catalysts

MDPI

Supplementary Materials

Green Synthesis of 2-Oxazolidinones by an Efficient and Recyclable CuBr/Ionic Liquid System via CO_2, Propargylic Alcohols, and 2-Aminoethanols

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1. TON reported in the previous literatures.

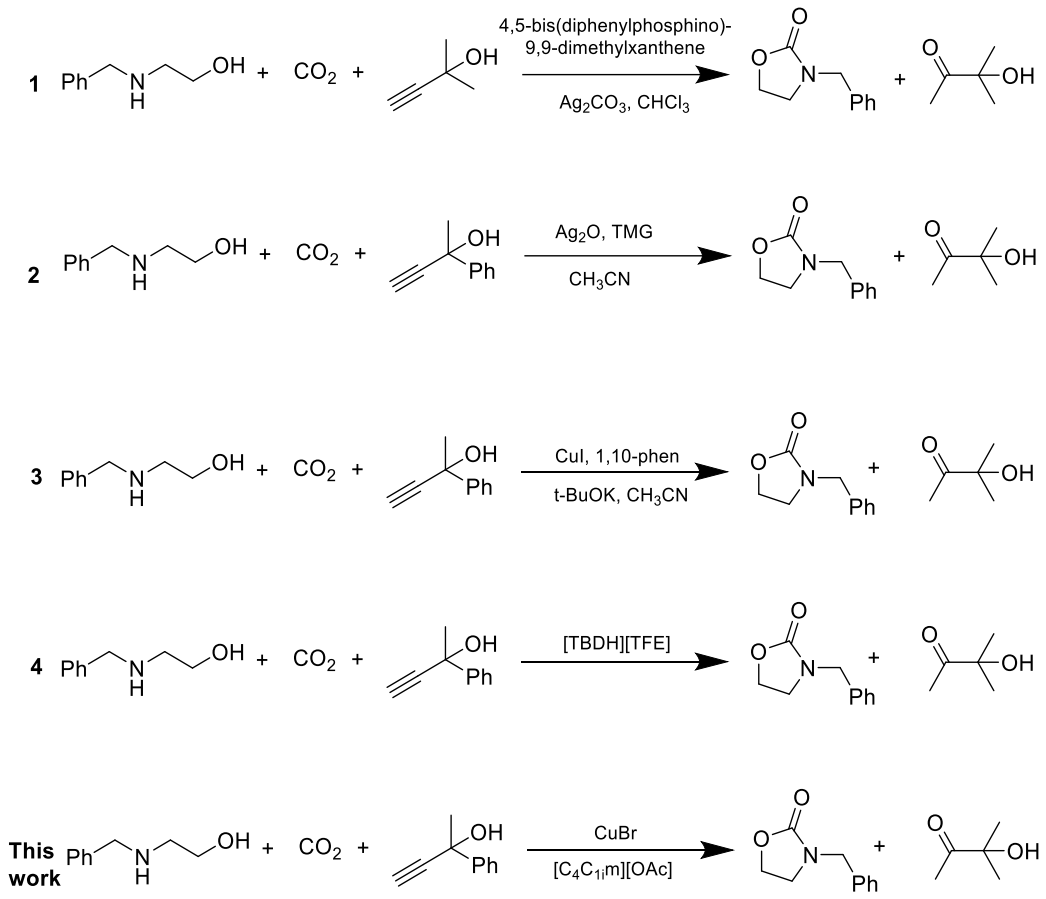


Figure S1. The literatures reported for the three-component reactions.

Figure S1 showed the literatures reported for the three-component reactions up to now. A TON of 1260 could be obtained by the Ag_2O/TMG catalytic system. Particularly, a higher TON of 2960 was reached by this CuBr/[C_4C_1im][OAc] system. For the other systems, no TON data was expressly reported, indicating that 2960 is the highest TON up to now for this three-component reaction.

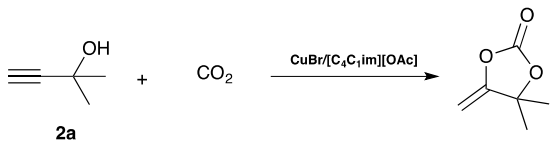
Table S1. TON reported in the previous literatures.

Ref.	TON
1[1]	No mention
2[2]	1260
3[3]	No mention
4[4]	No mention
This work	2960

2. The reaction of propargylic alcohols and CO_2

To prove whether the cyclic carbonates could be produced without the use of aminoethanols, the control experiment was performed as follows: **2a** (7.5 mmol), CuBr (0.025 mmol) and [C_4C_1im][OAc] (6.5 mmol) were prepared in a Schlenk tube under 1 atm of CO_2 at 100 °C. After 12 h, the reaction mixture was directly in situ examined by ^{1}H NMR in $CDCl_3$ (Figure S2). A characteristic

double peak of the α-alkylidene cyclic carbonate at δ = 4.788 was clearly observed [1], indicating the formation of the corresponding carbonate.



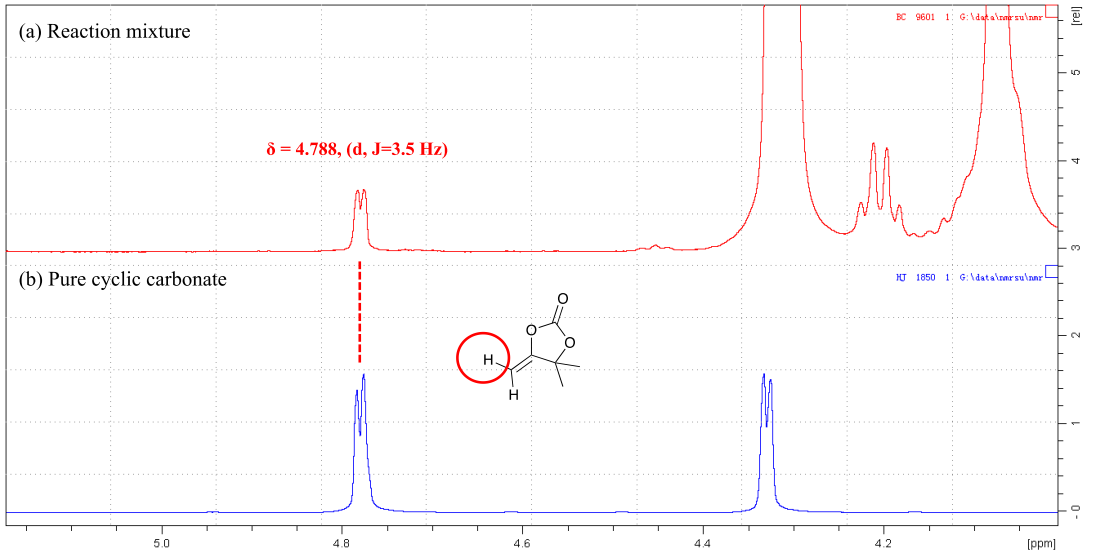
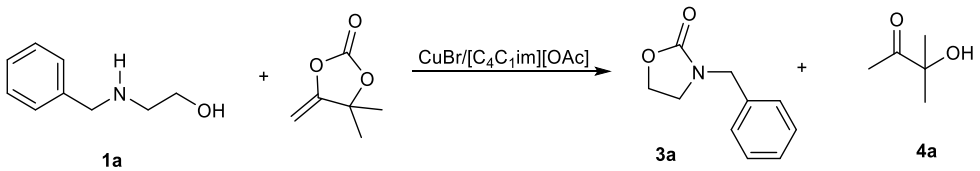


Figure S2. ^{1}H NMR of the control experiment mixture (red) and the pure cyclic carbonate (blue).

3. The reaction of cyclic carbonates and 2-aminoethanols

To prove whether the cyclic carbonates from step 1 would react with 2-aminoethanols to give the desired products using the CuBr/[C4C1im][OAc] catalytic system, the following control experiment was performed: **1a** (5 mmol), cyclic carbonates (7.5 mmol), CuBr (0.025 mmol, 0.5 mol%) and [C4C1im][OAc] (6.5 mmol) were mixed in a Schlenk tube under 1 atm of CO_2 at 100 °C for 12 h. Once the reaction was finished, the reaction mixture was directly in situ examined by ^{1}H NMR in $CDCl_3$ (Figure S3). The characteristic peaks of **3a** at δ = 3.43, 4.32, 4.45 and **4a** at δ = 1.41, 2.26, 3.77 were clearly observed, indicating the formation of the final products from cyclic carbonates and 2-aminoethanols [2].



