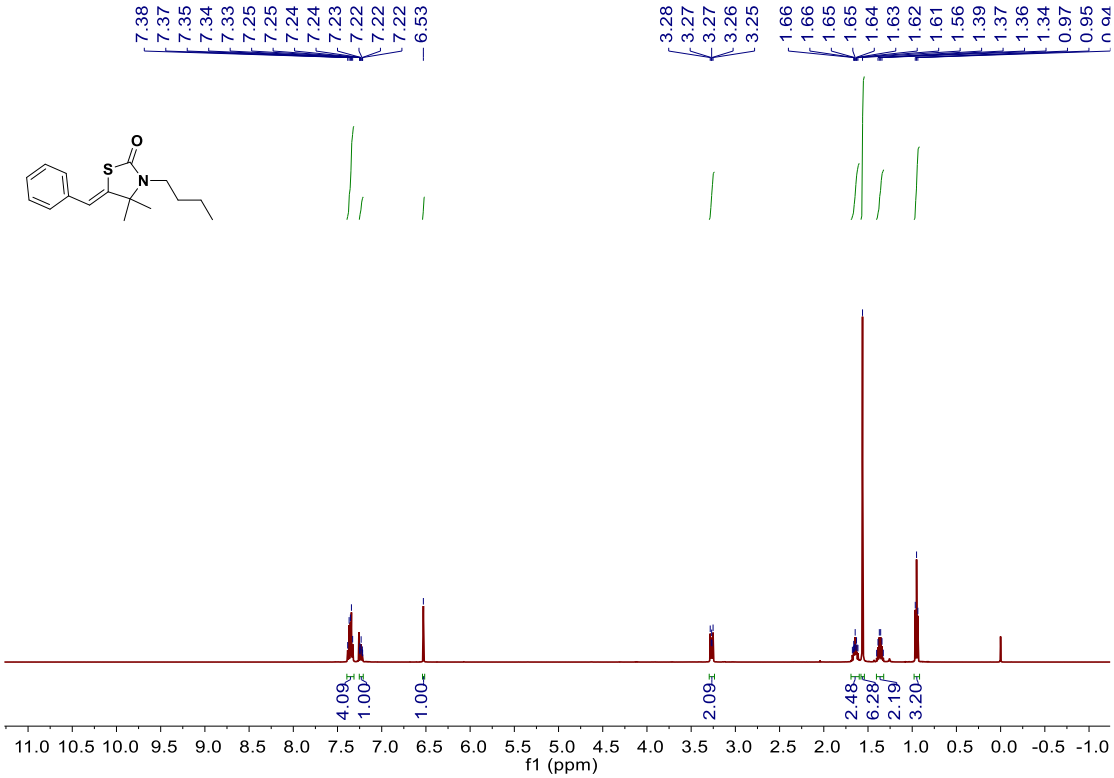
^{1}H NMR ($CDCl_3$, 500 MHz) of 4,4-dimethyl-5-methylenethiazolidin-2-one (2a)



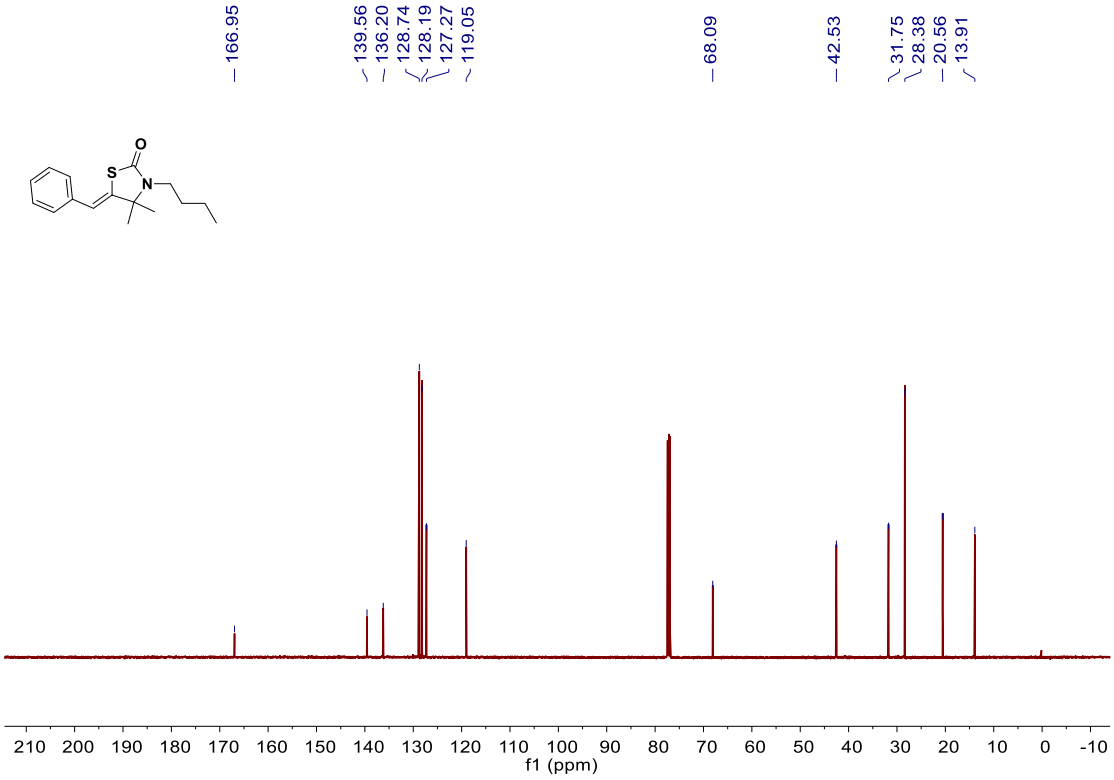
^{13}C NMR ($CDCl_3$, 125 MHz) of 4,4-dimethyl-5-methylenethiazolidin-2-one (2a)



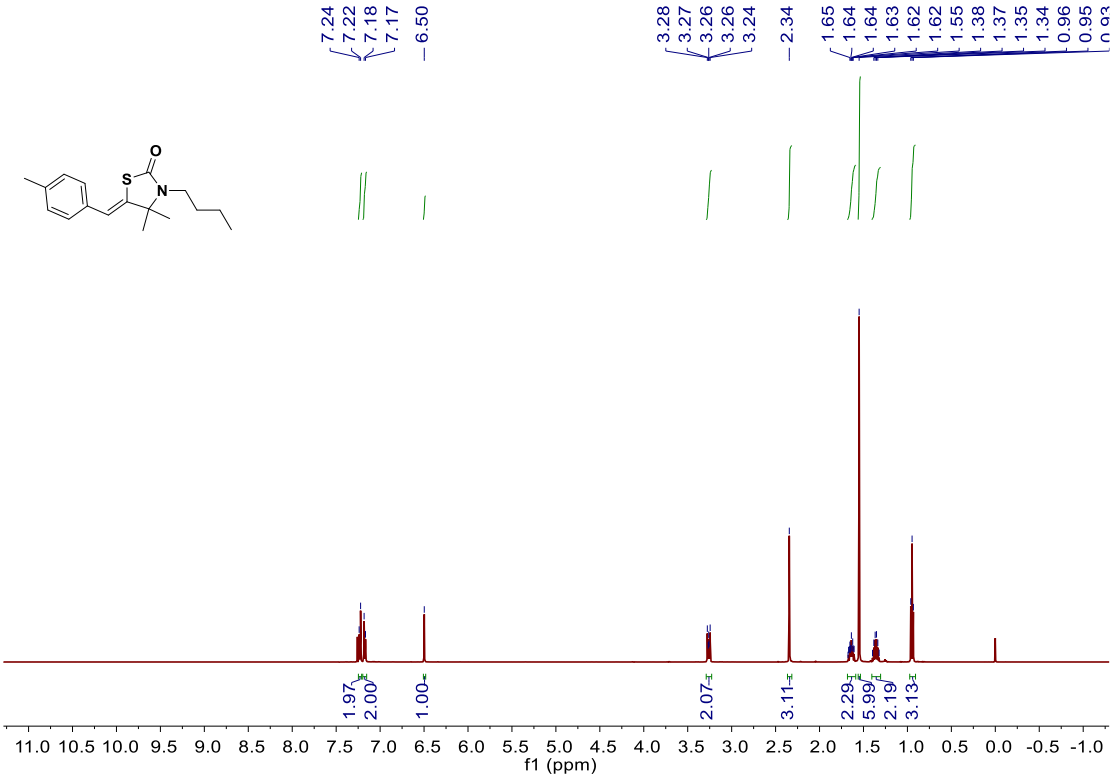
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-5-benzylidene-3-butyl-4,4-dimethylthiazolidin-2-one (2b)



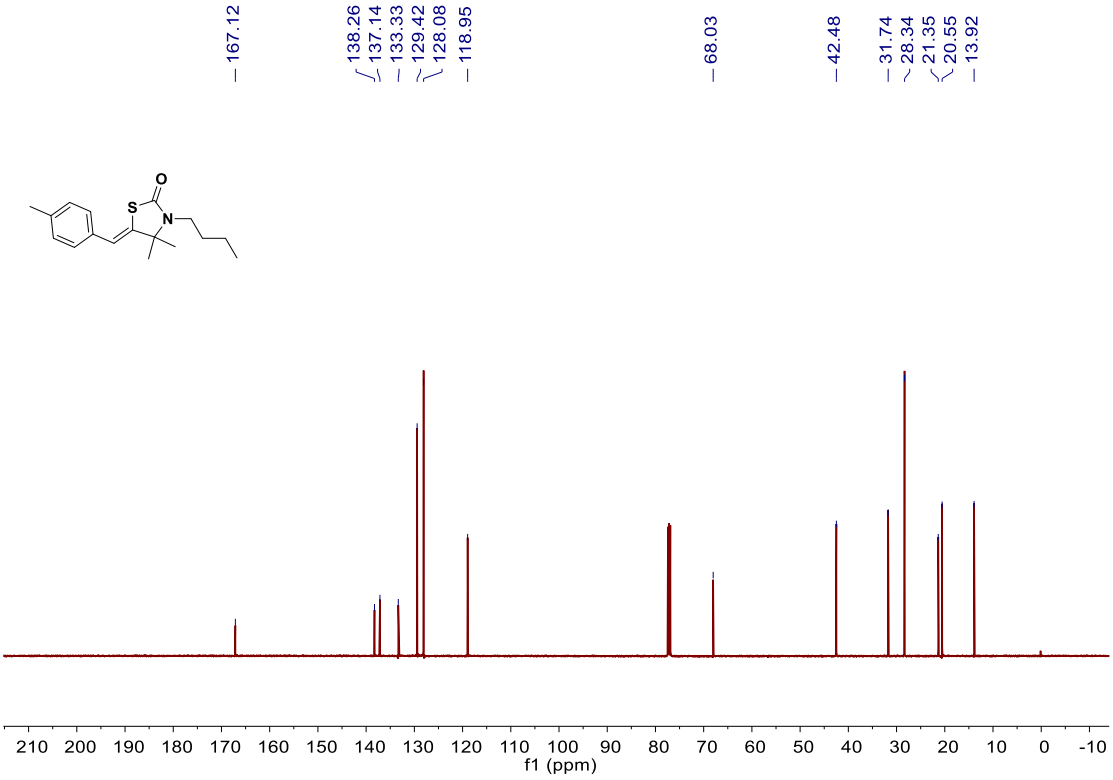
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-5-benzylidene-3-butyl-4,4-dimethylthiazolidin-2-one (2b)



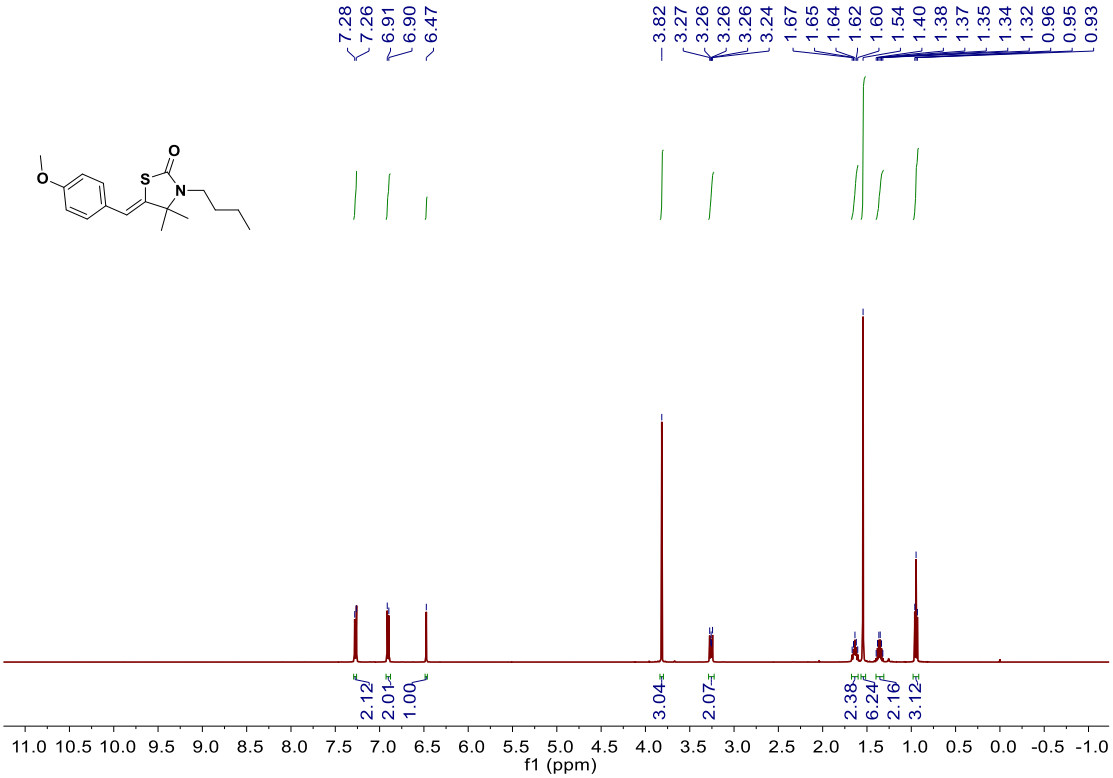
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-3-butyl-4,4-dimethyl-5-(4-methylbenzylidene)thiazolidin-2-one (2c)



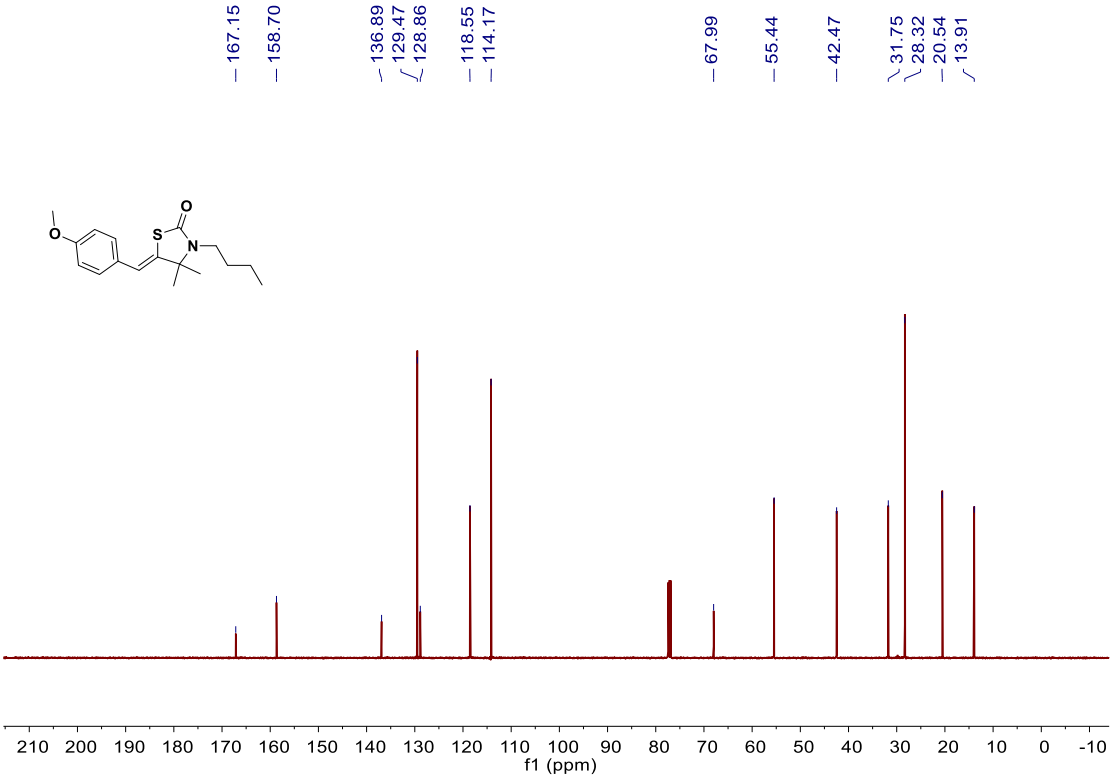
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-3-butyl-4,4-dimethyl-5-(4-methylbenzylidene)thiazolidin-2-one (2c)



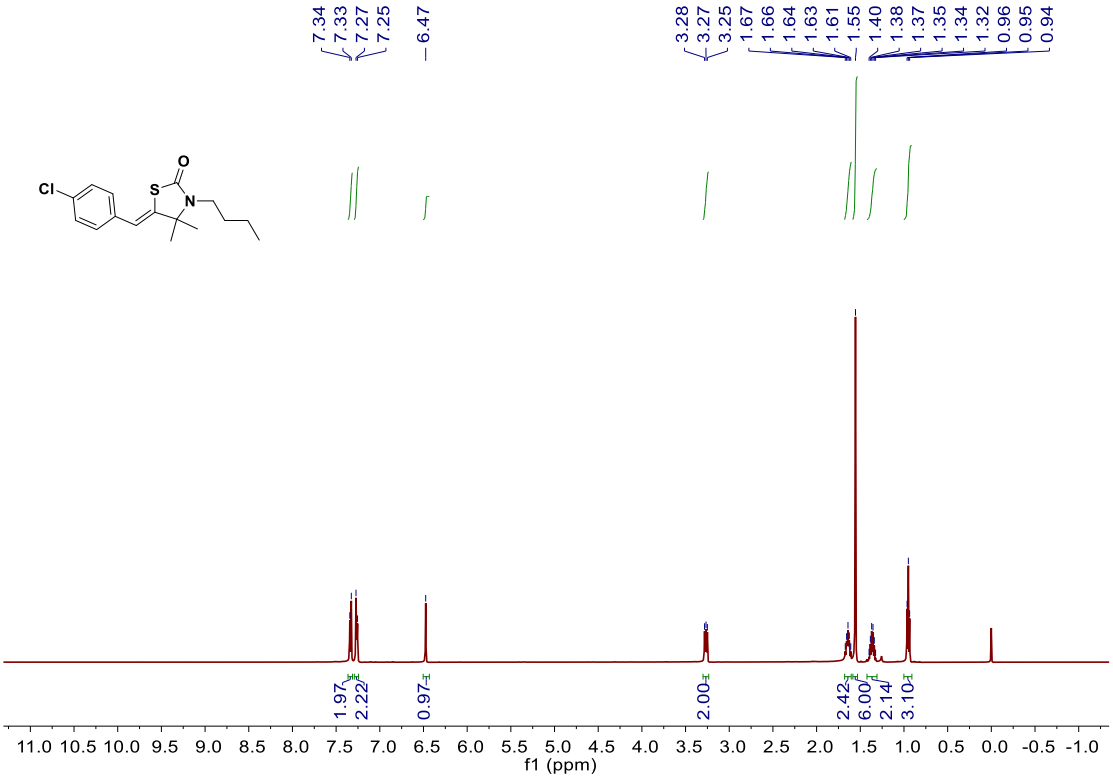
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-3-butyl-5-(4-methoxybenzylidene)-4,4-dimethylthiazolidin-2-one (2d)