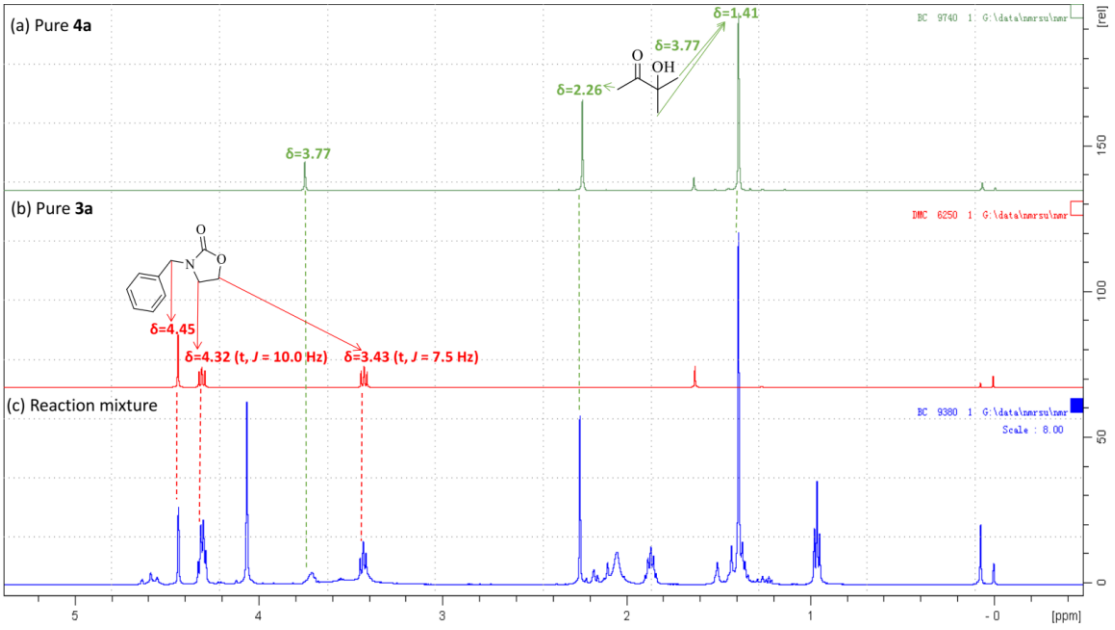


Figure S3. ^{1}H NMR of pure **4a** (green), pure **3a** (red) and the control reaction mixture (blue).

4. Activation of the hydroxyl group in the presence of 1 atm of CO_2

Substrate **2a**, and the mixture of **2a**/[C_4C_1im][OAc] (1.5:1.3), **2a**/**1a** (1.5:1) were respectively prepared in the closed Schlenk tubes at 1 atm of CO_2 at 100 °C. After 5 mins, three samples were respectively taken from them into DMSO-d_6 and examined by ^{1}H NMR (Figure S4). As the NMR results showed, in the presence of 1 atm of CO_2, the hydroxyl groups of propargylic alcohols could be strongly activated by [C_4C_1im][OAc] and slightly activated by 2-aminoethanols.

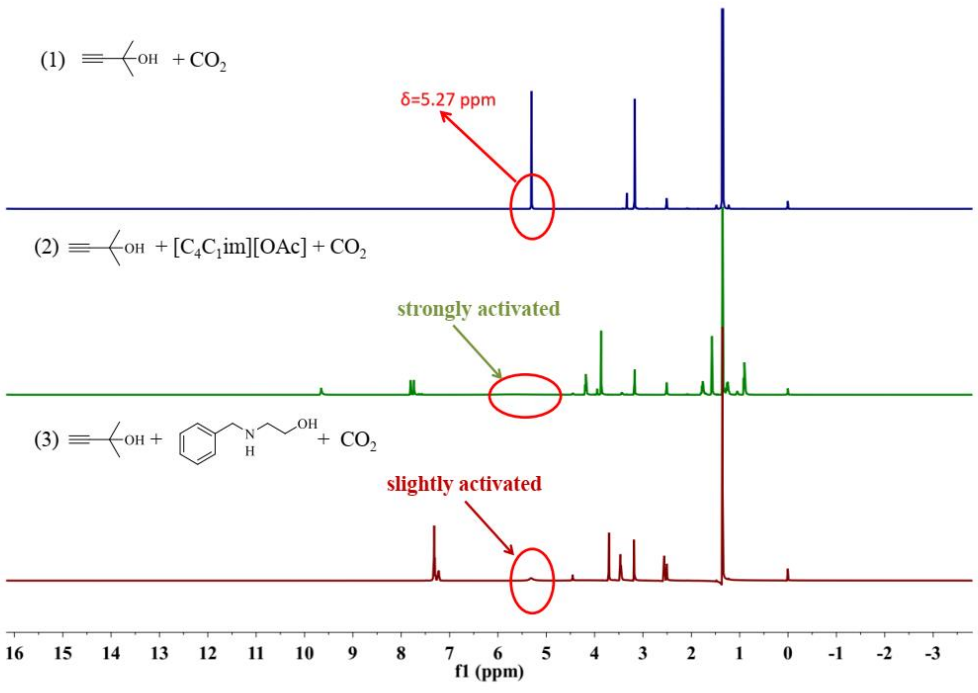


Figure S4. Investigations on the activation of hydroxyl protons in the presence of 1 atm of CO_2.

5. Exploration of metal leaching in the recycling experiments

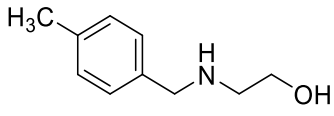
The metal leaching occurred in the extraction process was explored by the ICP analysis after each round. The results were showed below:

Table S2. Exploration of metal leaching in the recycling experiments.

	initial amount	after round 1	after round 2	after round 3
CuBr amount (mmol)	0.02516	0.02124	0.01915	0.01732
Leaching (%)	/	15.58	9.84	9.56

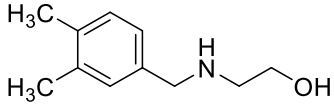
Based on the ICP data, we could calculate that after the first round, 15.58% of CuBr was leached from the system, while after 2nd and 3rd rounds, similar leaching values of 9.84% and 9.56% were obtained respectively.

6. Characterization Data for Substrates and Products



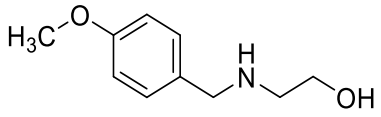
1b

2-(4-Methylbenzylamino)ethanol. Colourless oil. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.22 (d, *J* = 7.9 Hz, 2H), 7.16 (d, *J* = 7.8 Hz, 2H), 3.78 (s, 2H), 3.66 (t, *J* = 5.0 Hz, 2H), 2.81 (t, *J* = 5.0Hz, 2H), 2.36 (s, 3H), 2.19(-OH, -NH) ppm. ^{13}C NMR (126 MHz, Chloroform-*d*) δ 137.0, 136.7, 129.2, 128.1, 61.0, 53.2, 50.5, 21.1 ppm. These data are matched with the reported publication [3]. HRMS (ESI) calcd for $C_{10}H_{16}NO^{+}$ [M+H]$^{+}$ 166.1226; found 166.1227.



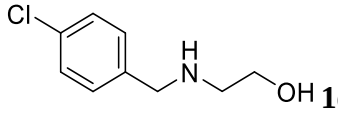
1c

2-(3,4-Dimethylbenzylbenzylamino)ethanol. Colorless solid. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.17–7.03 (m, 3H), 3.76 (s, 2H), 3.67 (t, *J* = 5.0 Hz, 2H), 2.82 (t, *J* = 5.0 Hz, 2H), 2.27 (d, *J* = 6.1 Hz, 6H), 2.07(-OH, -NH) ppm. ^{13}C NMR (126 MHz, Chloroform-*d*) δ 137.4, 136.7, 135.4, 129.7, 129.5, 125.5, 60.9, 53.2, 50.5, 19.7, 19.4 ppm. These data are matched with the reported publication [3]. HRMS (ESI) calcd for $C_{11}H_{18}NO^{+}$ [M+H]$^{+}$ 180.1383; found 180.1384.



1d

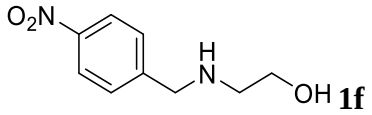
2-(4-Methoxybenzylamino)ethanol. Light yellow oil. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.24 (d, *J* = 8.6 Hz, 2H), 6.87 (d, *J* = 8.6 Hz, 2H), 3.80 (s, 3H), 3.74 (s, 2H), 3.65 (t, *J* = 5.3 Hz, 2H), 2.77 (t, *J* = 5.0 Hz, 2H), 2.69(-OH, -NH) ppm. ^{13}C NMR (126 MHz, Chloroform-*d*) δ 158.8 , 131.78, 129.4, 113.9, 60.8, 55.3, 52.9, 50.5 ppm. These data are matched with the reported publication [3]. HRMS (ESI) calcd for $C_{10}H_{16}NO_{2}^{+}$ [M+H]$^{+}$ 182.1176; found 182.1177.



1e

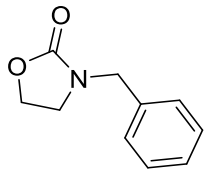
2-(4-Chlorobenzylbenzylamino)ethanol. Light yellow oil. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.33–7.26 (m, 4H), 3.80 (s, 2H), 3.69–3.67 (t, *J* = 5.0 Hz, 2H), 2.82–2.80 (t, *J* = 5.0 Hz, 2H), 2.05(-OH, -NH) ppm. ^{13}C NMR (126 MHz, Chloroform-*d*) δ 138.5, 132.8, 129.4, 128.6, 61.0, 52.8, 50.5 ppm. These

data are matched with the reported publication [3]. HRMS (ESI) calcd for $C_9H_{13}ClNO_2^+$ $[M+H]^+$ 186.0680; found 186.0681.