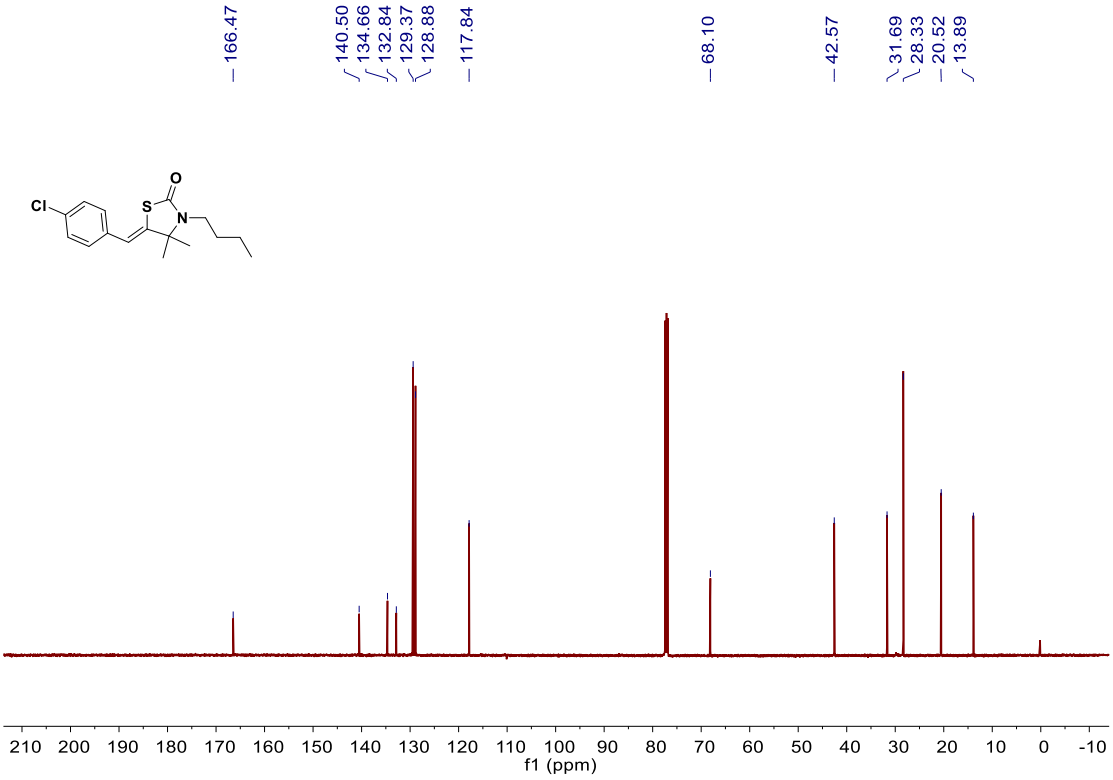
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-3-butyl-5-(4-methoxybenzylidene)-4,4-dimethylthiazolidin-2-one (2d)



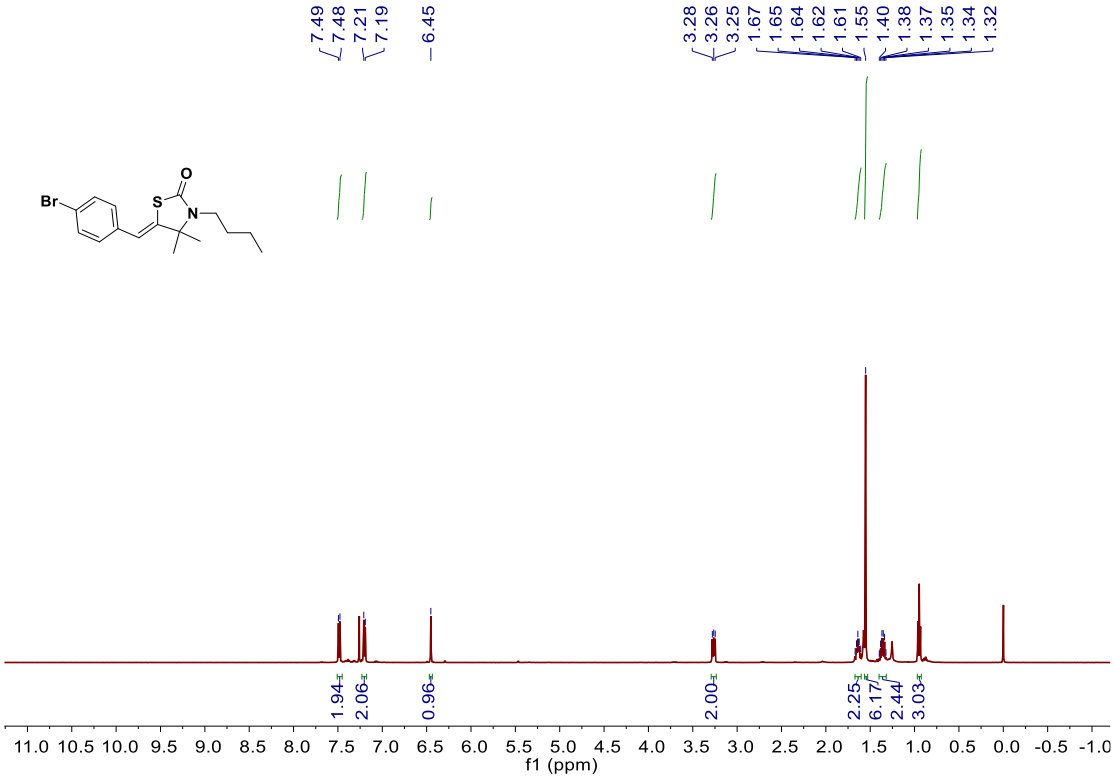
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-3-butyl-5-(4-chlorobenzylidene)-4,4-dimethylthiazolidin-2-one (2e)



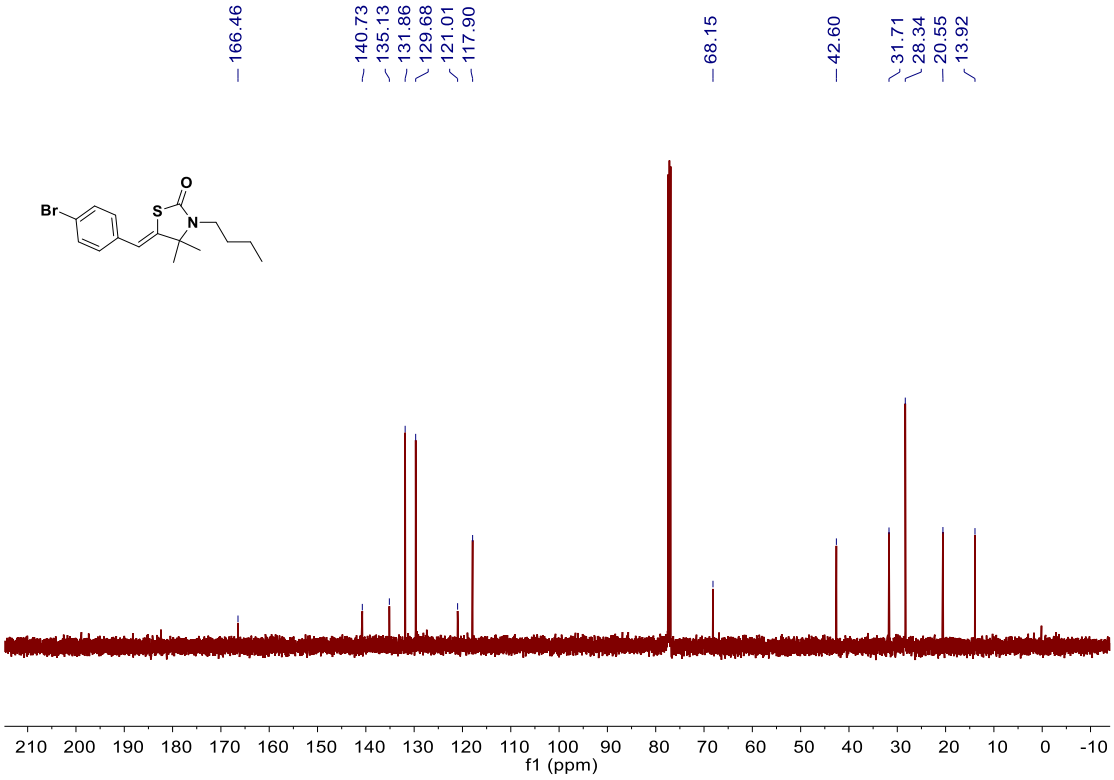
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-3-butyl-5-(4-chlorobenzylidene)-4,4-dimethylthiazolidin-2-one (2e)



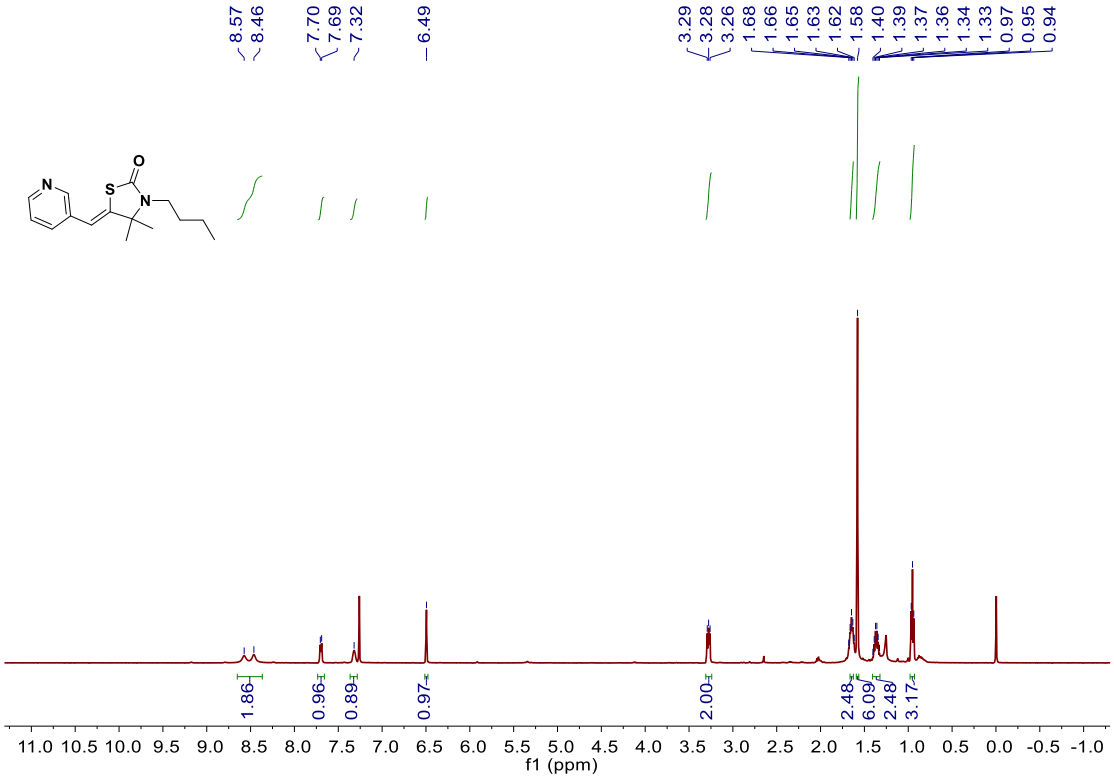
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-5-(4-bromobenzylidene)-3-butyl-4,4-dimethylthiazolidin-2-one (2f)



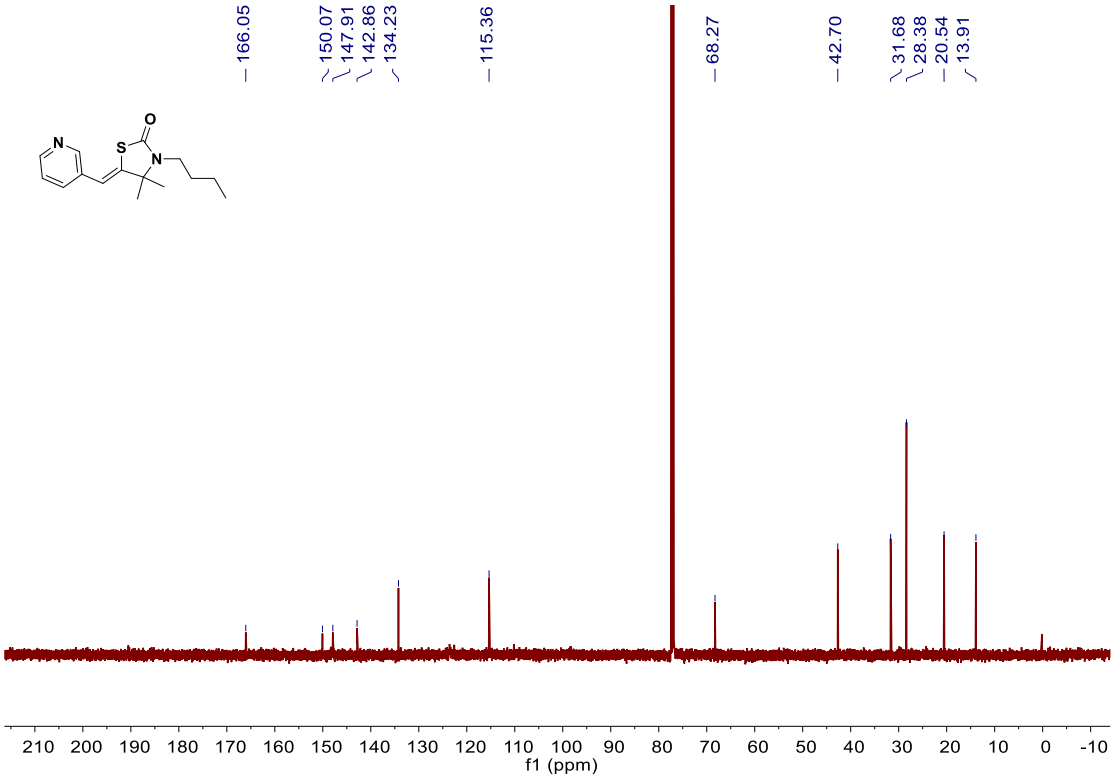
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-5-(4-bromobenzylidene)-3-butyl-4,4-dimethylthiazolidin-2-one (2f)



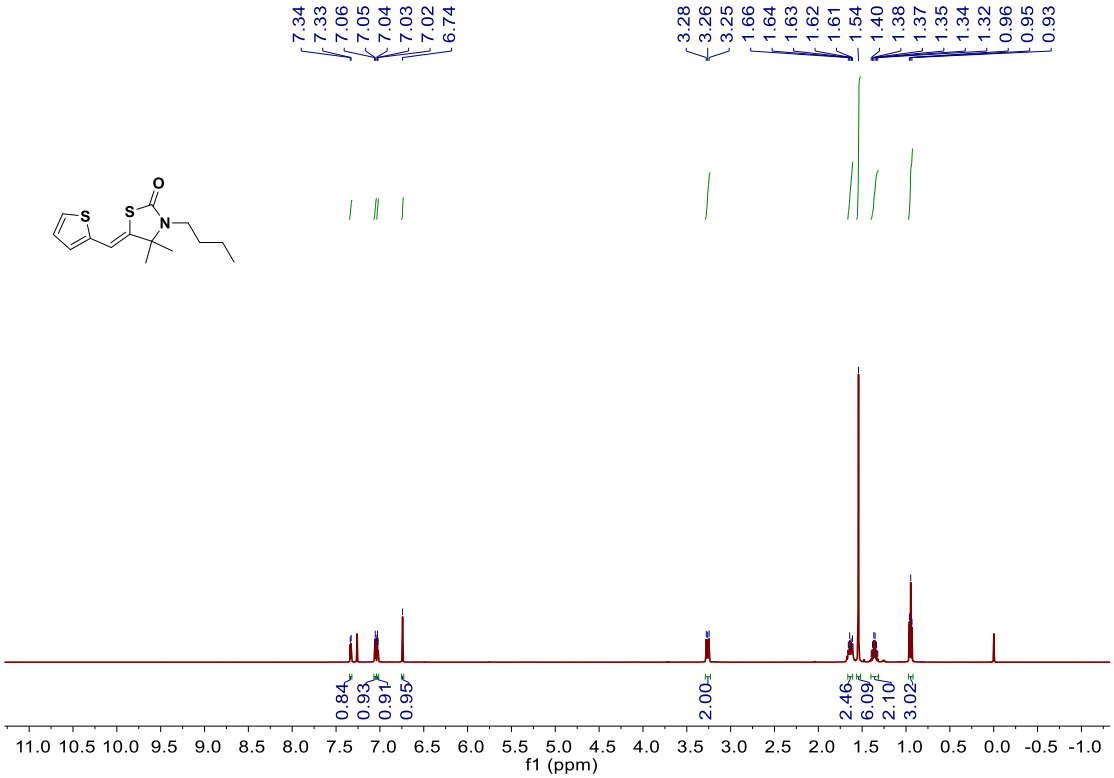
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-3-butyl-4,4-dimethyl-5-(pyridin-3-ylmethylene)thiazolidin-2-one (2g)



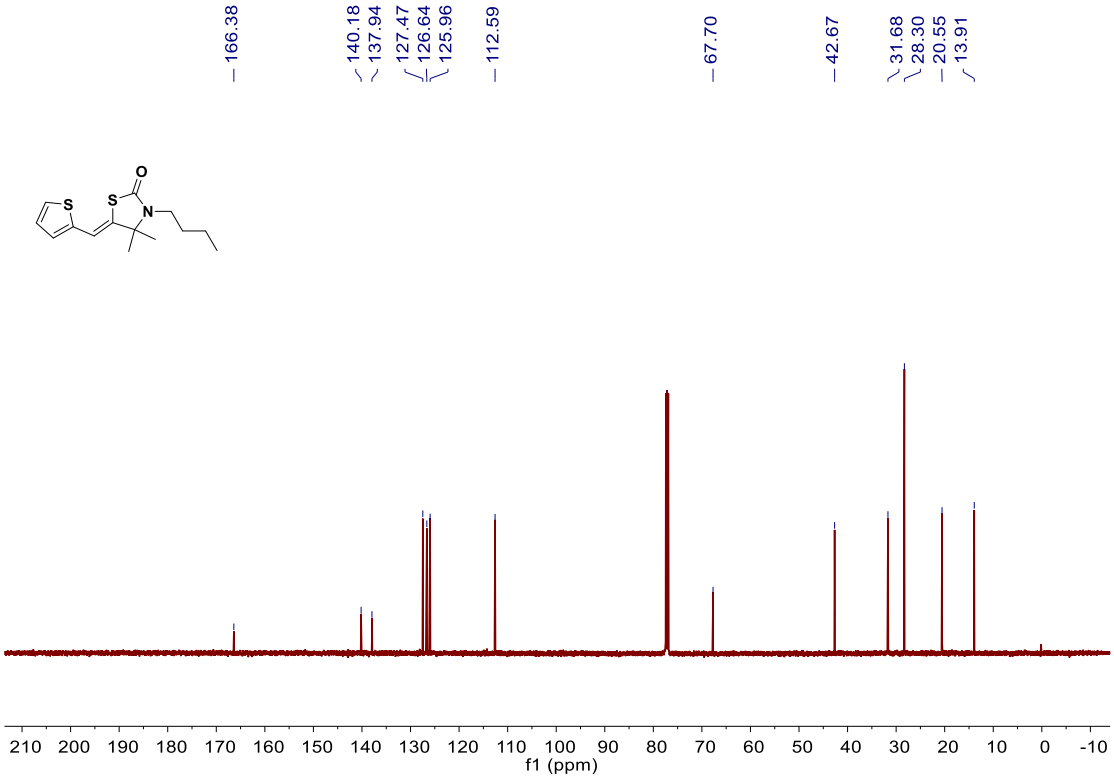
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-3-butyl-4,4-dimethyl-5-(pyridin-3-ylmethylene)thiazolidin-2-one (2g)



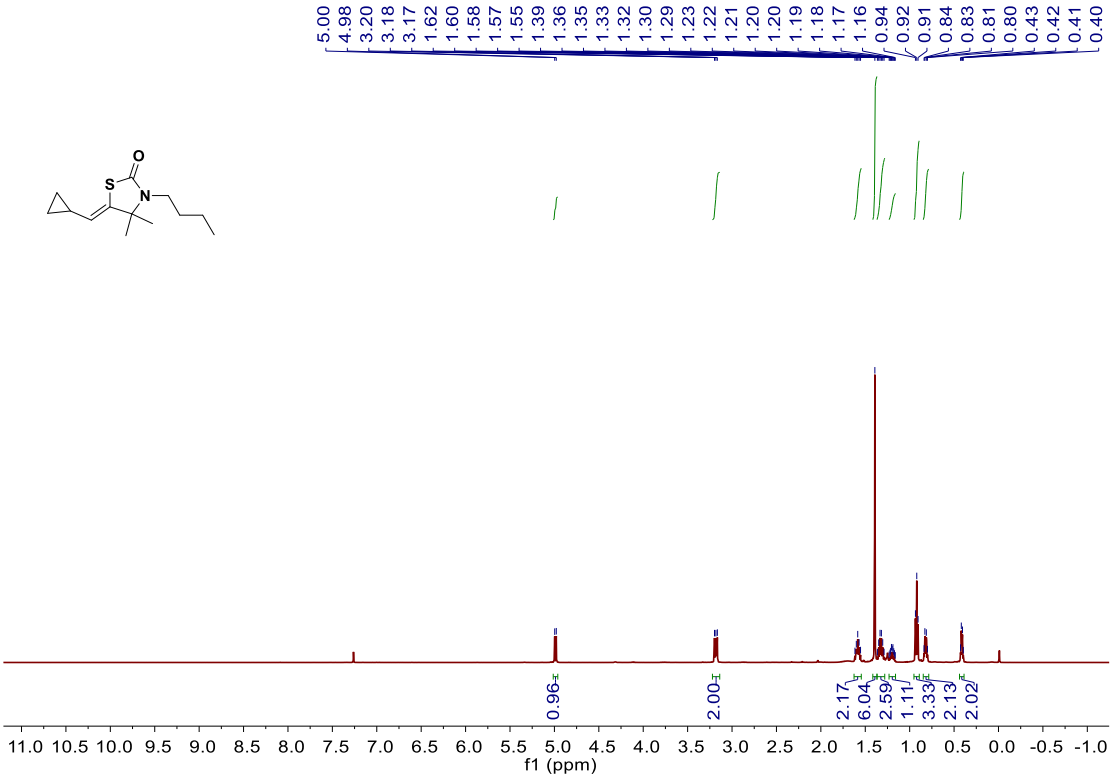
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-3-butyl-4,4-dimethyl-5-(thiophen-2-ylmethylene)thiazolidin-2-one (2h)



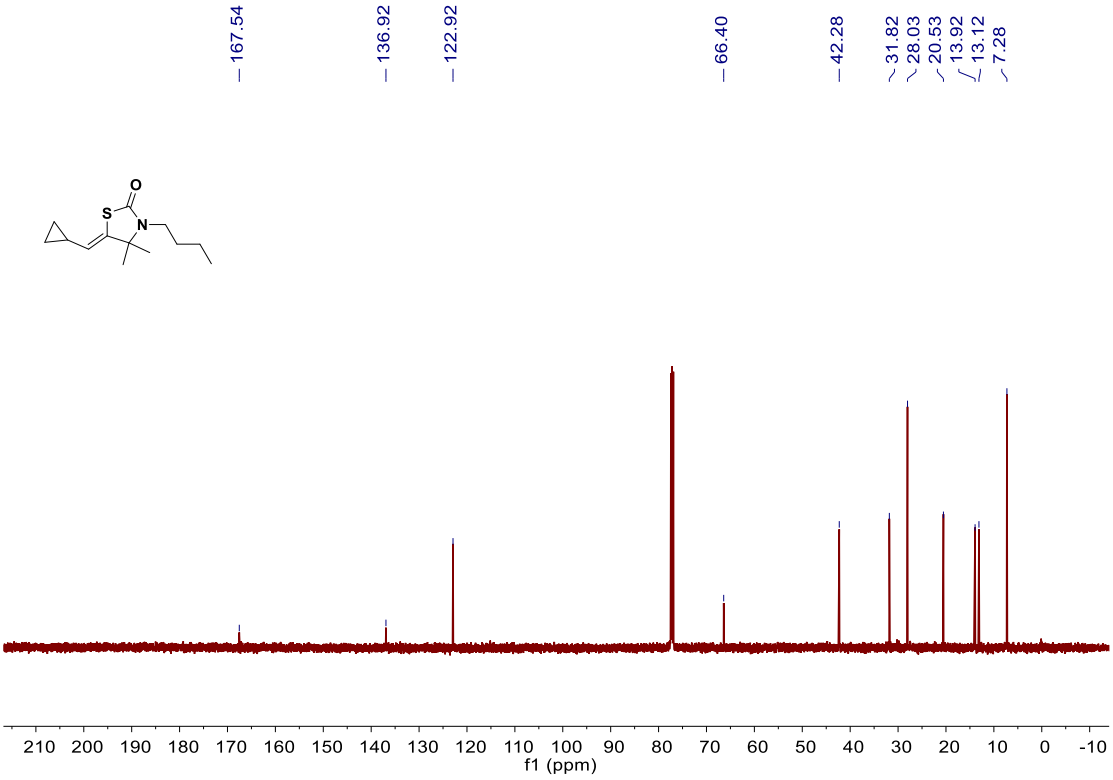
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-3-butyl-4,4-dimethyl-5-(thiophen-2-ylmethylene)thiazolidin-2-one (2h)



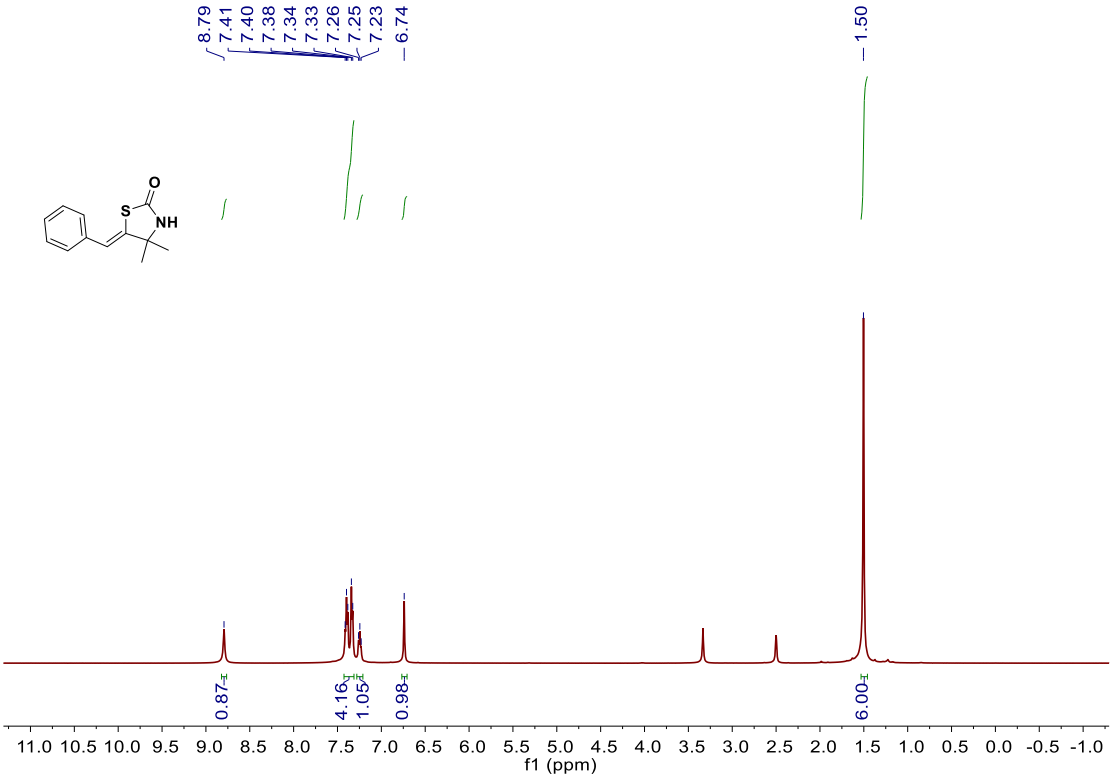
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-3-butyl-5-(cyclopropylmethylene)-4,4-dimethylthiazolidin-2-one (2i)



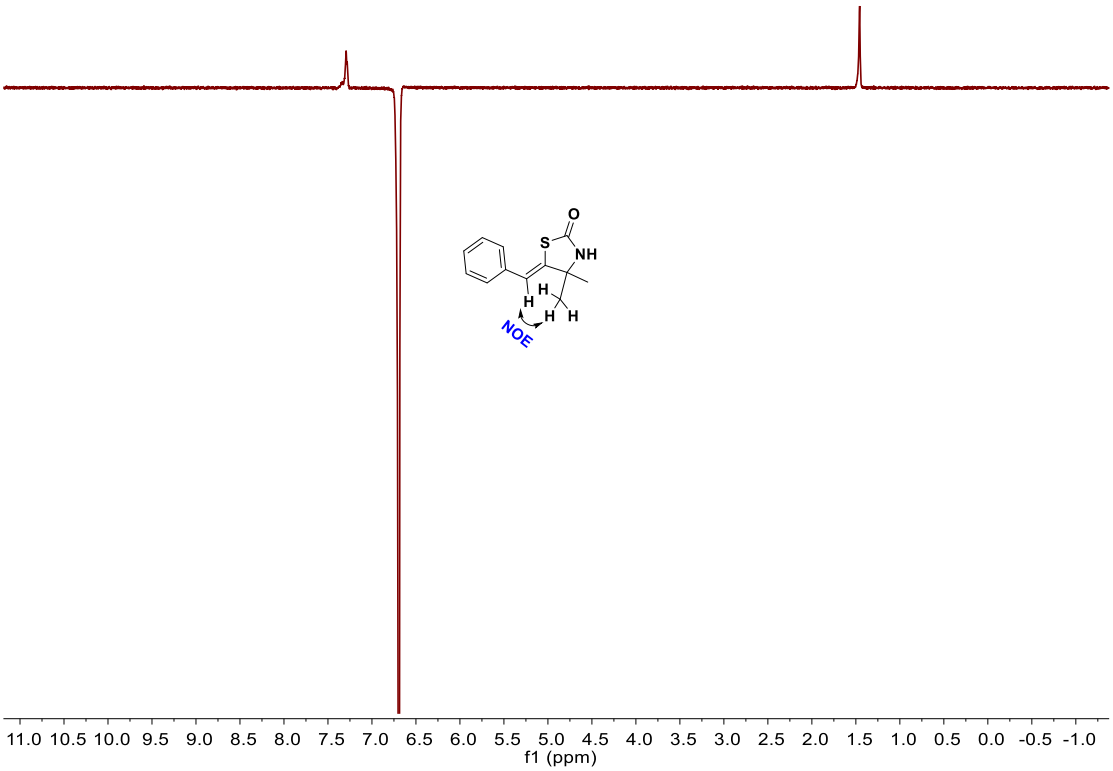
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-3-butyl-5-(cyclopropylmethylene)-4,4-dimethylthiazolidin-2-one (2i)



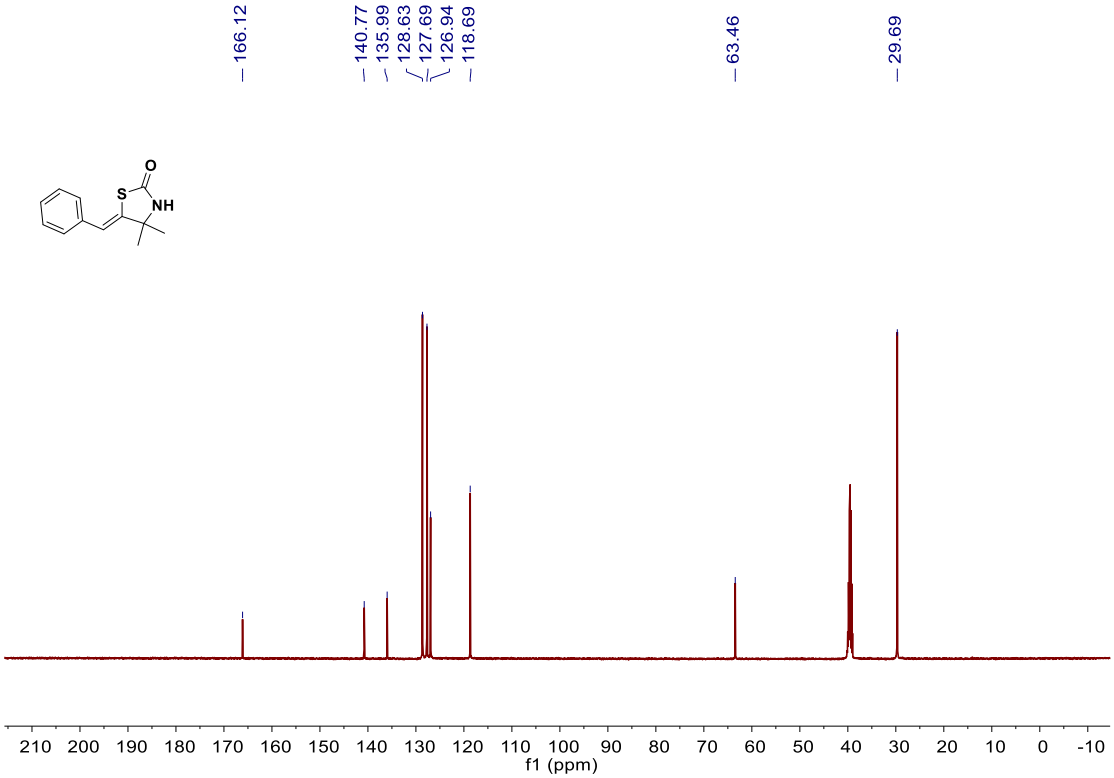
^{1}H NMR (DMSO-d_6, 500 MHz) of <u>(*Z*)-5-benzylidene-4,4-dimethylthiazolidin-2-one (2j)</u>



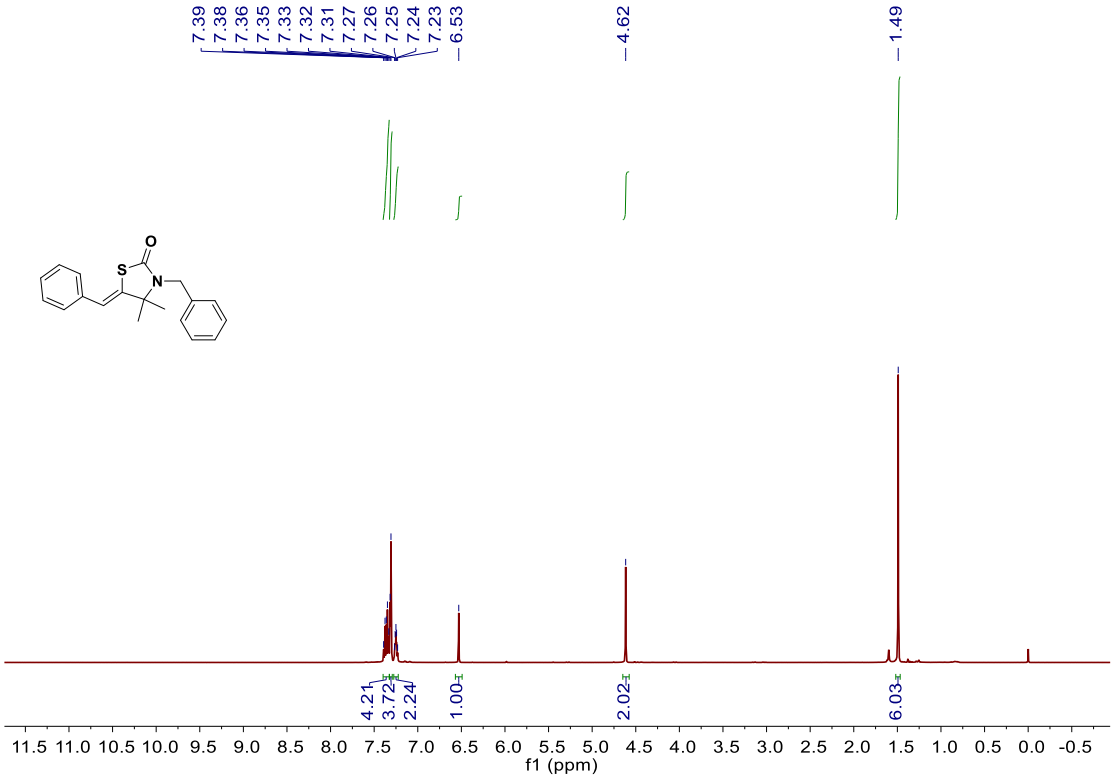
NOE spectrum of <u>(*Z*)-5-benzylidene-4,4-dimethylthiazolidin-2-one (2j)</u>



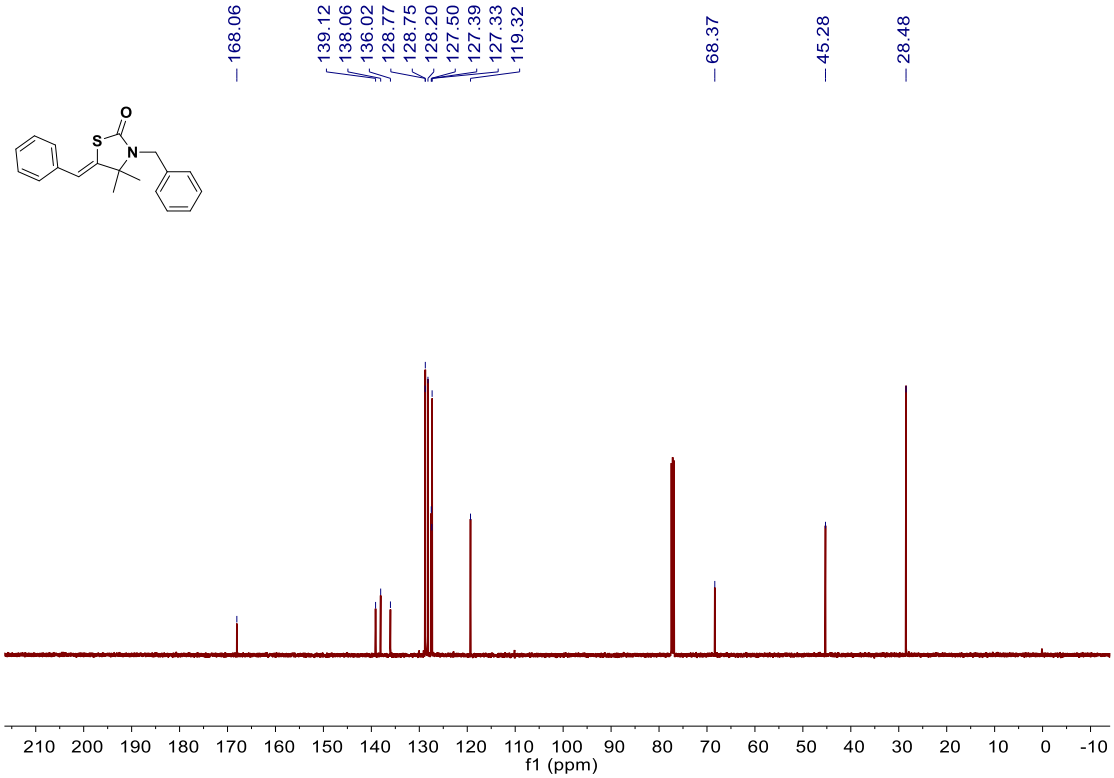
^{13}C NMR (DMSO-d_6, 125 MHz) of (*Z*)-5-benzylidene-4,4-dimethylthiazolidin-2-one (2j)