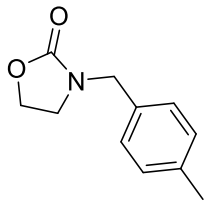
1f

2-(4-Nitrobenzylbenzylamino)ethanol. Brown solid. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.21 (d, *J* = 8.6 Hz, 2H), 7.53 (d, *J* = 8.5 Hz, 2H), 3.96 (s, 2H), 3.73−3.71 (t, *J* = 5.0 Hz, 2H), 2.86-2.84 (t, *J* = 5.0 Hz, 2H), 2.05(-OH, -NH) ppm. ^{13}C NMR (126 MHz, Chloroform-*d*) δ 147.8, 147.2, 128.6, 123.7, 61.2, 52.8, 50.7 ppm. These data are matched with the reported publication [3]. HRMS (ESI) calcd for $C_9H_{13}N_2O_3^+$ $[M+H]^+$ 197.0921; found 197.0922.



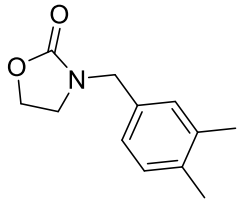
3a

3-Benzyloxazolidin-2-one. Light yellow solid. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.39–7.28 (m, 5H), 4.45 (s, 2H), 4.32 (t, *J* = 10.0 Hz, 2H), 3.46–3.43 (t, *J* = 7.5 Hz, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 135.8, 128.8, 128.2, 128.0, 61.8, 48.5, 44.0 ppm. These data are matched with the reported publication [3]. HRMS (ESI) calcd for $C_{10}H_{11}NNaO_2^+$ $[M+Na]^+$ 200.0682; found 200.0683.



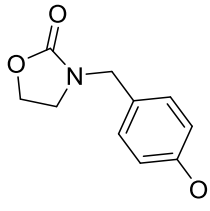
3b

3-(4-Methylbenzyl)oxazolidin-2-one. Colorless oil. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.21–7.17 (m, 4H), 4.40 (s, 2H), 4.30 (t, *J* = 10.0 Hz, 2H), 3.42 (t, *J* =10.0 Hz, 2H), 2.36 (s, 3H) ppm. ^{13}C NMR (126 MHz, Chloroform-*d*) δ 158.5, 137.8, 122.7, 129.5, 128.2, 61.8, 48.1, 43.9, 21.1 ppm. These data are matched with the reported publication [3]. HRMS (ESI) calcd for $C_{11}H_{14}NO_2^+$ $[M+H]^+$ 192.1019; found 192.1020.



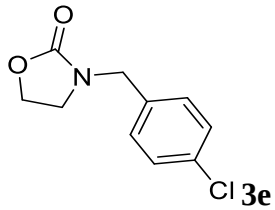
3c

3-(3,4-Dimethylbenzyl)oxazolidin-2-one. Light yellow oil. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.25–7.02 (m, 3H), 4.38 (s, 2H), 4.31 (t, *J* = 10.0 Hz, 2H), 3.43 (t, *J* = 7.5 Hz, 2H), 2.28 (s, 6H) ppm. ^{13}C NMR (126 MHz, Chloroform-*d*) δ 158.5, 137.1, 136.4, 130.0, 129.5, 125.7, 61.8, 48.2, 43.9, 19.7, 19.4 ppm. These data are matched with the reported publication [3]. HRMS (ESI) calcd for $C_{12}H_{16}NO_2^+$ $[M+H]^+$ 206.1176; found 206.1177.



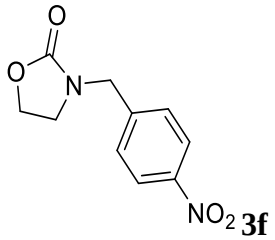
3d

3-(4-Methoxybenzyl)oxazolidin-2-one. Colorless solid. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.14 (d, *J* = 8.5 Hz, 2H), 6.81 (d, *J* = 8.5 Hz, 2H), 4.29 (s, 2H), 4.21 (t, *J* = 10.0 Hz, 2H), 3.73 (s, 3H), 3.33 (t, *J* = 10.0 Hz, 2H) ppm. ^{13}C NMR (126 MHz, Chloroform-*d*) δ 159.4, 158.5, 129.6, 127.8, 114.2, 61.8, 55.3, 47.8, 43.8 ppm. These data are matched with the reported publication [3]. HRMS (ESI) calcd for $C_{11}H_{14}NO_3^+$ $[M+H]^+$ 208.0968; found 208.0968.



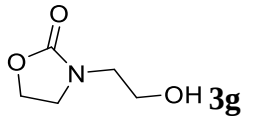
3e

3-(4-Chlorobenzyl)oxazolidin-2-one. Light yellow oil. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.35 (d, *J* = 8.3 Hz, 2H), 7.25 (d, *J* = 8.3 Hz, 2H), 4.42 (s, 2H), 4.33 (t, *J* = 10.0 Hz, 2H), 3.44 (t, *J* = 7.5 Hz, 2H) ppm. ^{13}C NMR (126 MHz, Chloroform-*d*) δ 158.5, 134.3, 133.9, 129.5, 129.0, 61.8, 47.8, 44.0 ppm. These data are matched with the reported publication [3]. HRMS (ESI) calcd for $C_{10}H_{11}ClNO_2^+$ $[M+H]^+$ 212.0473; found 212.1473.



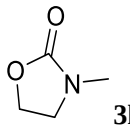
3f

3-(4-Nitrobenzyl)oxazolidin-2-one. Light yellow solid. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.25 (d, *J* = 8.0 Hz, 2H), 7.50 (d, *J* = 8.0 Hz, 2H), 4.56 (s, 2H), 4.40 (t, *J* = 7.8 Hz, 2H), 3.50 (t, *J* = 7.8 Hz, 2H) ppm. ^{13}C NMR (126 MHz, Chloroform-*d*) δ 158.5, 147.8, 143.2, 128.8, 124.1, 61.9, 47.9, 44.3 ppm. These data are matched with the reported publication [6]. HRMS (ESI) calcd for C10H11$N_2O_4^+$ $[M+H]^+$ 223.0713; found 223.0713.



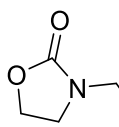
3g

3-(2-Hydroxyethyl)oxazolidin-2-one. Colorless oil. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 4.36 (t, *J* = 7.5 Hz, 2H), 3.81 (t, *J* = 5.1 Hz, 2H), 3.71 (t, *J* = 7.5 Hz, 2H), 3.41 (t, *J* = 5.0 Hz, 2H) ppm. ^{13}C NMR (126 MHz, Chloroform-*d*) δ 159.4, 62.2, 60.5, 46.9, 45.7 ppm. These data are matched with the reported publication.[3] HRMS (ESI) calcd for $C_5H_{10}NO_3^+$ $[M+H]^+$ 132.0655; found 132.0656.



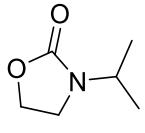
3h

3-Methyl-2-oxazolidinone. Oil. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 4.23 (t, *J* = 8.0 Hz, 2H), 3.50 (t, *J* = 8.0 Hz, 2H), 2.80 (s, 3H) ppm. ^{13}C NMR (126 MHz, Chloroform-*d*) δ 61.5, 46.7, 31.0. These data are matched with the reported publication [7]. HRMS (ESI) calcd for $C_4H_8NO_2^+$ $[M+H]^+$ 102.0553; found 102.0550.



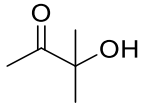
3i

3-Ethyl-2-oxazolidinone. Yellow liquid. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 4.31 (t, *J* = 8.0 Hz, 2H), 3.55 (t, *J* = 8.0 Hz, 2H), 3.31 (q, *J* = 7.2 Hz, 2H), 1.16 (t, *J* = 7.2 Hz, 3H) ppm. ^{13}C NMR (126 MHz, Chloroform-*d*) δ 61.6, 43.9, 38.9, 12.5 ppm. These data are matched with the reported publication [8]. HRMS (ESI) calcd for $C_5H_{10}NO_2^+$ $[M+H]^+$ 116.0708; found 116.0706.



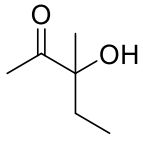
3j

3-Isopropyl-2-oxazolidinone. Yellow liquid. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 4.31 (t, *J* = 8.0 Hz, 2H), 4.11 (hept, *J* = 6.8 Hz, 1H), 3.51 (t, *J* = 8.0 Hz, 2H), 1.19 (d, *J* = 6.8 Hz, 6H) ppm. ^{13}C NMR (126 MHz, Chloroform-*d*) δ 61.9, 44.8, 39.6, 19.7 ppm. These data are matched with the reported publication [8]. HRMS (ESI) calcd for $C_6H_{12}NO_2^+$ $[M+H]^+$ 130.0863; found 130.0863.



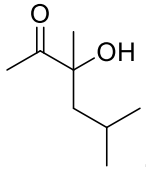
4a

3-Hydroxy-3-methylbutan-2-one. Colorless oil. ^{1}H NMR (500 MHz, DMSO-d_6) δ 5.22 (s, 1H), 2.16 (s, 3H), 1.18 (s, 6H) ppm. ^{13}C NMR (126 MHz, DMSO-d_6) δ 214.3, 76.2, 26.6, 24.7 ppm. These data are matched with the reported publication [3].