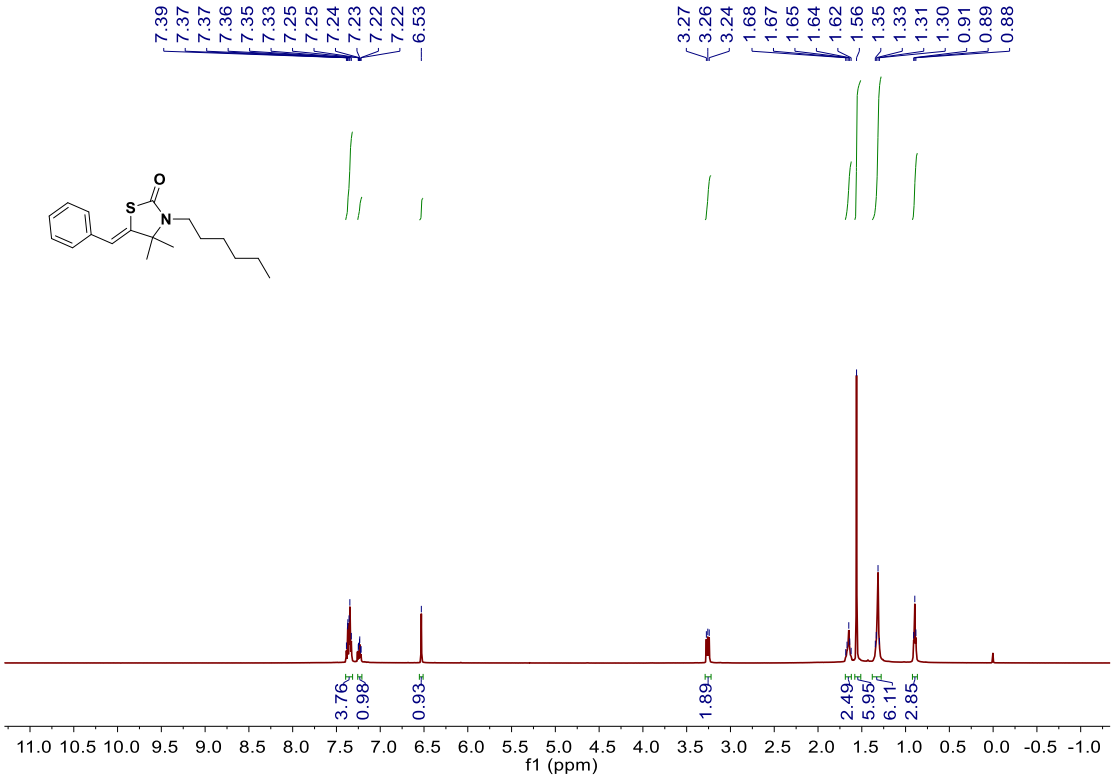
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-3-benzyl-5-benzylidene-4,4-dimethylthiazolidin-2-one (2k)



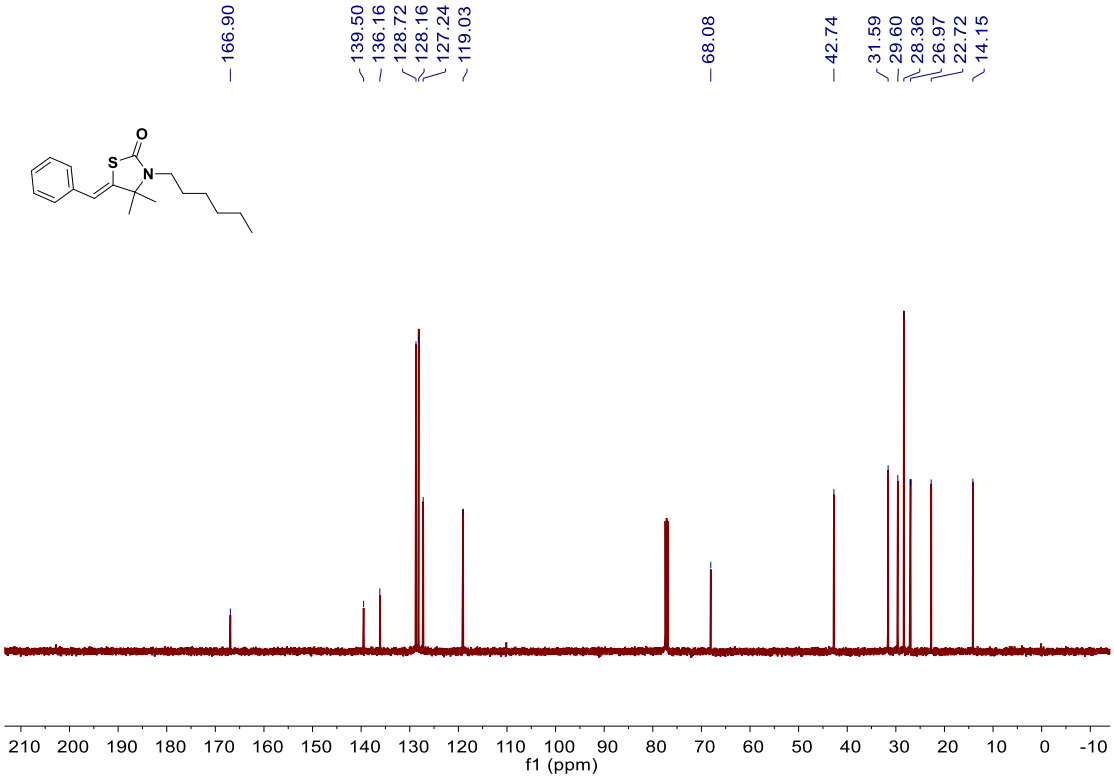
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-3-benzyl-5-benzylidene-4,4-dimethylthiazolidin-2-one (2k)



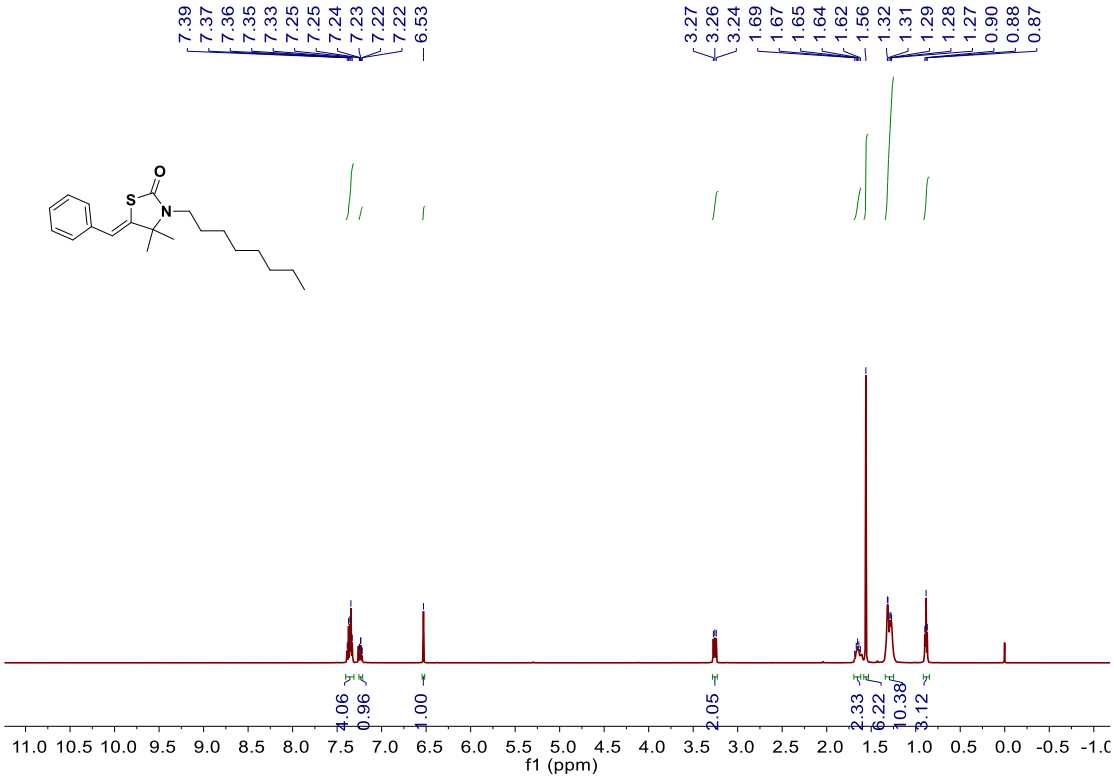
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-5-benzylidene-3-hexyl-4,4-dimethylthiazolidin-2-one (2l)



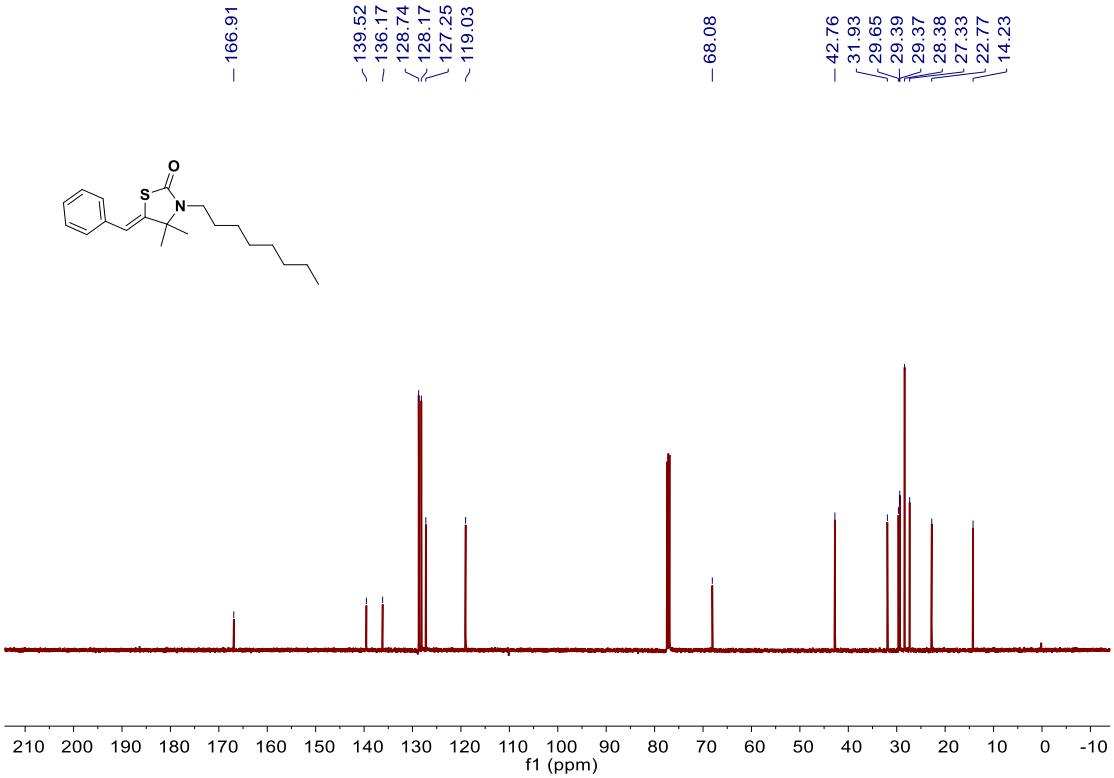
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-5-benzylidene-3-hexyl-4,4-dimethylthiazolidin-2-one (2l)



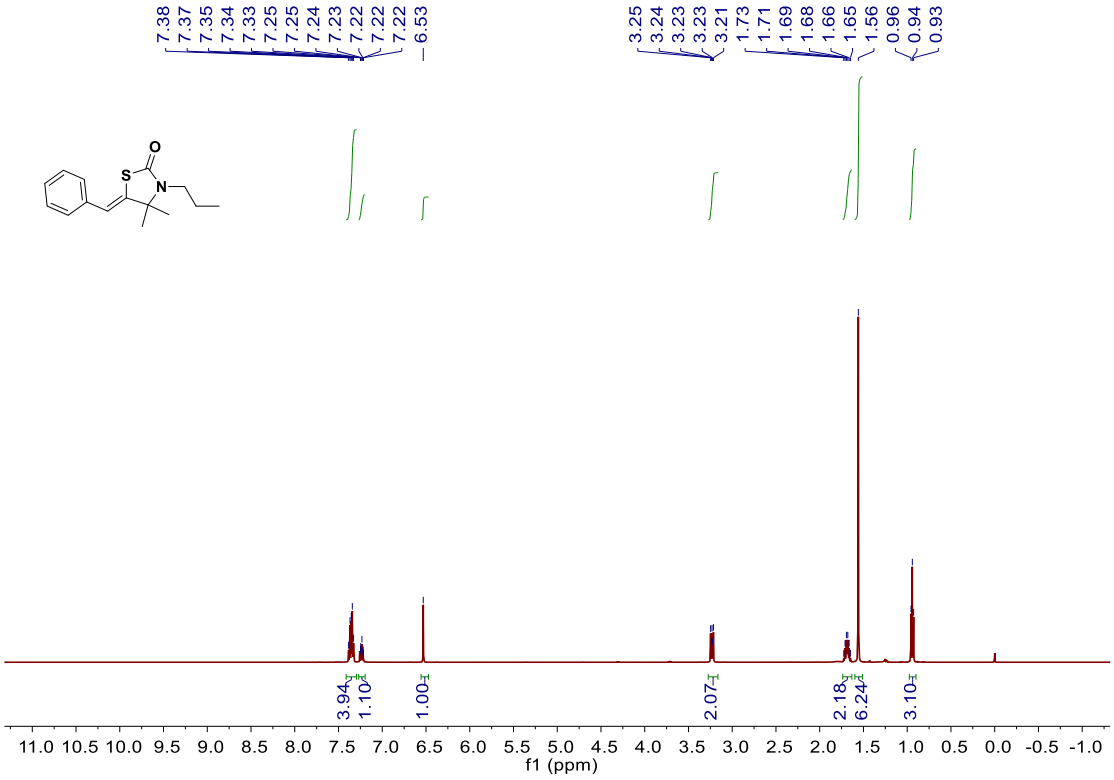
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-5-benzylidene-4,4-dimethyl-3-octylthiazolidin-2-one (2m)



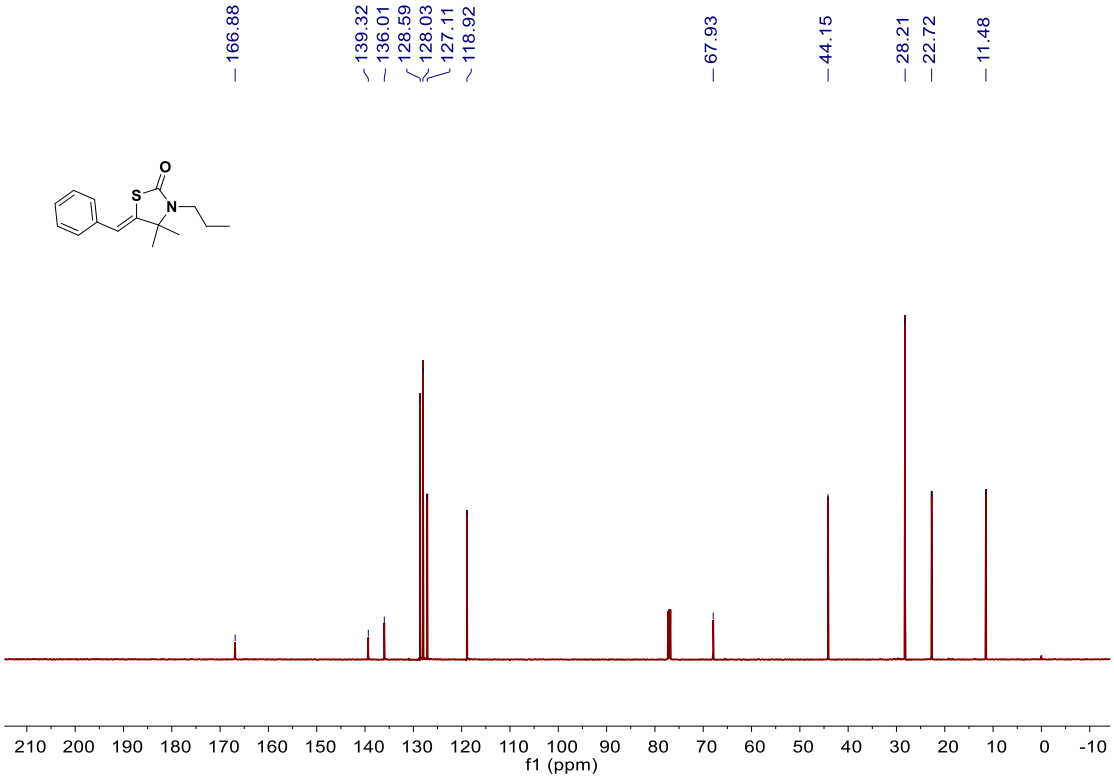
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-5-benzylidene-4,4-dimethyl-3-octylthiazolidin-2-one (2m)



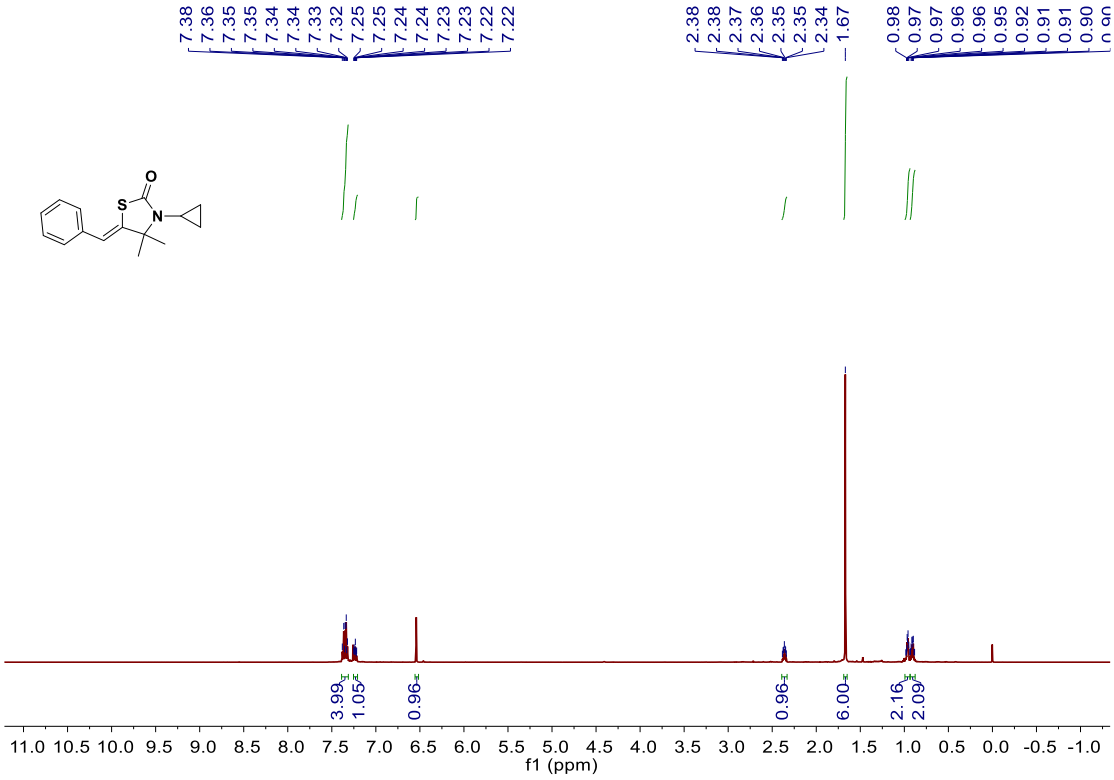
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-5-benzylidene-4,4-dimethyl-3-propylthiazolidin-2-one (2n)



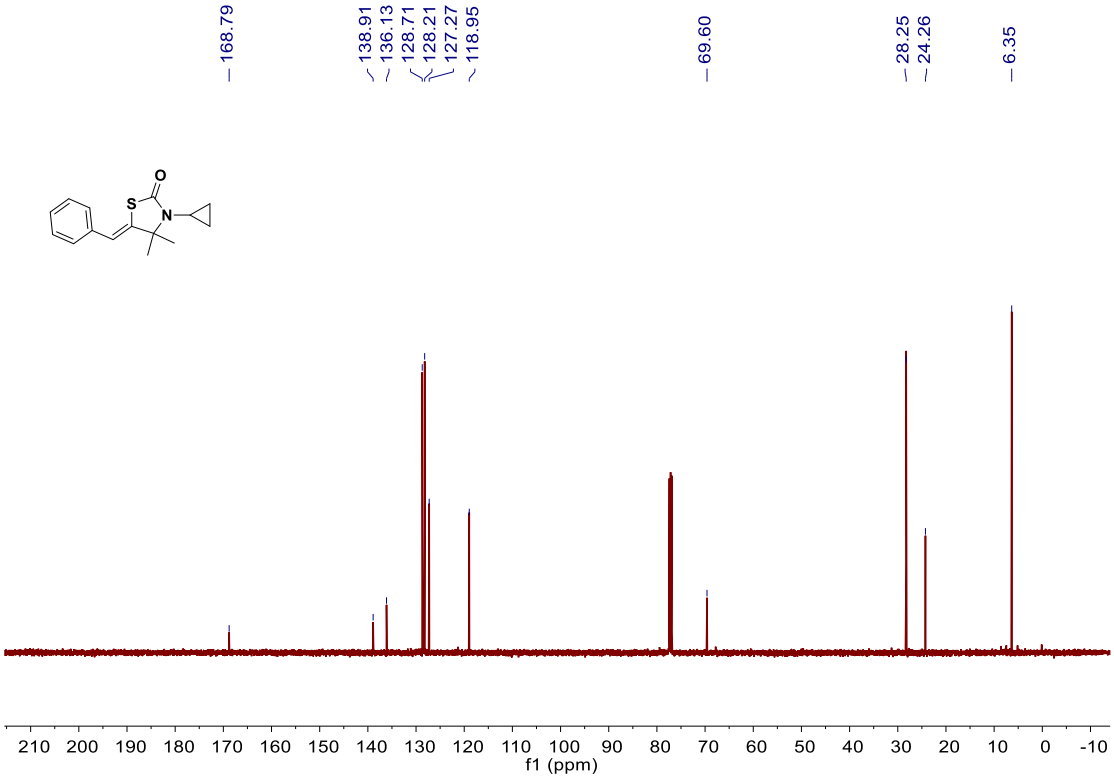
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-5-benzylidene-4,4-dimethyl-3-propylthiazolidin-2-one (2n)