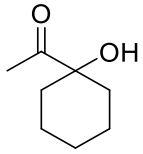
4b

3-Hydroxy-3-methylpentan-2-one. Colorless oil. ^{1}H NMR (500 MHz, DMSO-d_6) δ 5.04 (s, 1H), 2.14 (s, 3H), 1.61 (m, 1H), 1.47 (m, 1H), 1.13 (s, 3H), 0.76 (t, *J* = 7.5 Hz, 3H) ppm. ^{13}C NMR (126 MHz, DMSO-d_6) δ 214.5, 78.9, 32.2, 25.5, 24.5, 8.3. These data are matched with the reported publication [3].



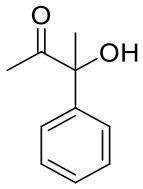
4c

3-Hydroxy-3-dimethyl-2-hexanone. ^{1}H NMR (500 MHz, DMSO-d_6) δ 4.99 (s, 1H), 2.17 (s, 3H), 1.59 (dd, *J* = 13.9, 6.9 Hz, 1H), 1.42 (dd, *J* = 13.9, 5.6 Hz, 1H), 1.15 (s, 3H), 0.87 (d, *J* = 6.6 Hz, 3H), 0.79 (d, *J* = 6.6 Hz, 3H) ppm. ^{13}C NMR (126 MHz, DMSO-d_6) δ 214.8, 79.0, 48.0, 26.4, 25.6, 24.7, 24.2 ppm. These data are matched with the reported publication [9].



4d

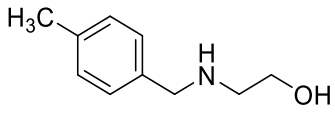
1-(1-Hydroxycyclohexyl)ethenone. Yellow oil. ^{1}H NMR (500 MHz, DMSO-d_6) δ 5.06 (s, 1H), 2.14 (s, 3H), 1.60–1.42 (m, 9H), 1.18 (m, 1H) ppm. ^{13}C NMR (126 MHz, DMSO-d_6) δ 214.6, 77.3, 33.3, 25.5, 24.9, 21.3 ppm. These data are matched with the reported publication [3].



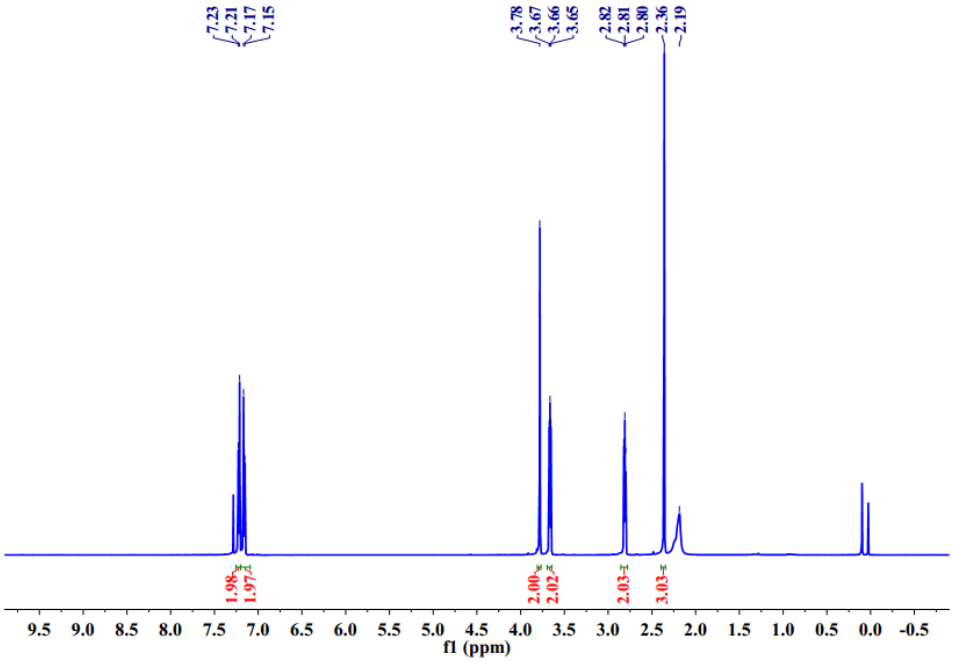
4e

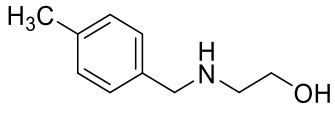
3-Hydroxy-3-phenylbutan-2-one. Brown oil. ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.44 (d, J = 7.2 Hz, 2H), 7.35 (t, J = 7.6 Hz, 2H), 7.27 (t, J = 7.3 Hz, 1H), 6.04 (s, 1H), 2.02 (s, 3H), 1.52 (s, 3H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 210.7, 143.7, 128.6, 127.6, 125.5, 80.2, 26.5, 24.7 ppm. These data are matched with the reported publication [3].

7. NMR Spectral Copies of the Substrates and Products

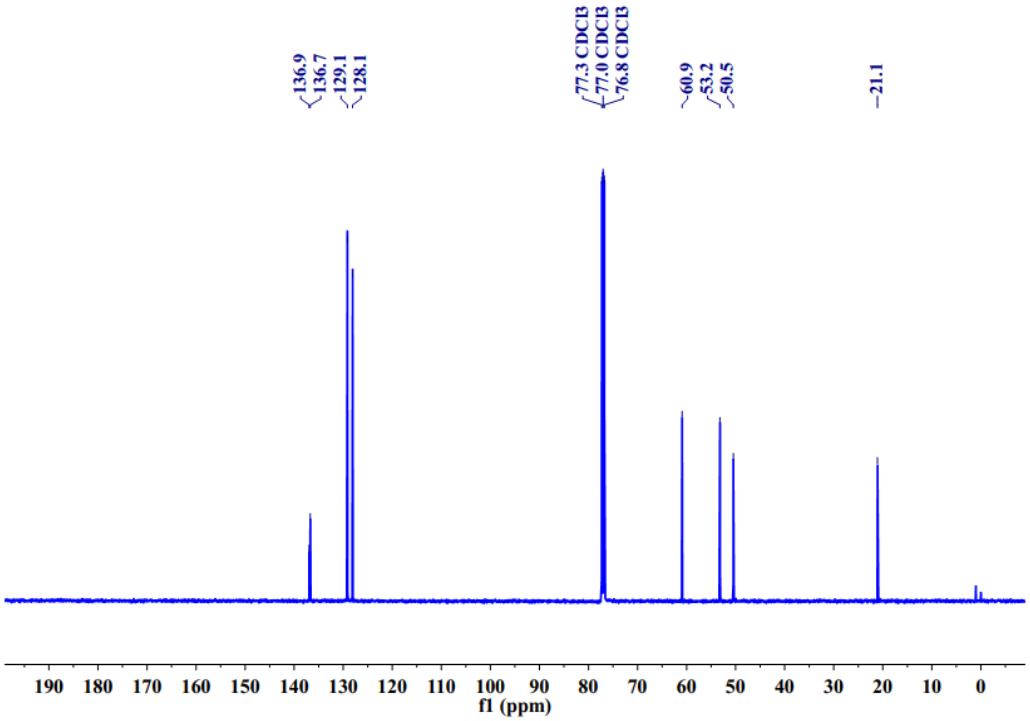


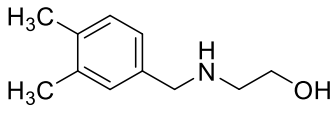
1b ^{1}H NMR (500 MHz, $CDCl_3$)



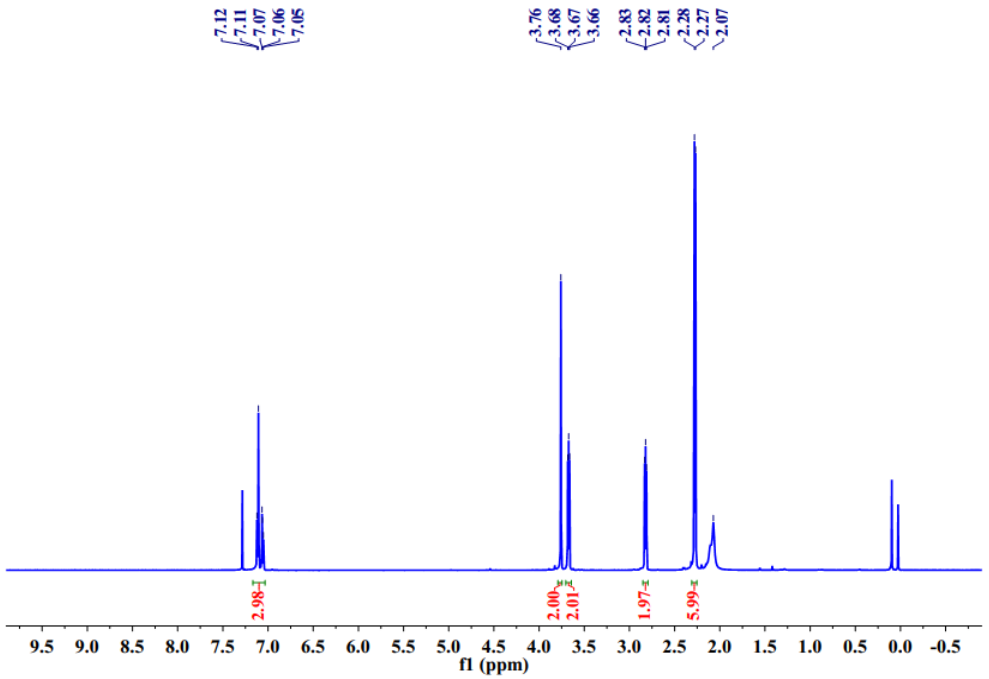


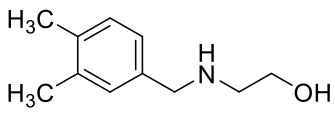
1b ^{13}C NMR (126 MHz, $CDCl_3$)