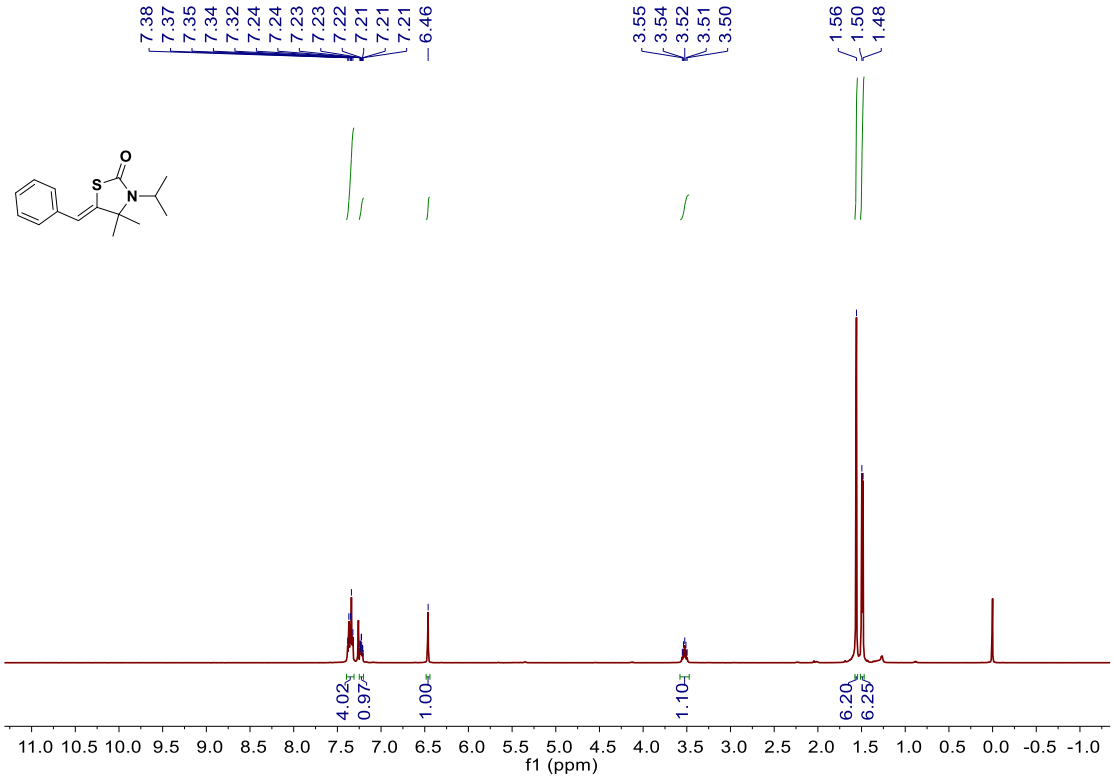
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-5-benzylidene-3-cyclopropyl-4,4-dimethylthiazolidin-2-one (2o)



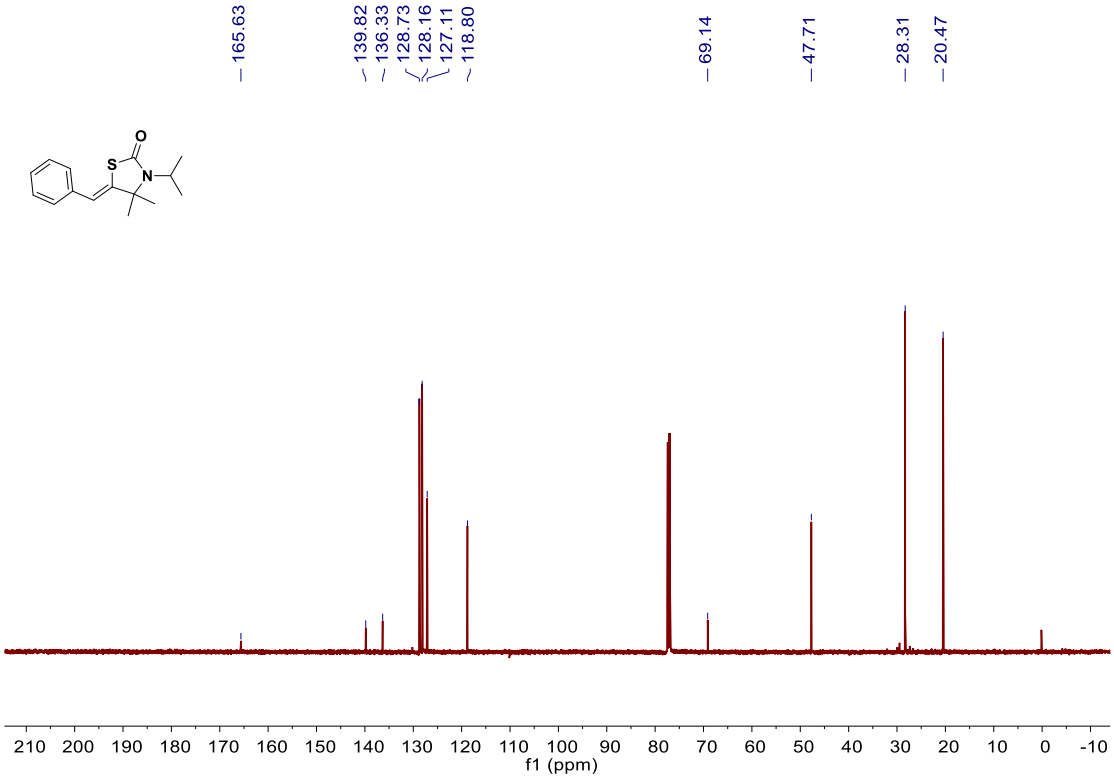
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-5-benzylidene-3-cyclopropyl-4,4-dimethylthiazolidin-2-one (2o)



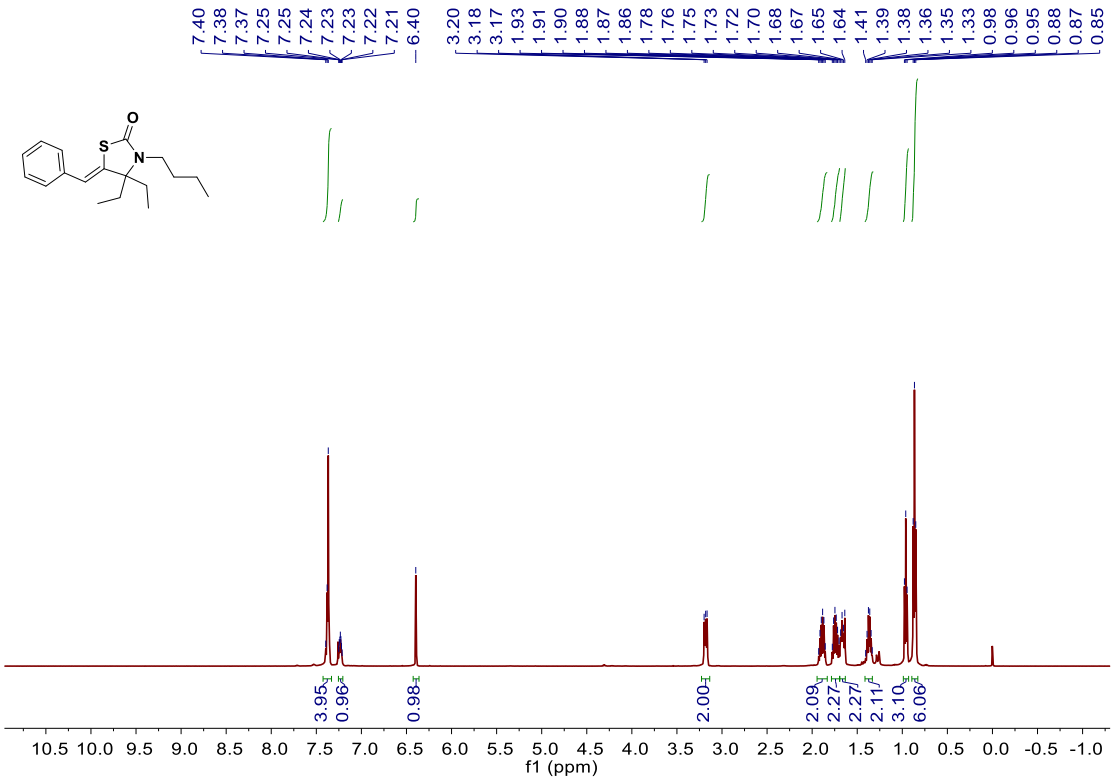
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-5-benzylidene-3-isopropyl-4,4-dimethylthiazolidin-2-one (2p)



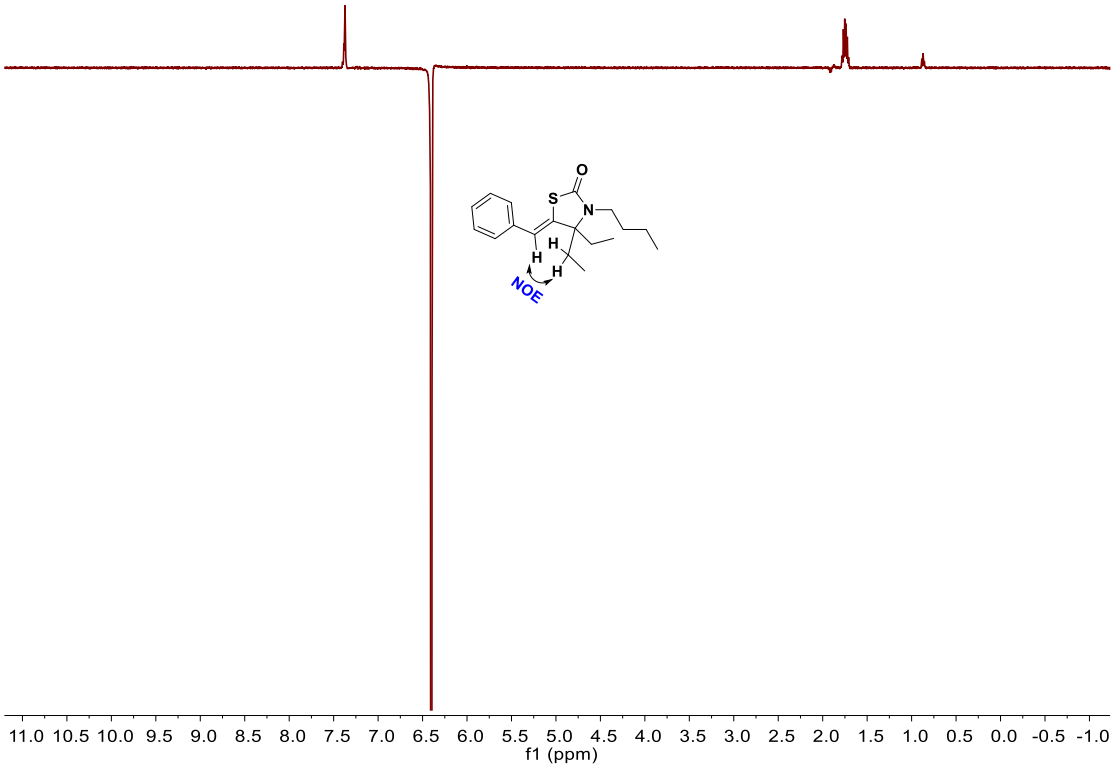
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-5-benzylidene-3-isopropyl-4,4-dimethylthiazolidin-2-one (2p)



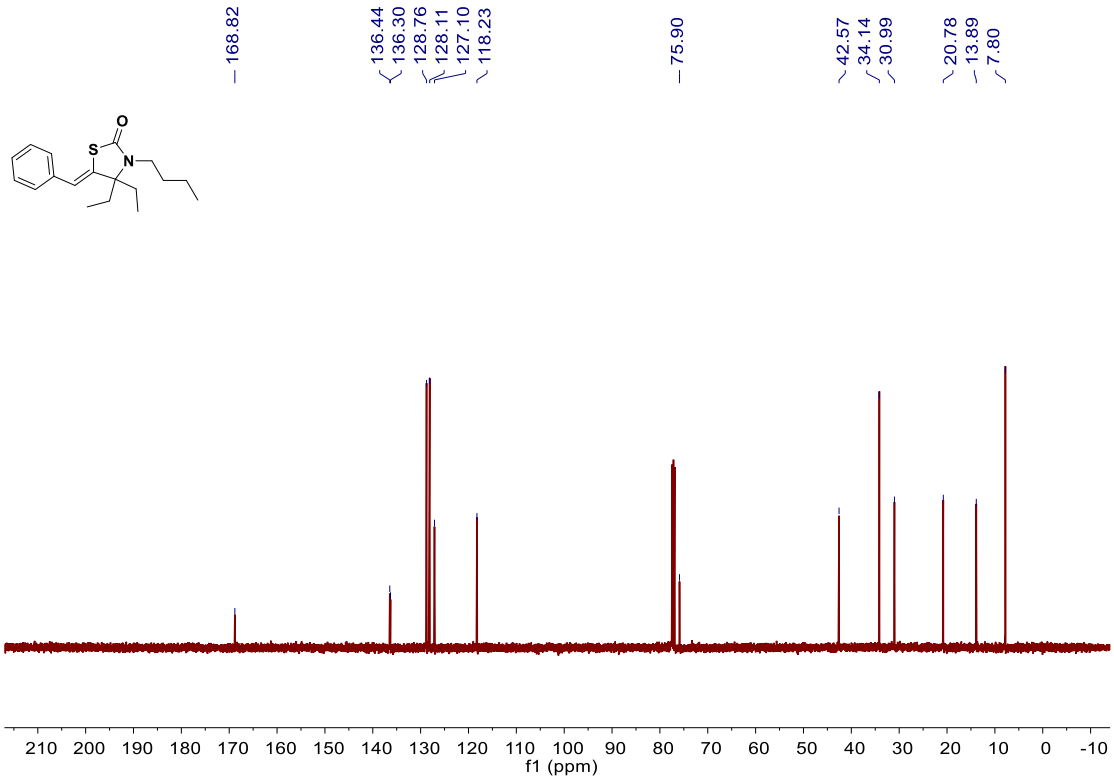
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-5-benzylidene-3-butyl-4,4-diethylthiazolidin-2-one (2s)



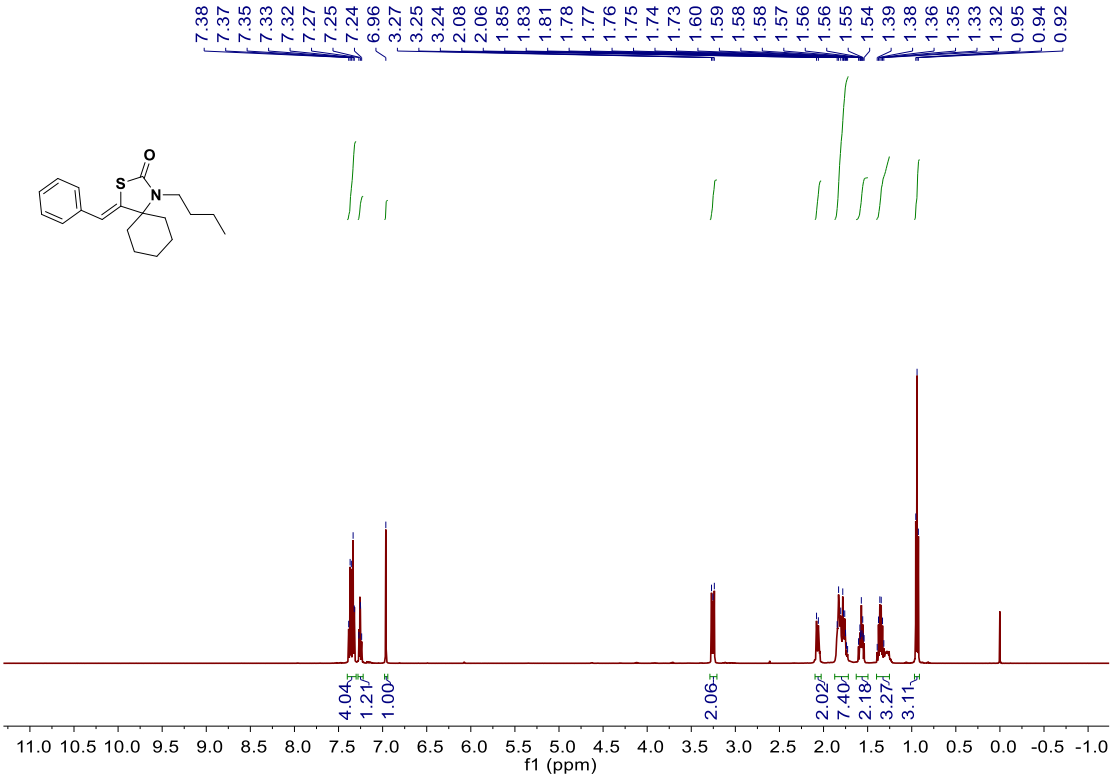
NOE spectrum of (*Z*)-5-benzylidene-3-butyl-4,4-diethylthiazolidin-2-one (2s)



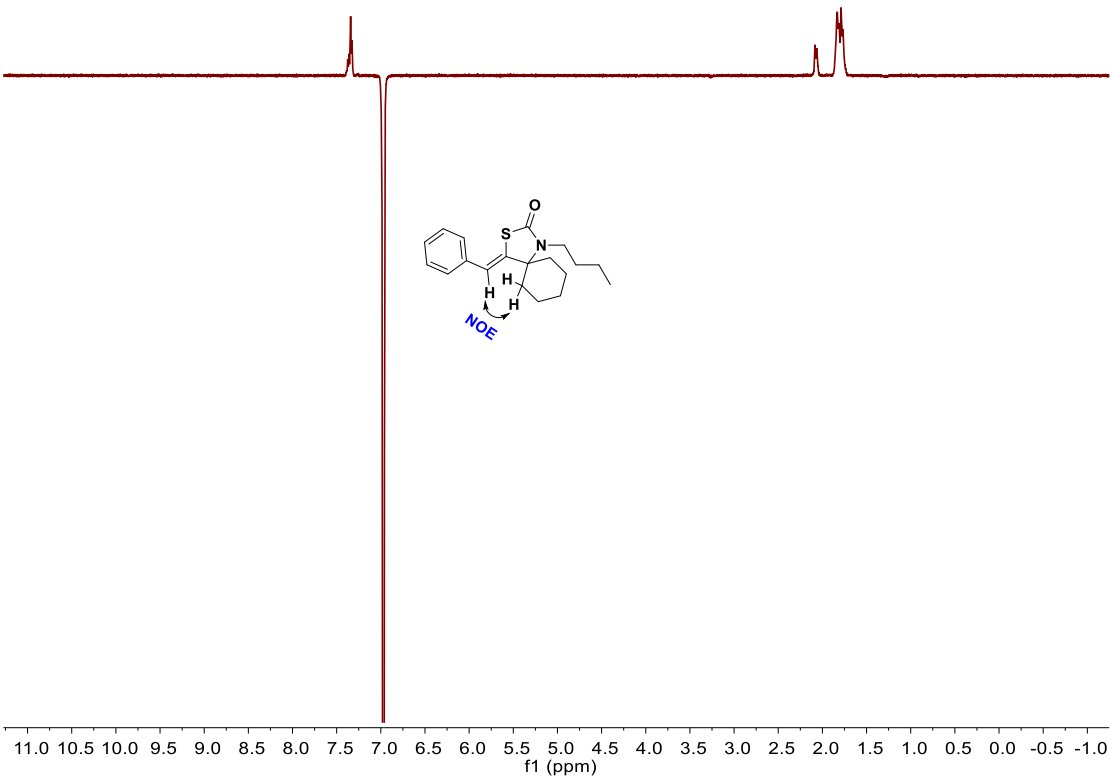
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-5-benzylidene-3-butyl-4,4-diethylthiazolidin-2-one (2s)



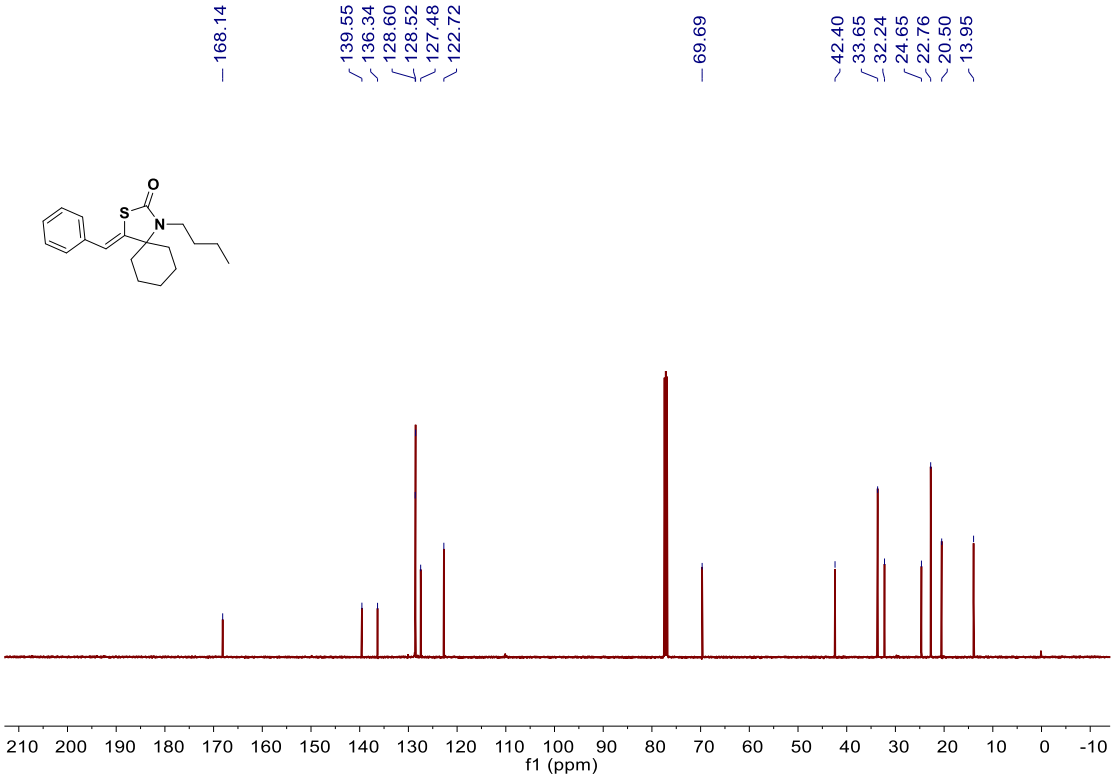
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-4-benzylidene-1-butyl-3-thia-1-azaspiro[4.5]decan-2-one (2t)



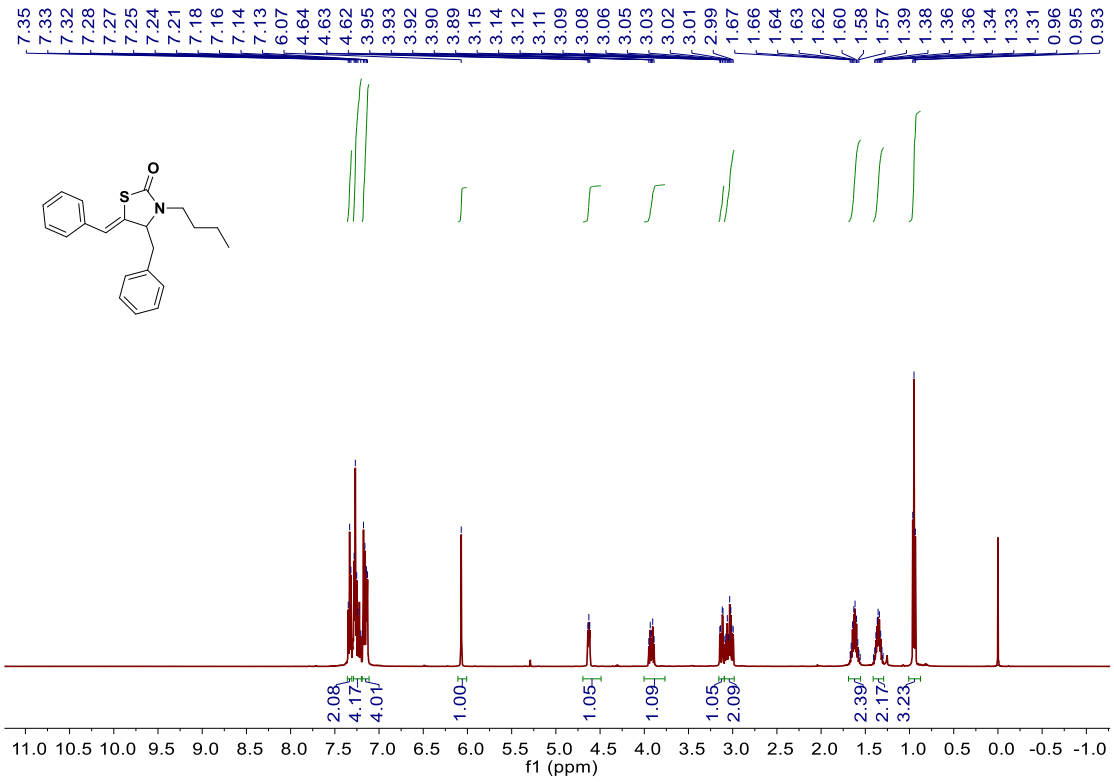
NOE spectrum of (*Z*)-4-benzylidene-1-butyl-3-thia-1-azaspiro[4.5]decan-2-one (2t)



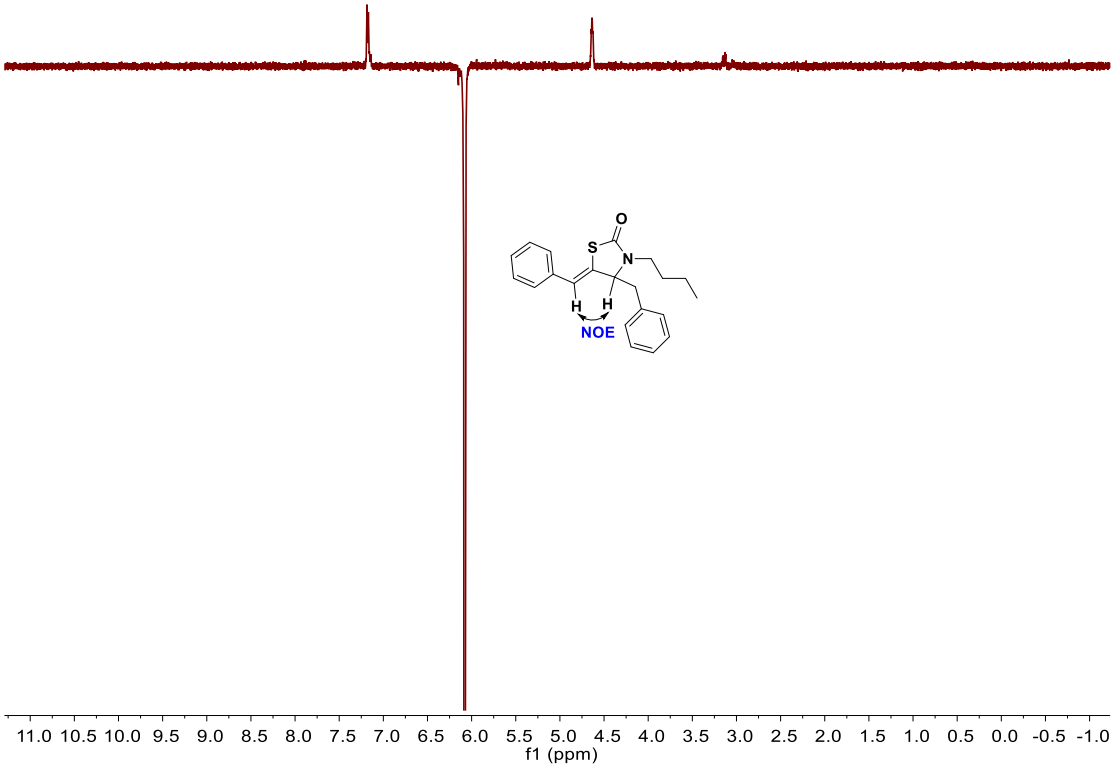
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-4-benzylidene-1-butyl-3-thia-1-azaspiro[4.5]decan-2-one (2t)



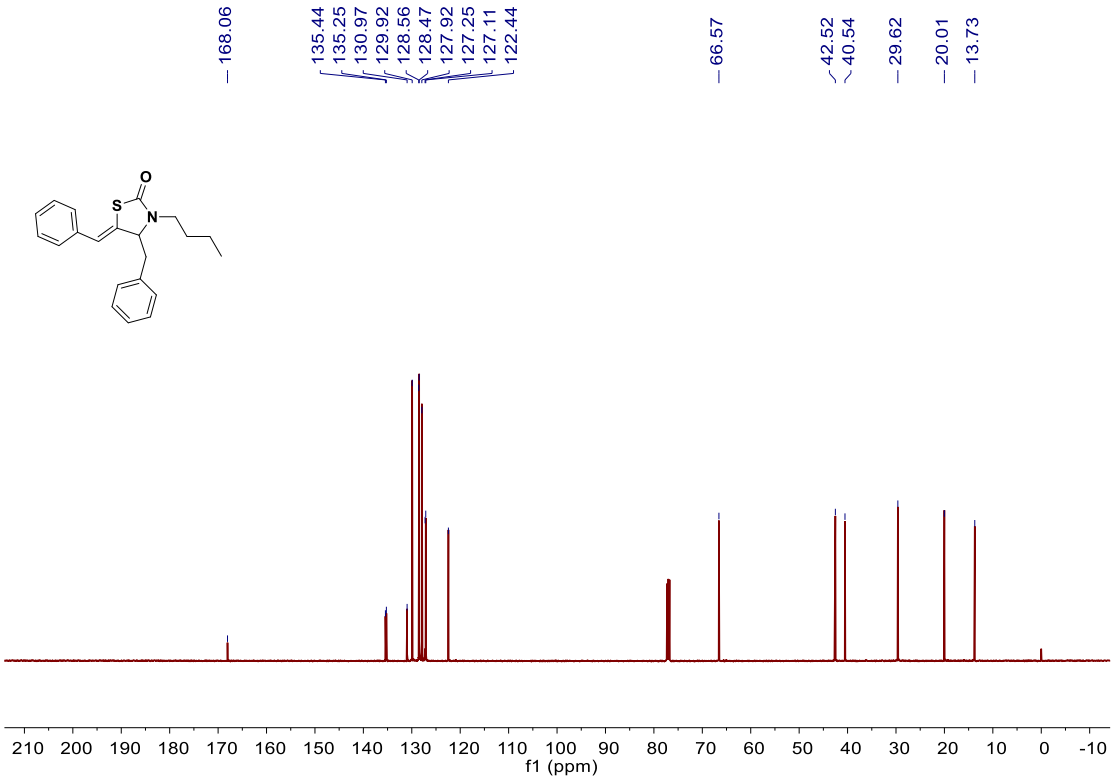
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-4-benzyl-5-benzylidene-3-butylthiazolidin-2-one (2u)



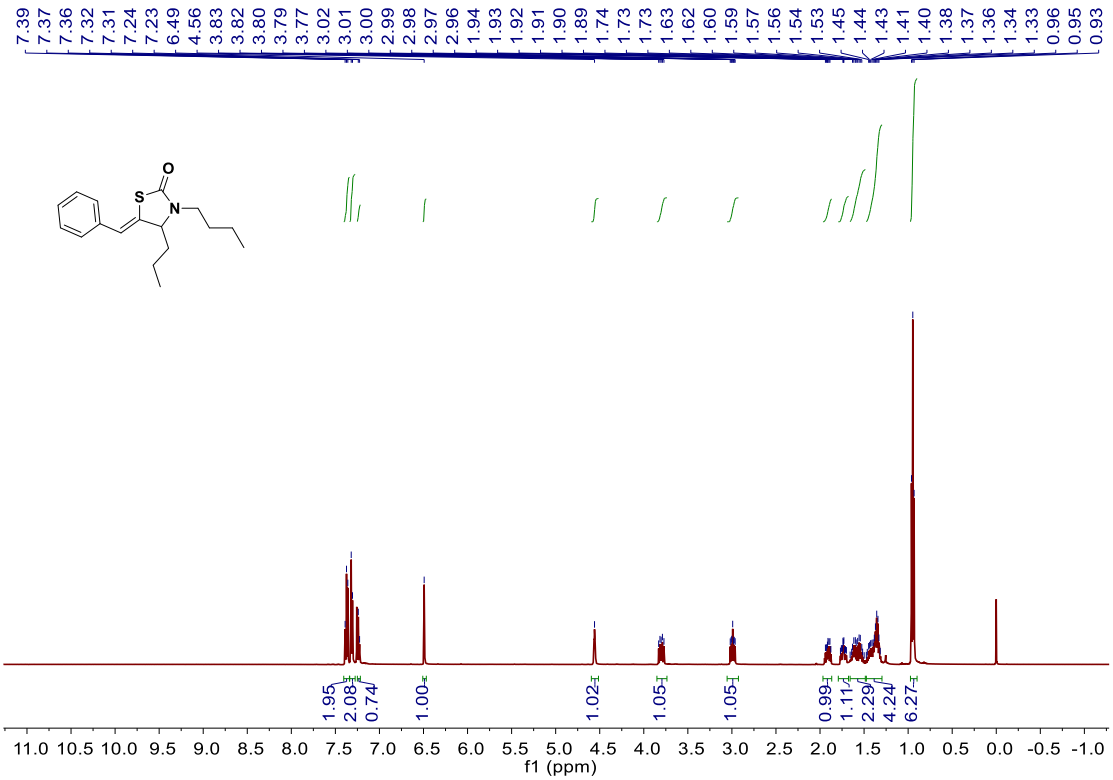
NOE spectrum of (*Z*)-4-benzyl-5-benzylidene-3-butylthiazolidin-2-one (2u)



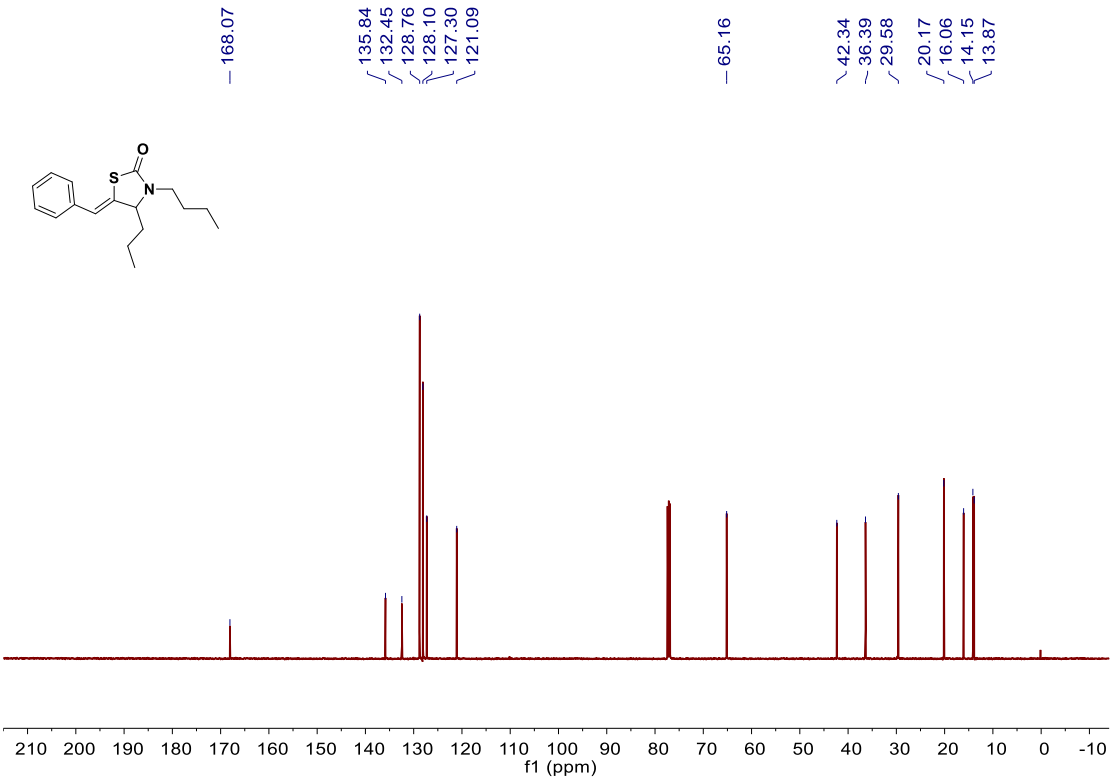
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-4-benzyl-5-benzylidene-3-butylthiazolidin-2-one (2u)



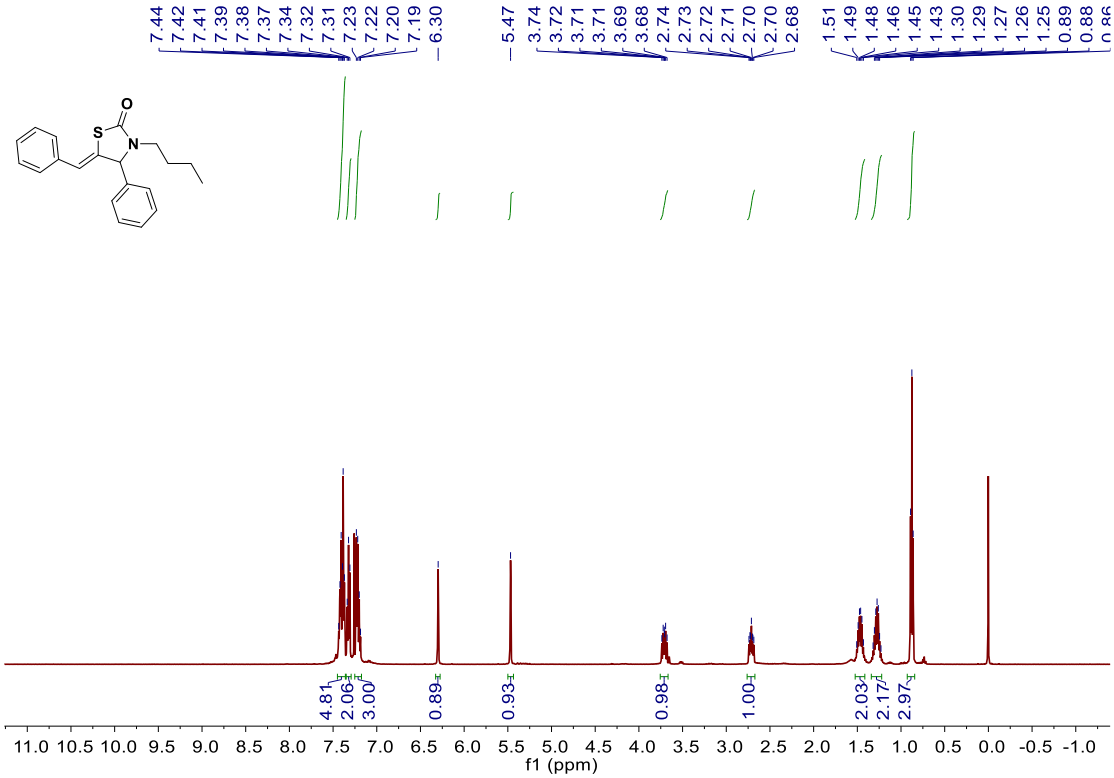
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-5-benzylidene-3-butyl-4-propylthiazolidin-2-one (2v)