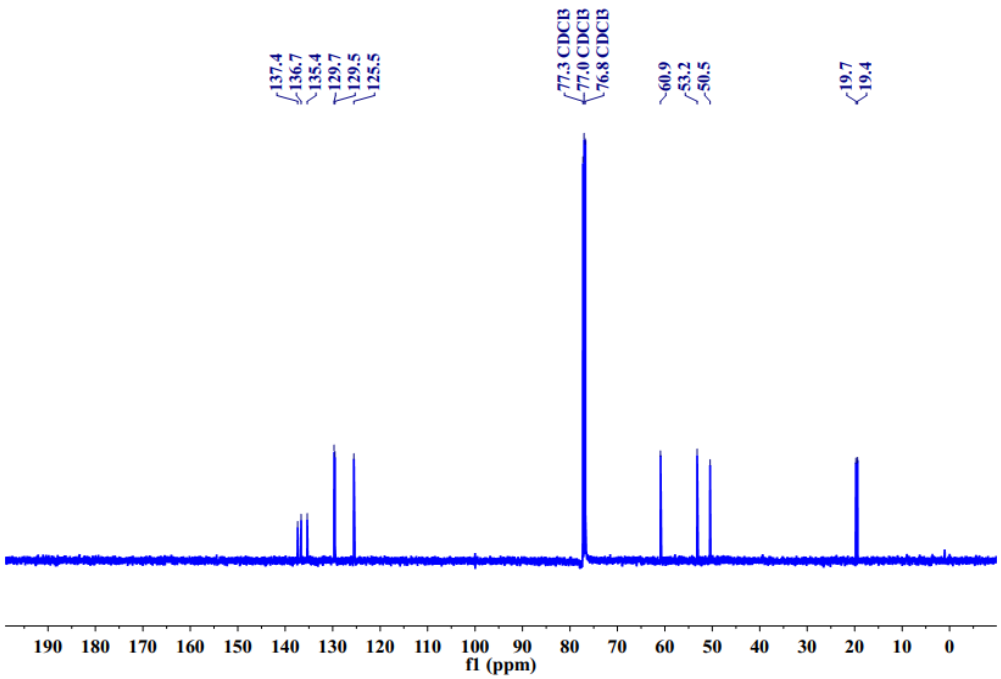


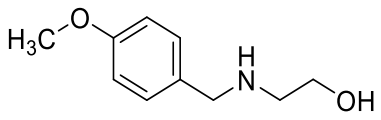
1c ^{1}H NMR (500 MHz, $CDCl_3$)



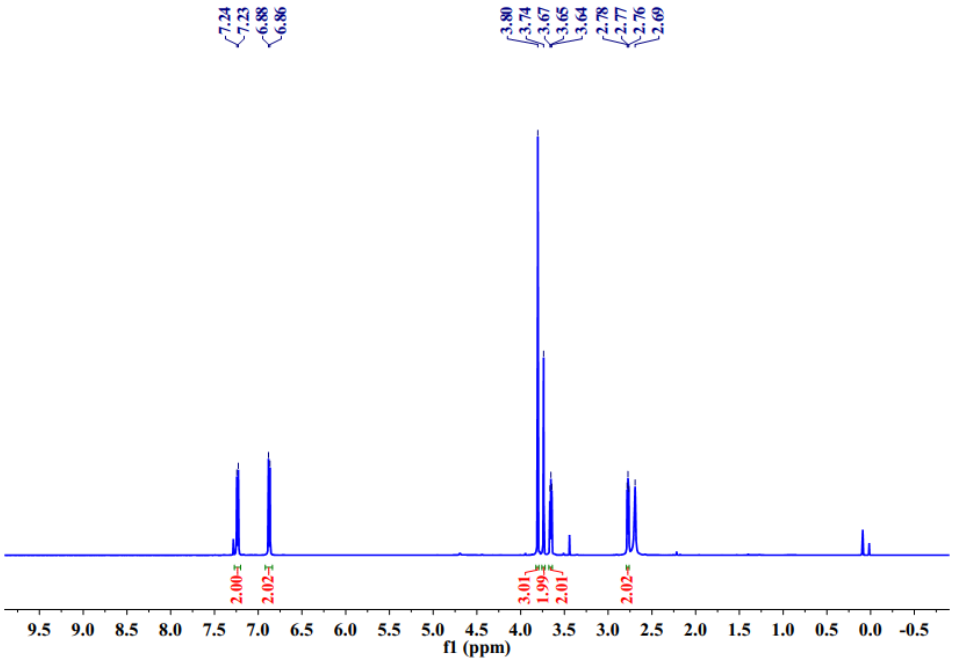


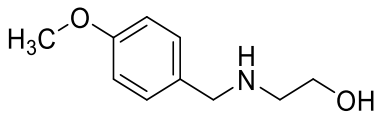
1c ^{13}C NMR (126 MHz, $CDCl_3$)



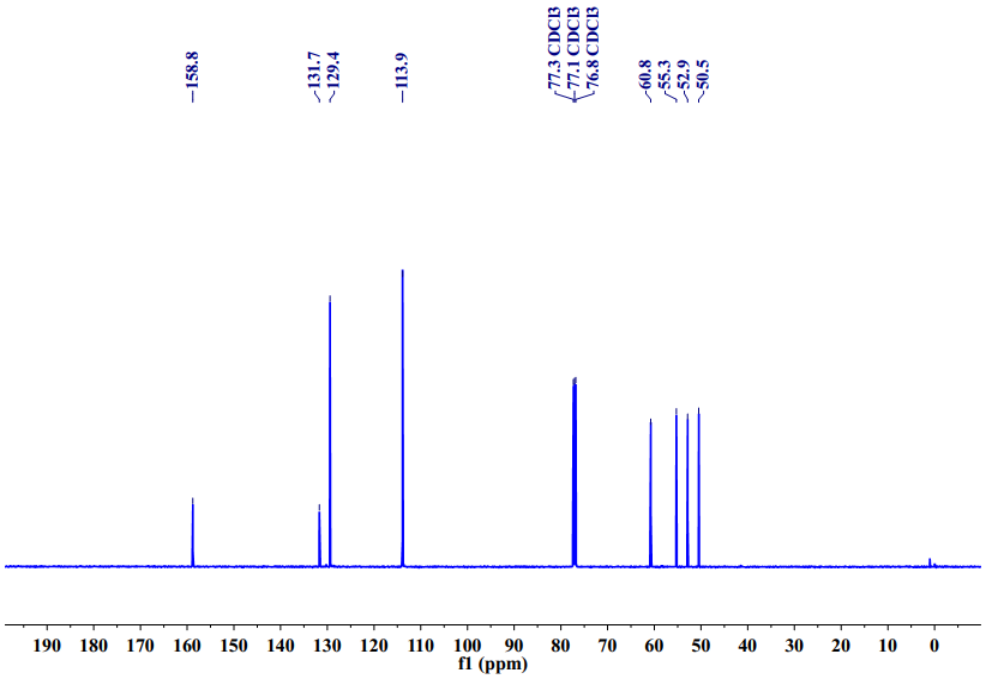


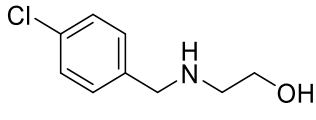
1d ^{1}H NMR (500 MHz, $CDCl_3$)