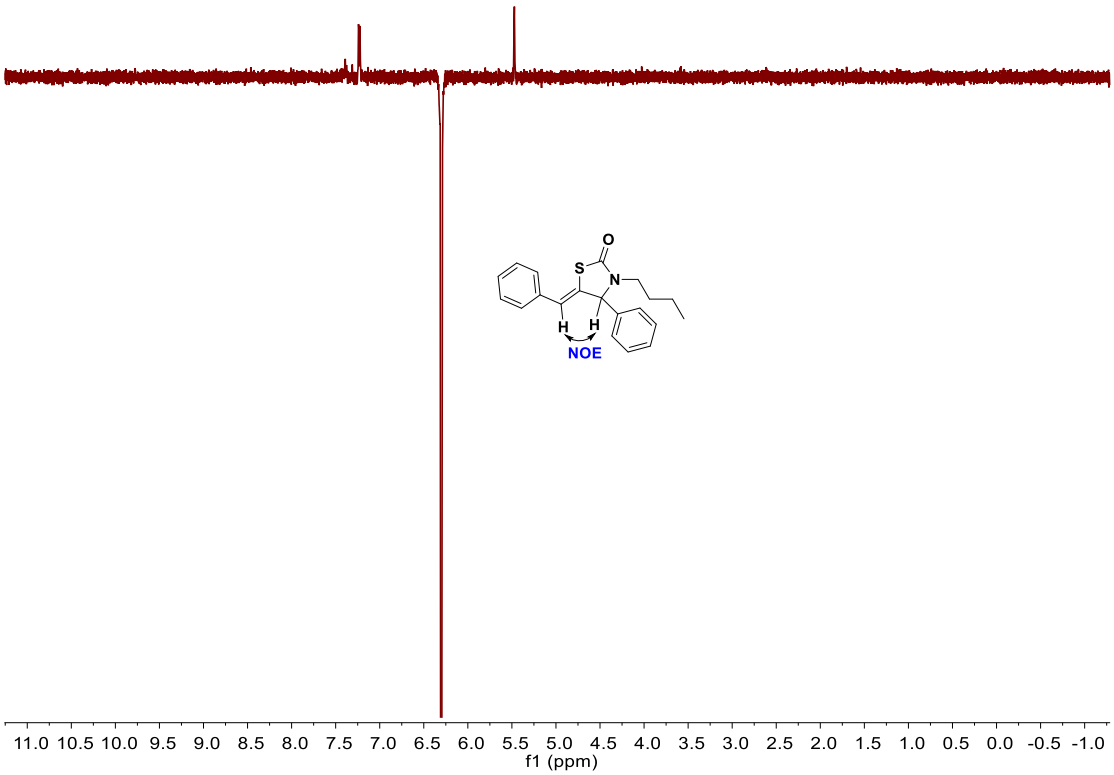
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-5-benzylidene-3-butyl-4-propylthiazolidin-2-one (2v)



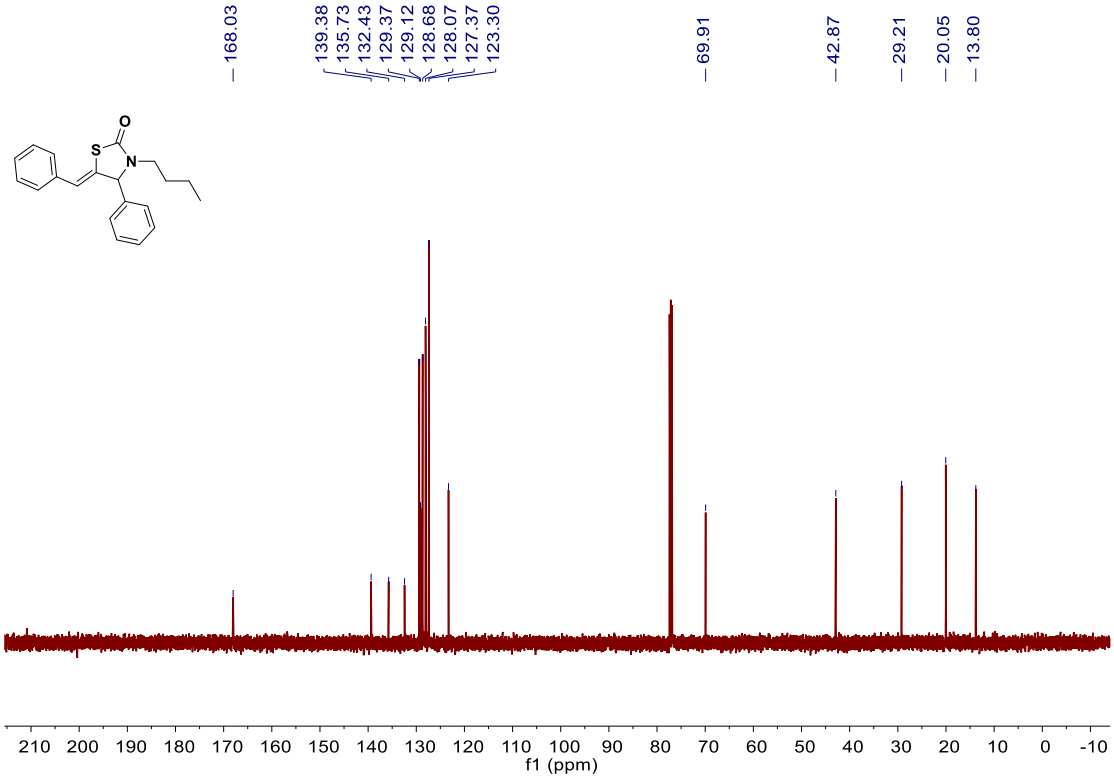
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-5-benzylidene-3-butyl-4-phenylthiazolidin-2-one (2w)



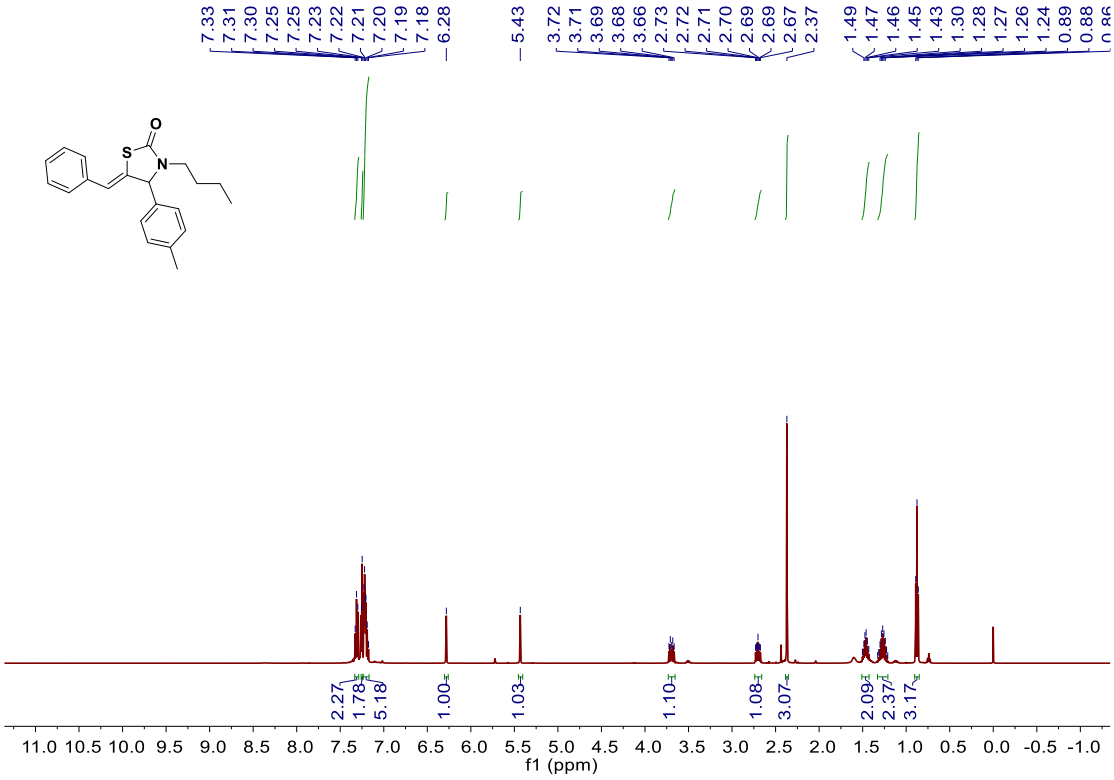
NOE spectrum of (*Z*)-5-benzylidene-3-butyl-4-phenylthiazolidin-2-one (2w)



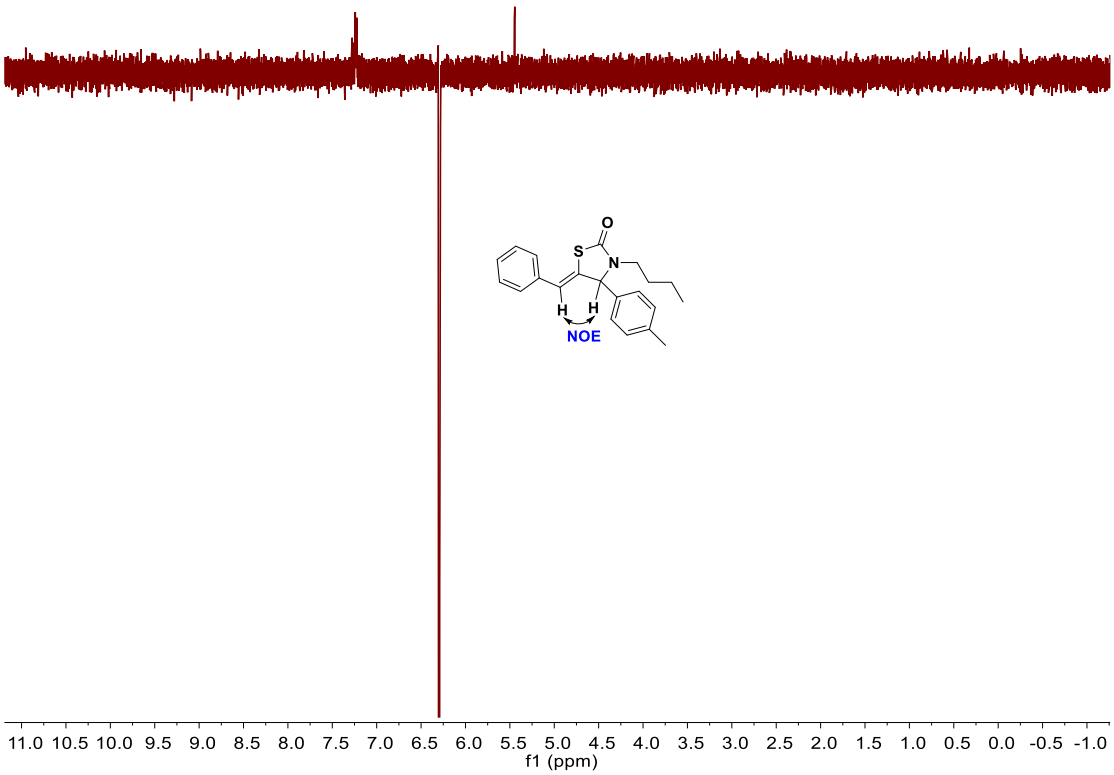
^{13}C NMR ($CDCl_3$, 125 MHz) of <u>(*Z*)-5-benzylidene-3-butyl-4-phenylthiazolidin-2-one (2w)</u>



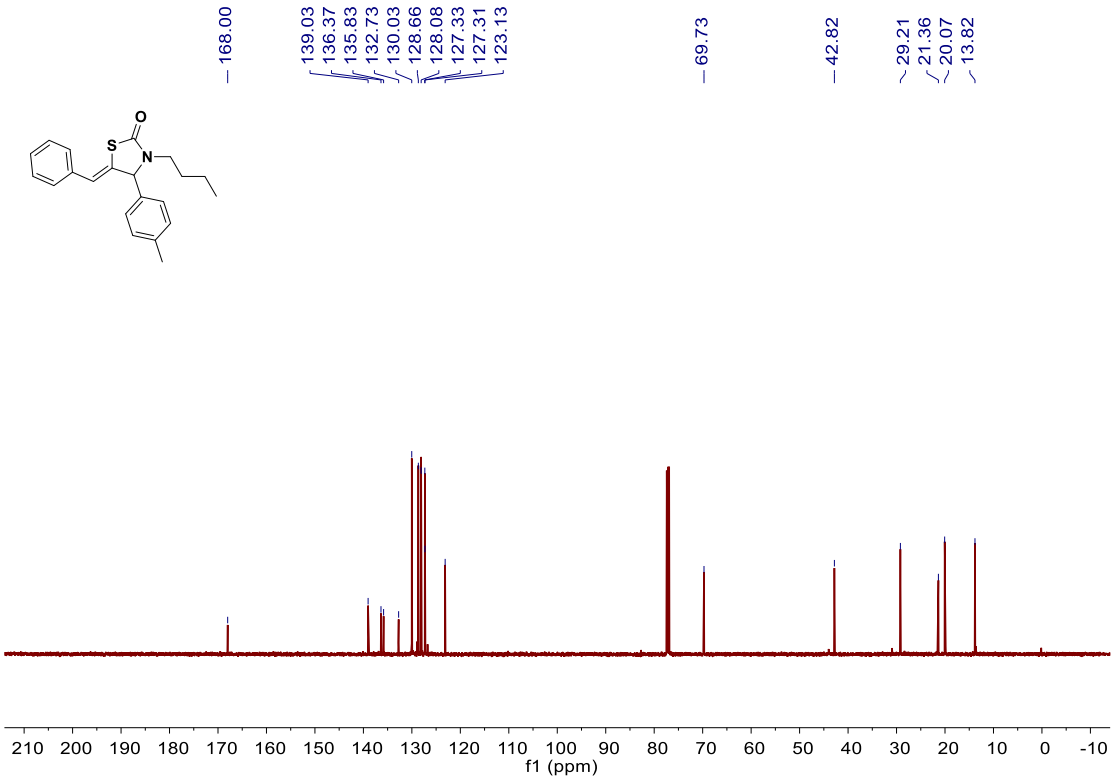
^{1}H NMR ($CDCl_3$, 500 MHz) of <u>(*Z*)-5-benzylidene-3-hexyl-4-(p-tolyl)thiazolidin-2-one (2x)</u>



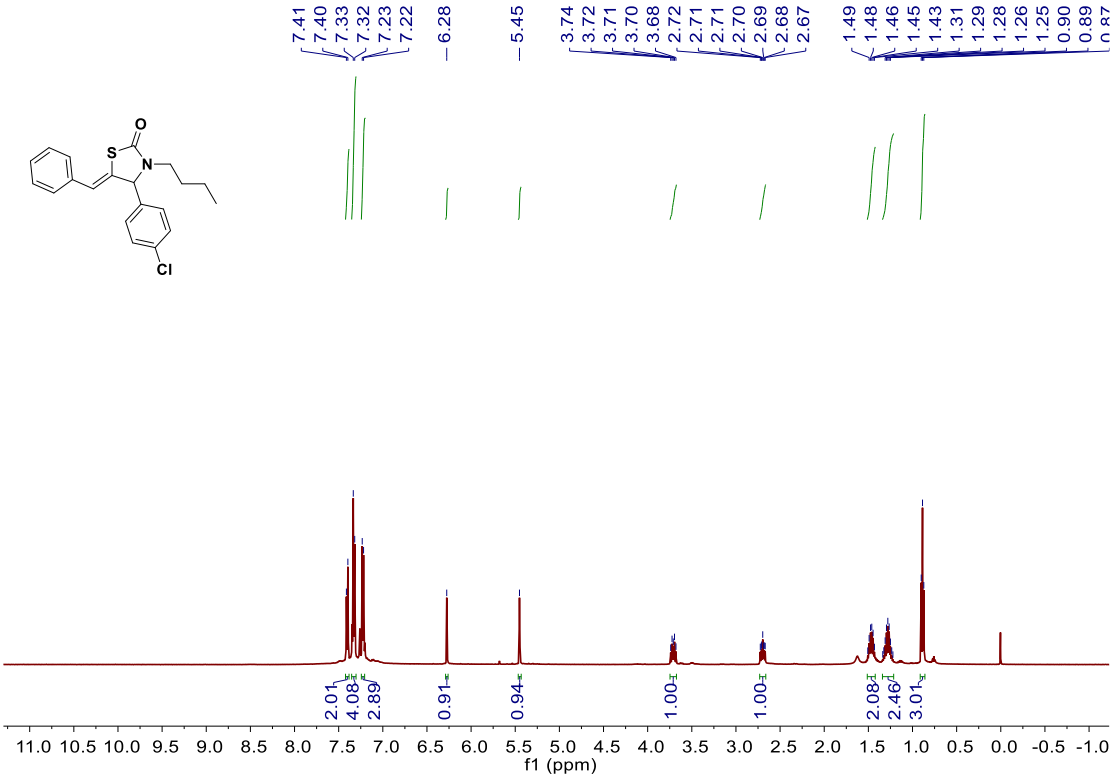
NOE spectrum of (*Z*)-5-benzylidene-3-hexyl-4-(p-tolyl)thiazolidin-2-one (2x)



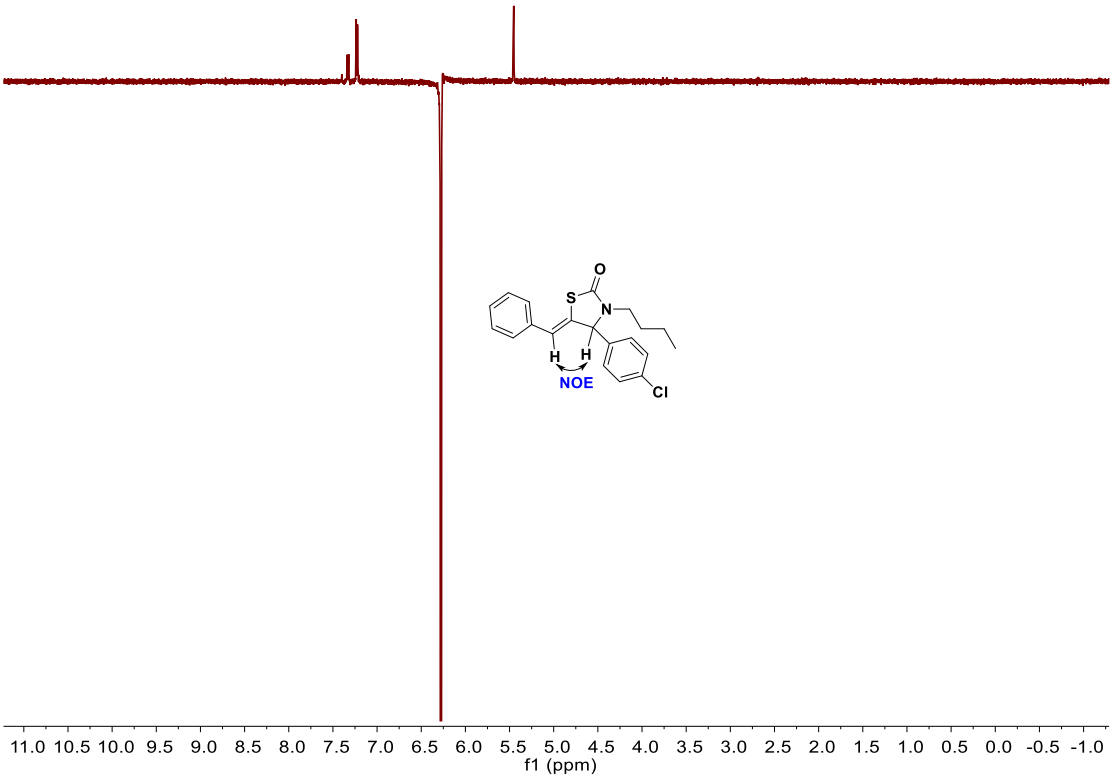
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-5-benzylidene-3-hexyl-4-(p-tolyl)thiazolidin-2-one (2x)