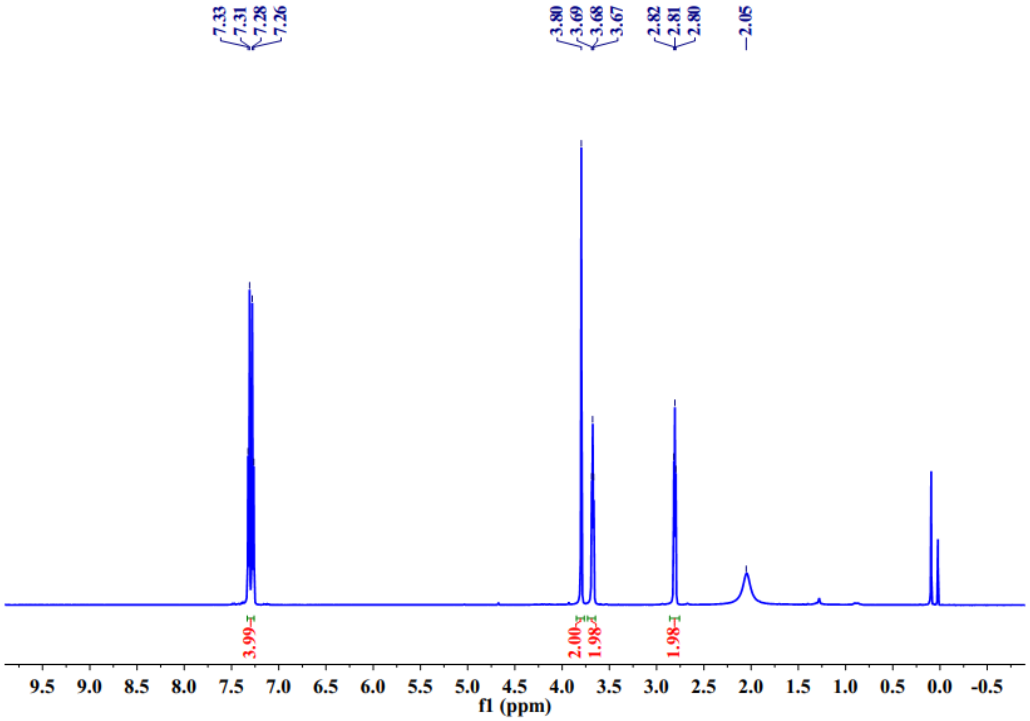


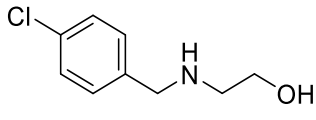
1d ^{13}C NMR (126 MHz, $CDCl_3$)



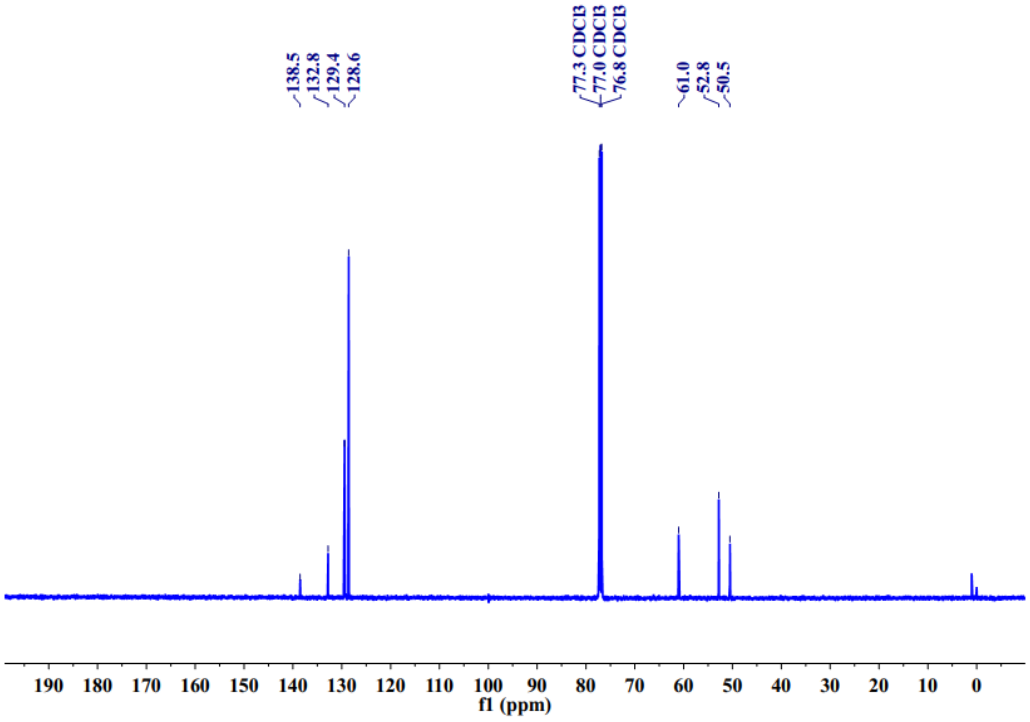


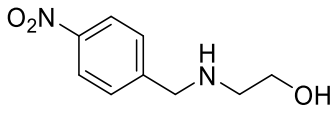
1e ^{1}H NMR (500 MHz, $CDCl_3$)



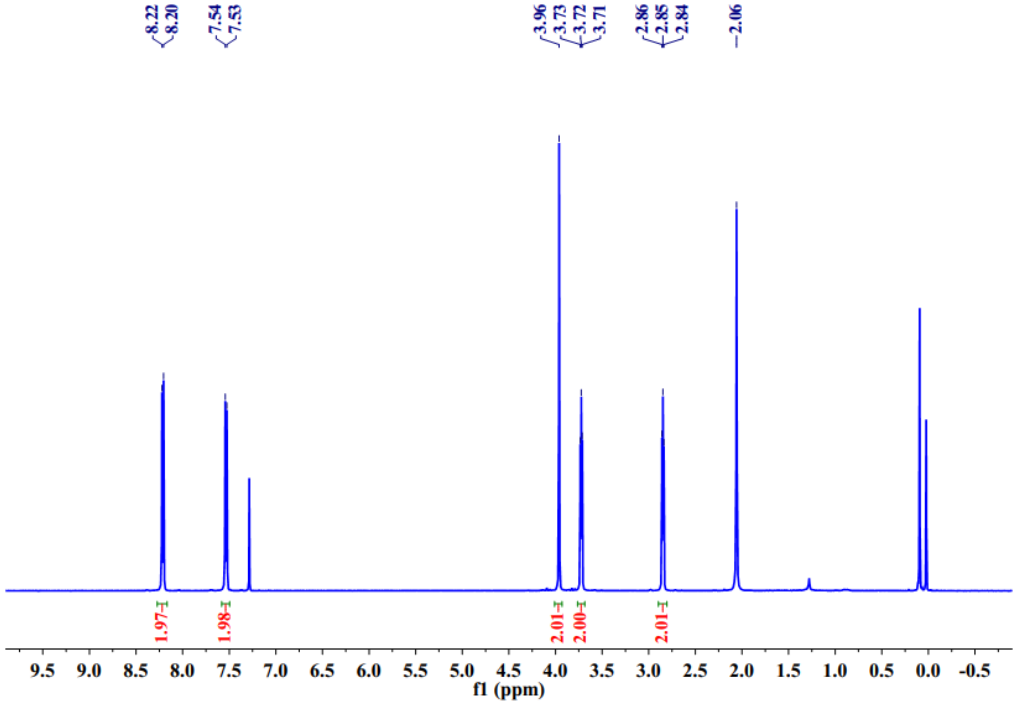


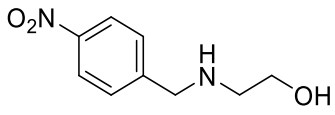
1e ^{13}C NMR (126 MHz, $CDCl_3$)



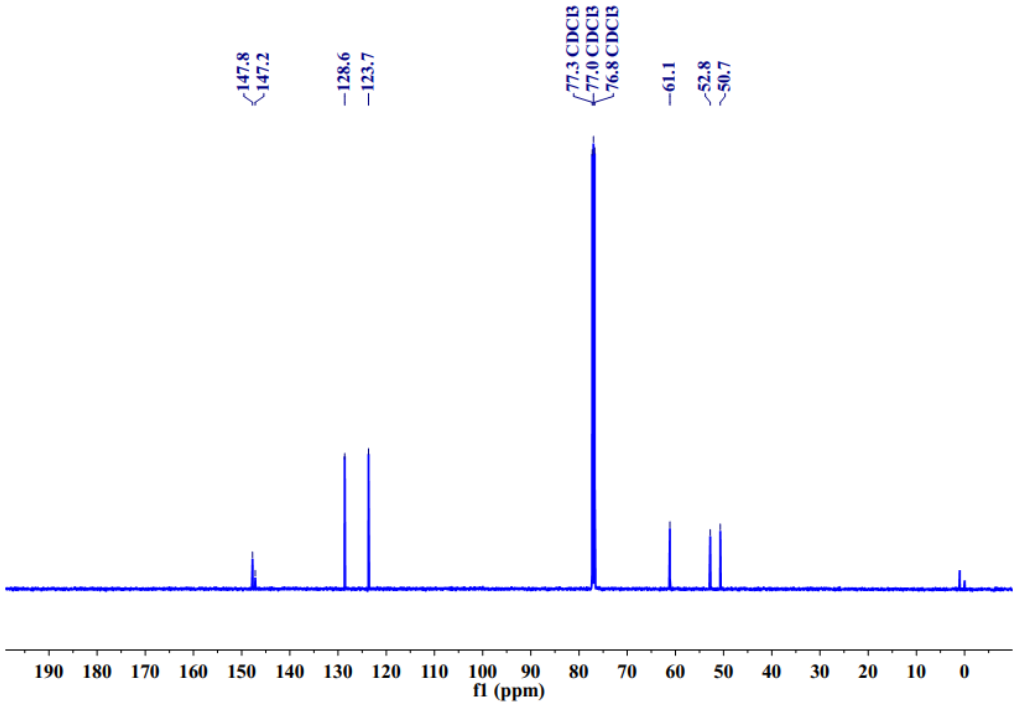


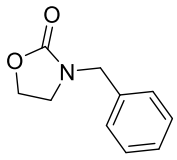
1f ^{1}H NMR (500 MHz, $CDCl_3$)