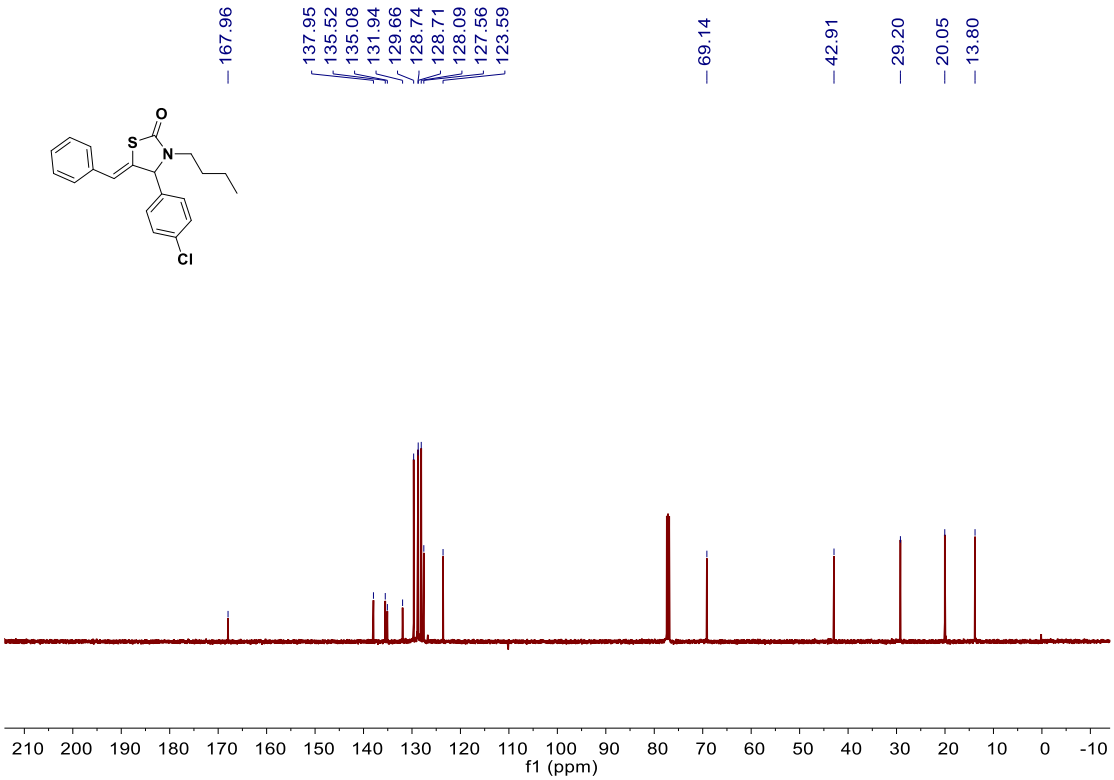
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-5-benzylidene-3-butyl-4-(4-chlorophenyl)thiazolidin-2-one (2y)



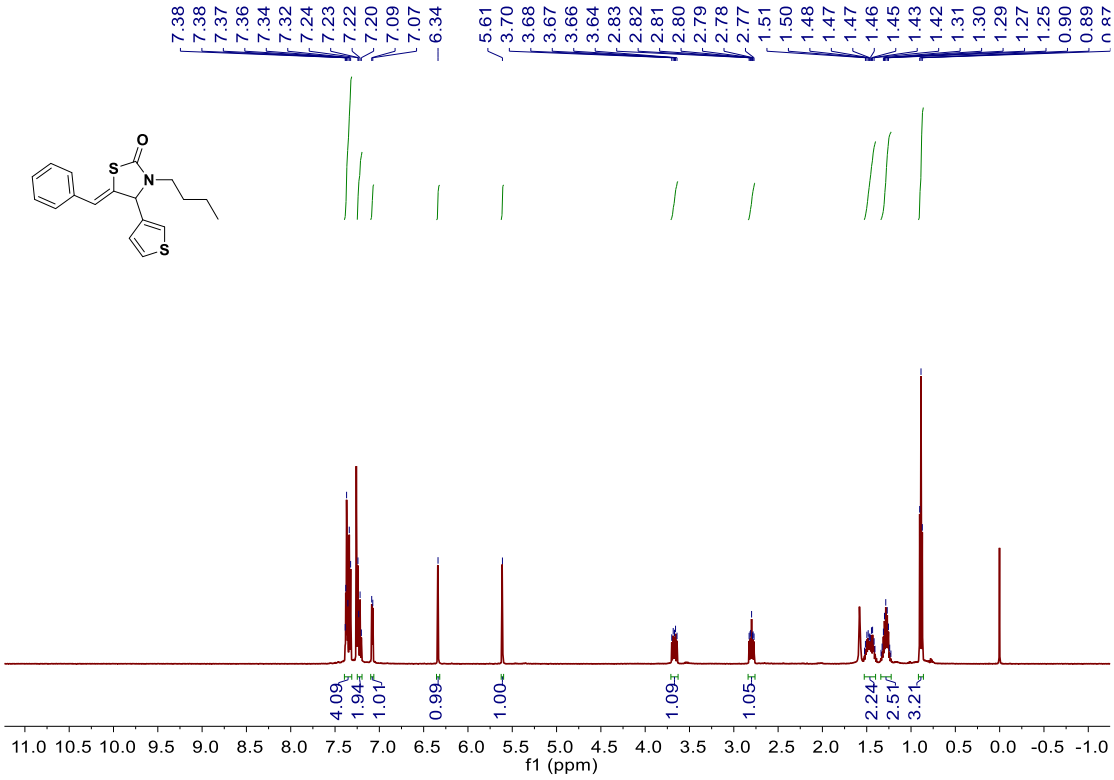
NOE spectrum of (*Z*)-5-benzylidene-3-butyl-4-(4-chlorophenyl)thiazolidin-2-one (2y)



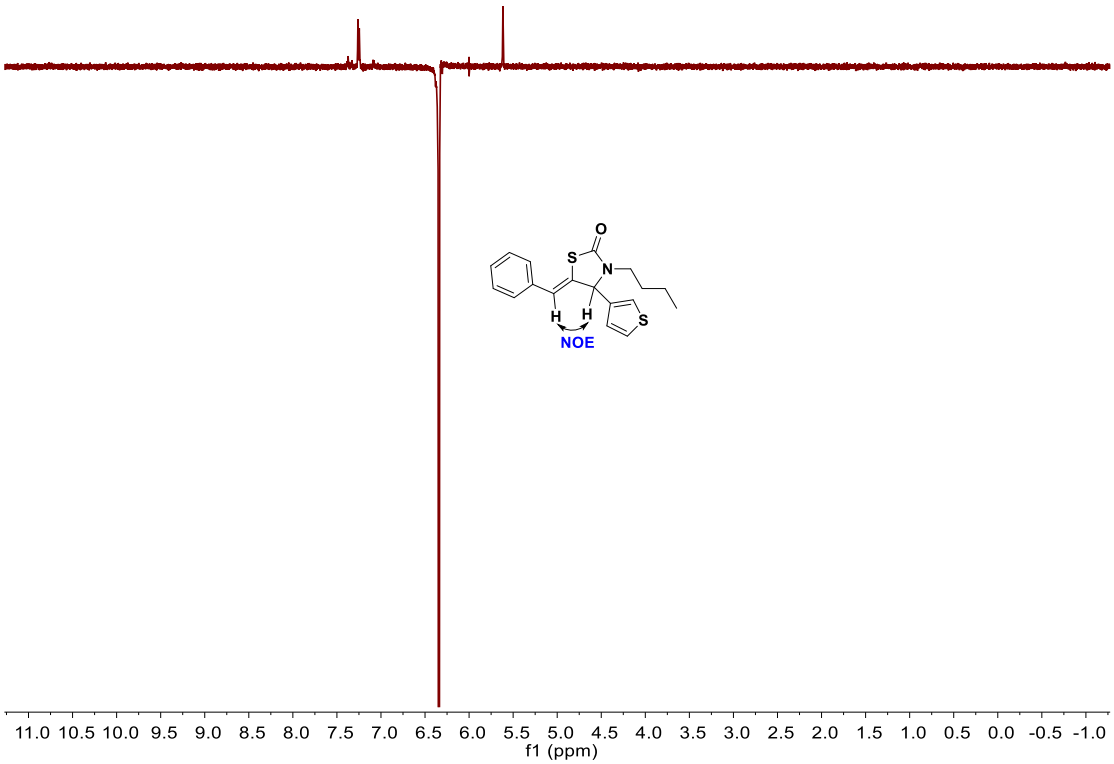
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-5-benzylidene-3-butyl-4-(4-chlorophenyl)thiazolidin-2-one (2y)



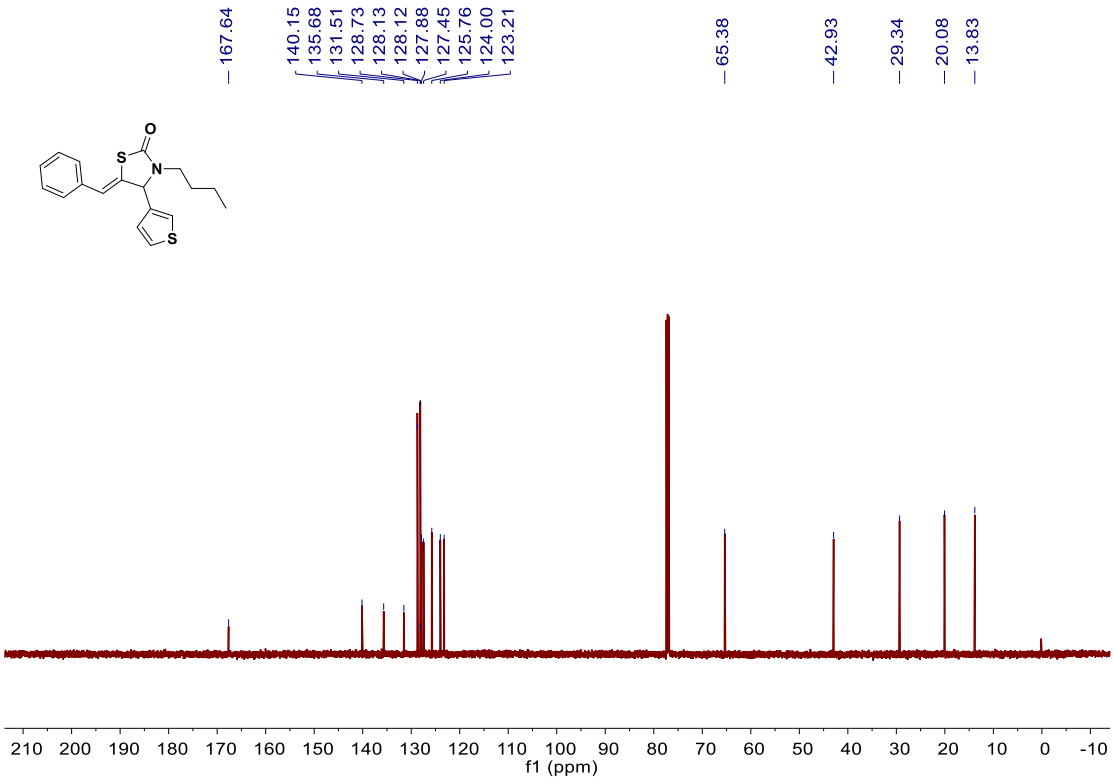
^{1}H NMR ($CDCl_3$, 500 MHz) of (*Z*)-5-benzylidene-3-butyl-4-(thiophen-3-yl)thiazolidin-2-one (2z)



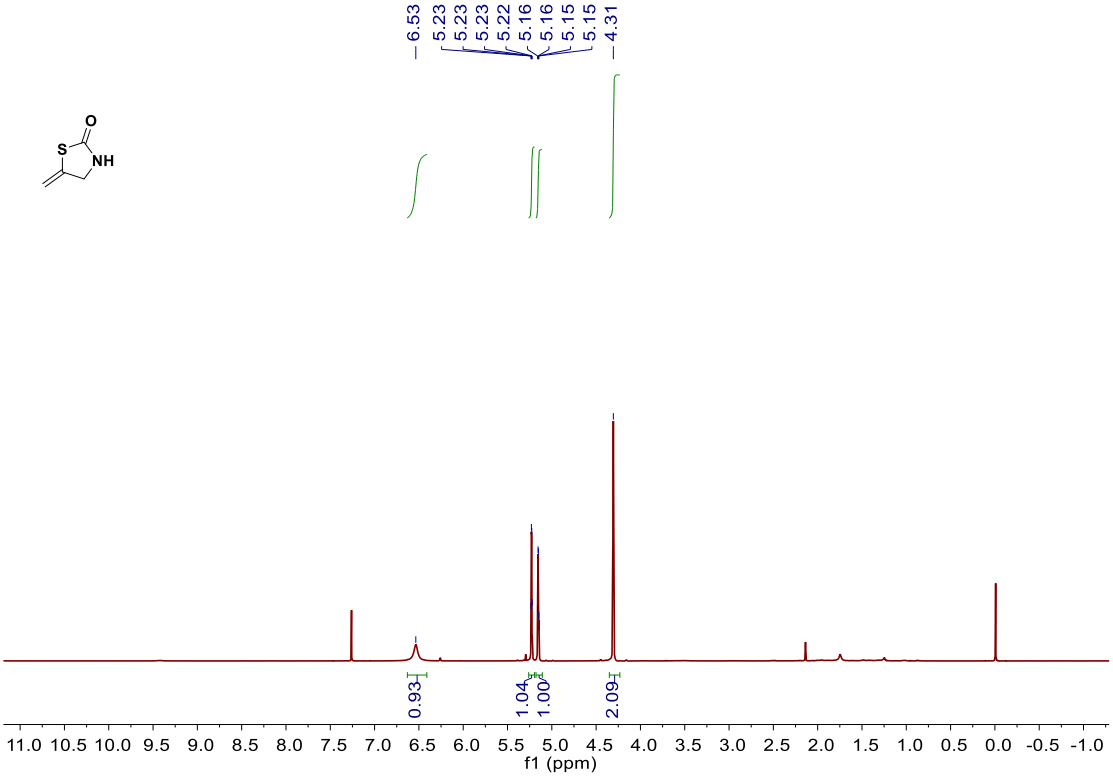
NOE spectrum of (*Z*)-5-benzylidene-3-butyl-4-(thiophen-3-yl)thiazolidin-2-one (2z)



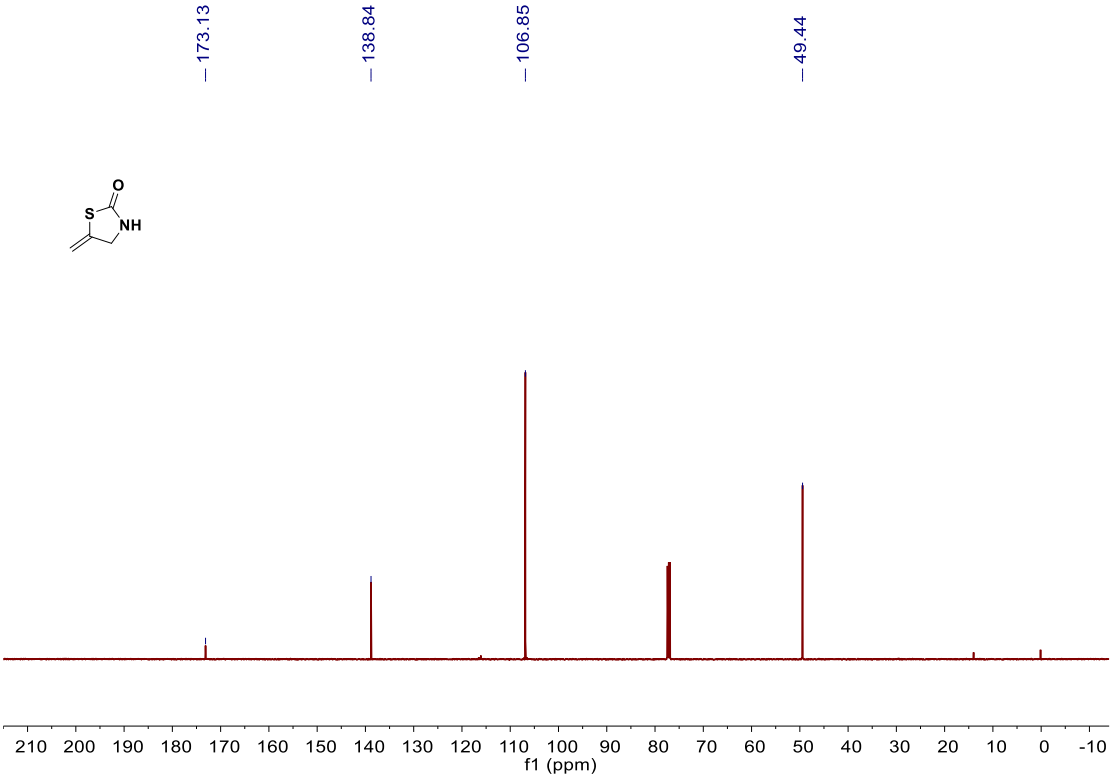
^{13}C NMR ($CDCl_3$, 125 MHz) of (*Z*)-5-benzylidene-3-butyl-4-(thiophen-3-yl)thiazolidin-2-one (2z)



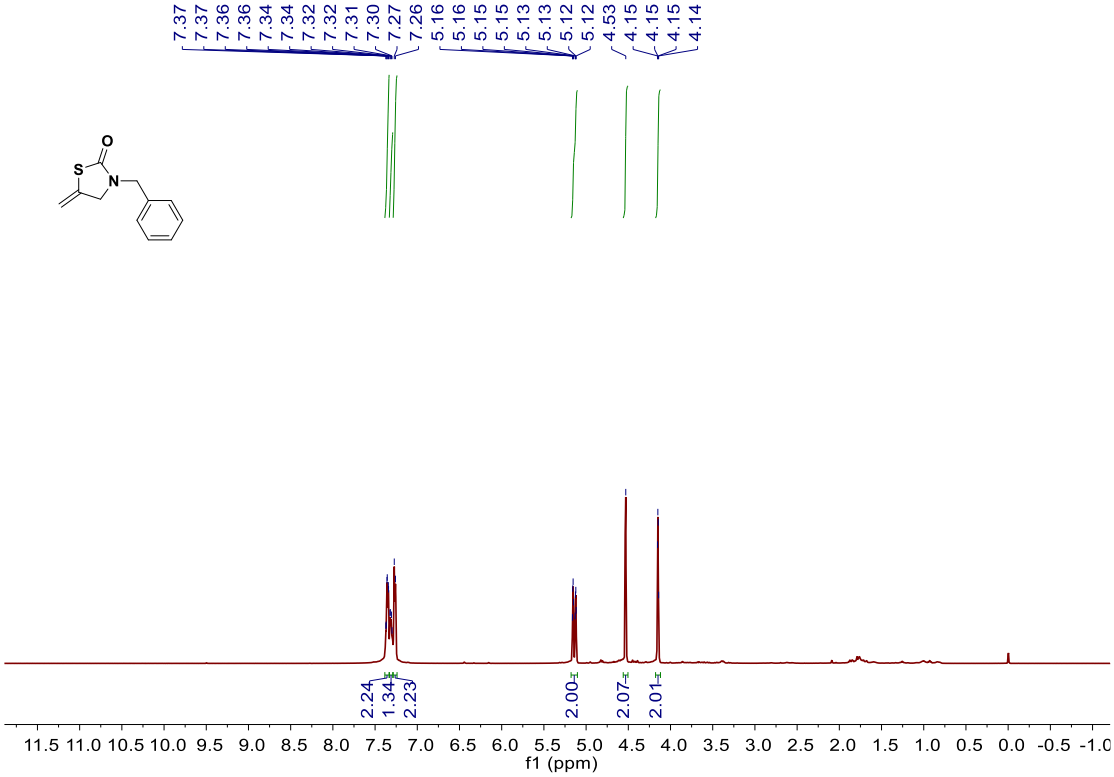
^{1}H NMR ($CDCl_3$, 500 MHz) of 5-methylenethiazolidin-2-one (2aa)



^{13}C NMR ($CDCl_3$, 125 MHz) of 5-methylenethiazolidin-2-one (2aa)



^{1}H NMR ($CDCl_3$, 500 MHz) of 3-benzyl-5-methylenethiazolidin-2-one (2ab)



^{13}C NMR ($CDCl_3$, 125 MHz) of 3-benzyl-5-methylenethiazolidin-2-one (2ab)