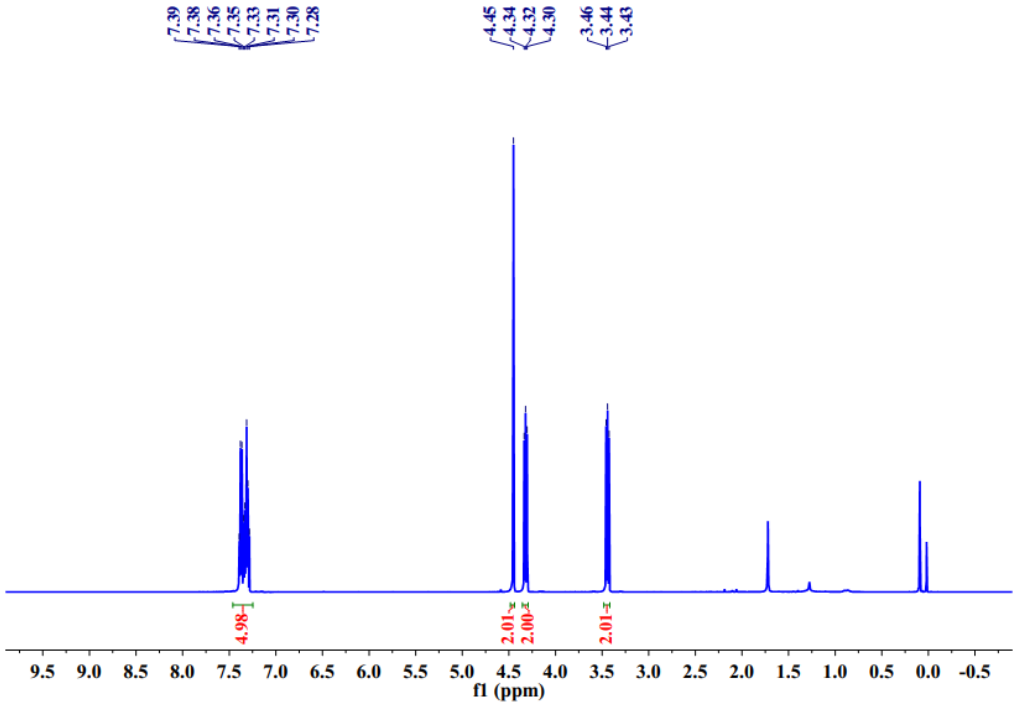


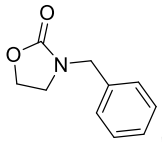
1f ^{13}C NMR (126 MHz, $CDCl_3$)



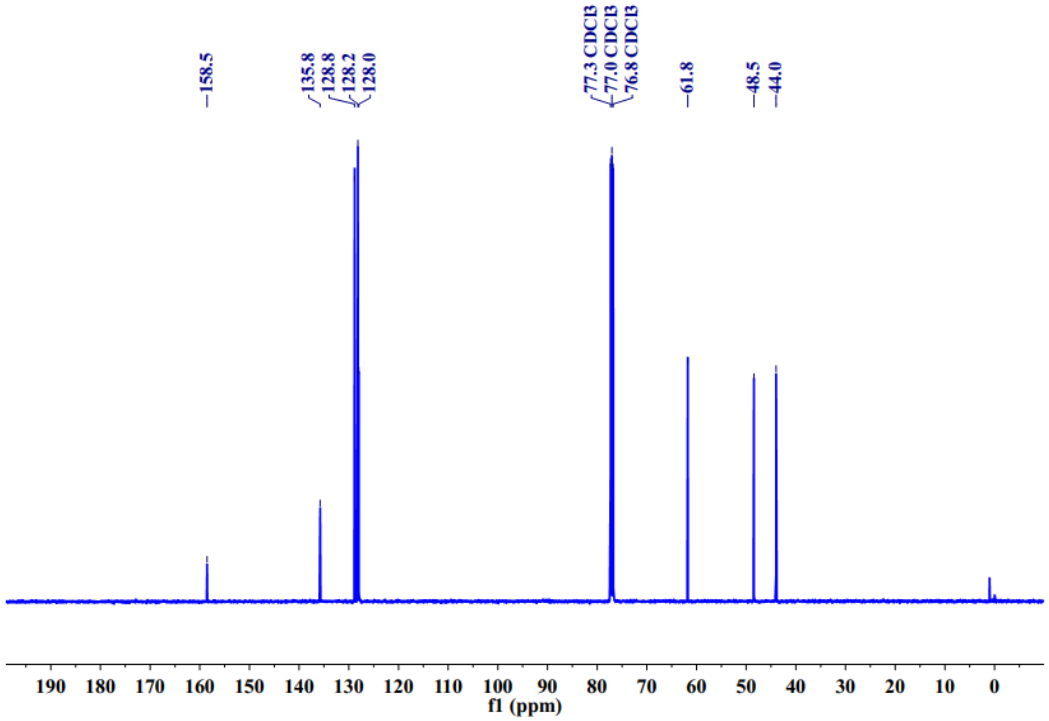


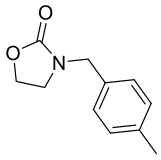
3a ^{1}H NMR (500 MHz, $CDCl_3$)



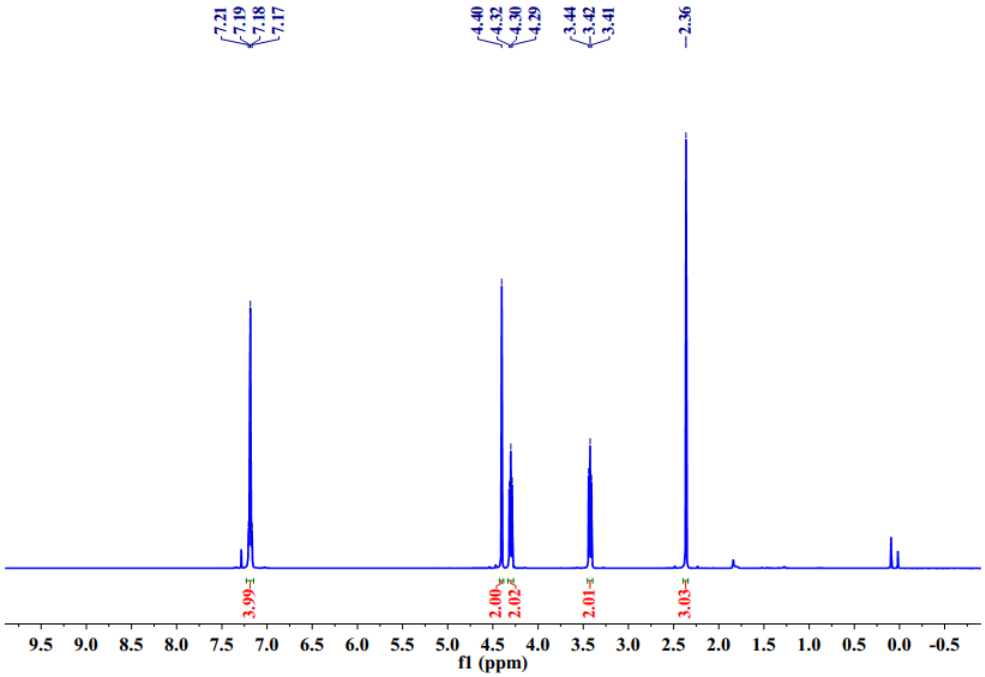


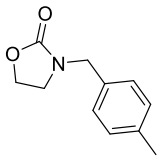
3a ^{13}C NMR (126 MHz, $CDCl_3$)



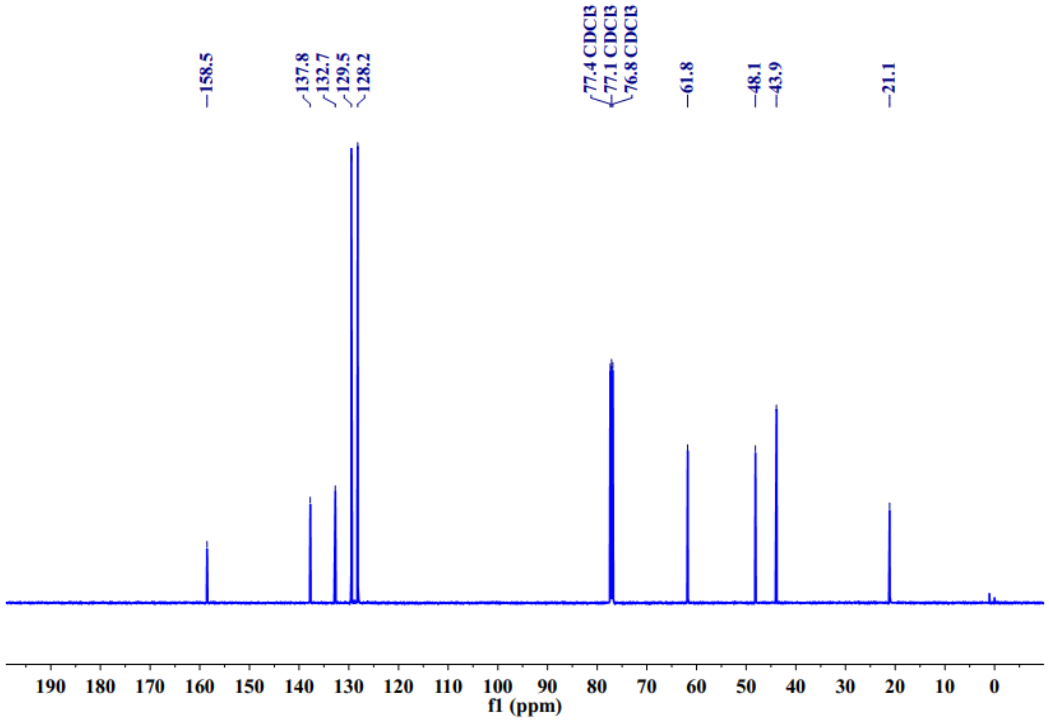


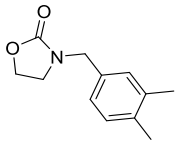
3b ^{1}H NMR (500 MHz, $CDCl_3$)



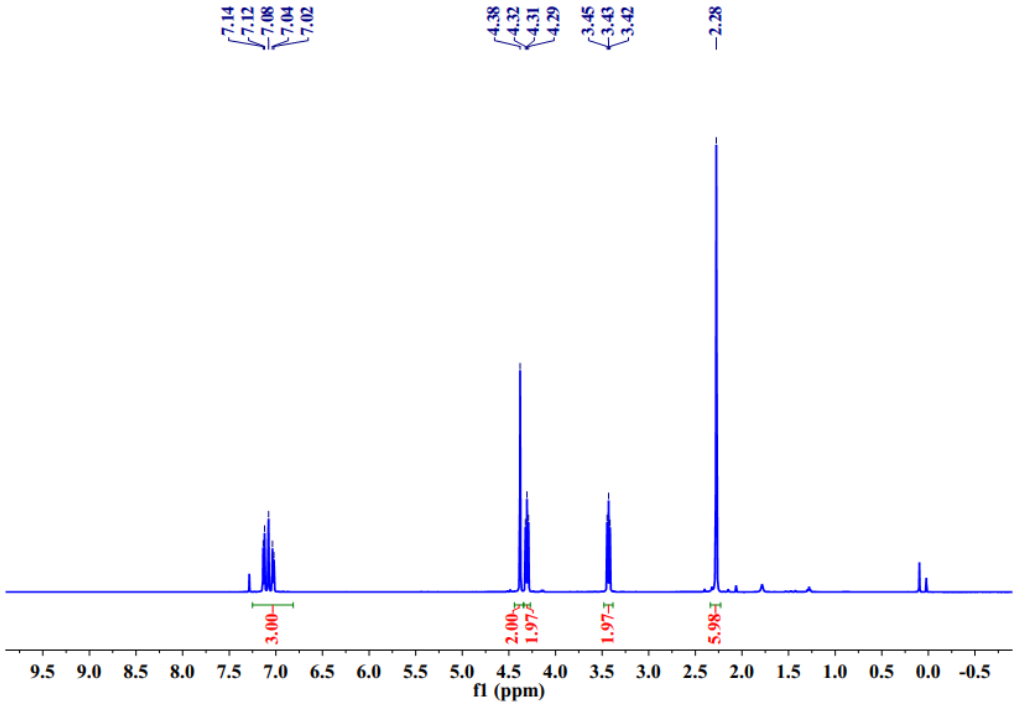


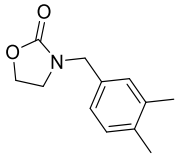
3b ^{13}C NMR (126 MHz, $CDCl_3$)



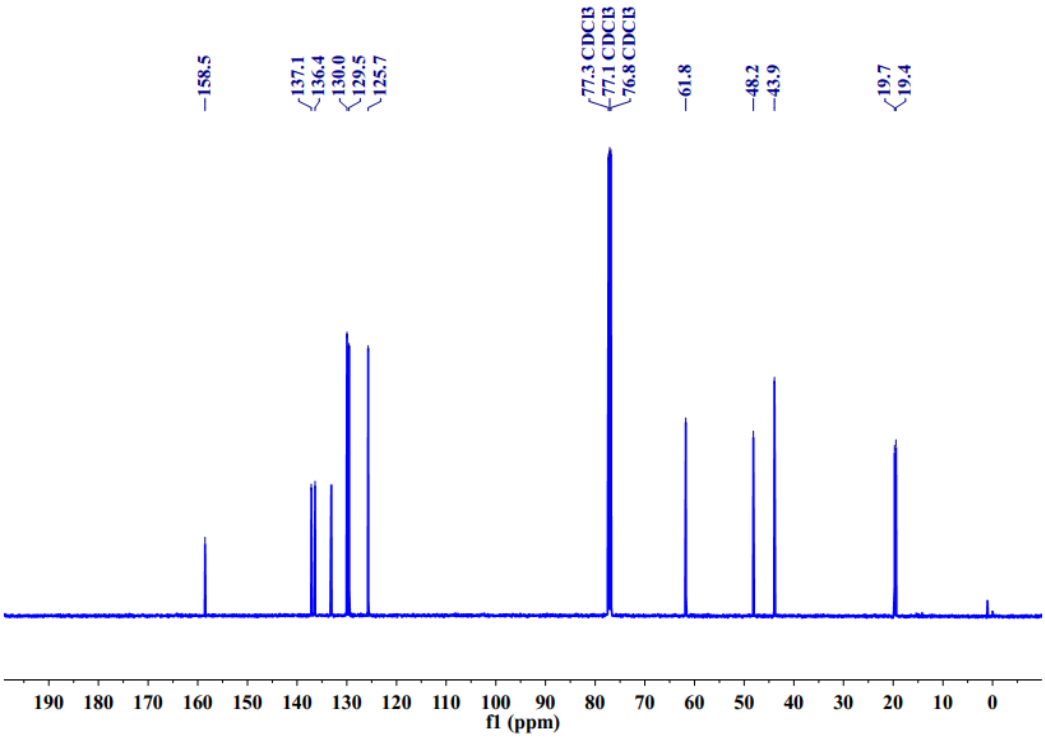


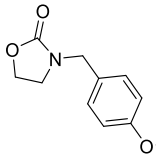
3c ^{1}H NMR (500 MHz, $CDCl_3$)



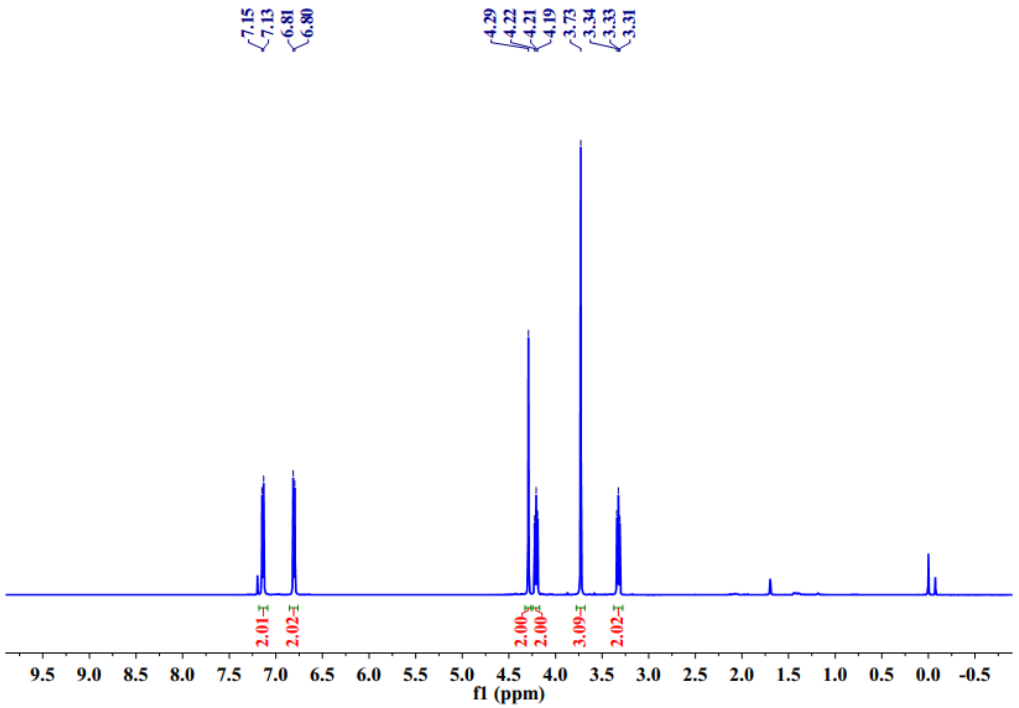


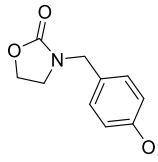
3c ^{13}C NMR (126 MHz, $CDCl_3$)



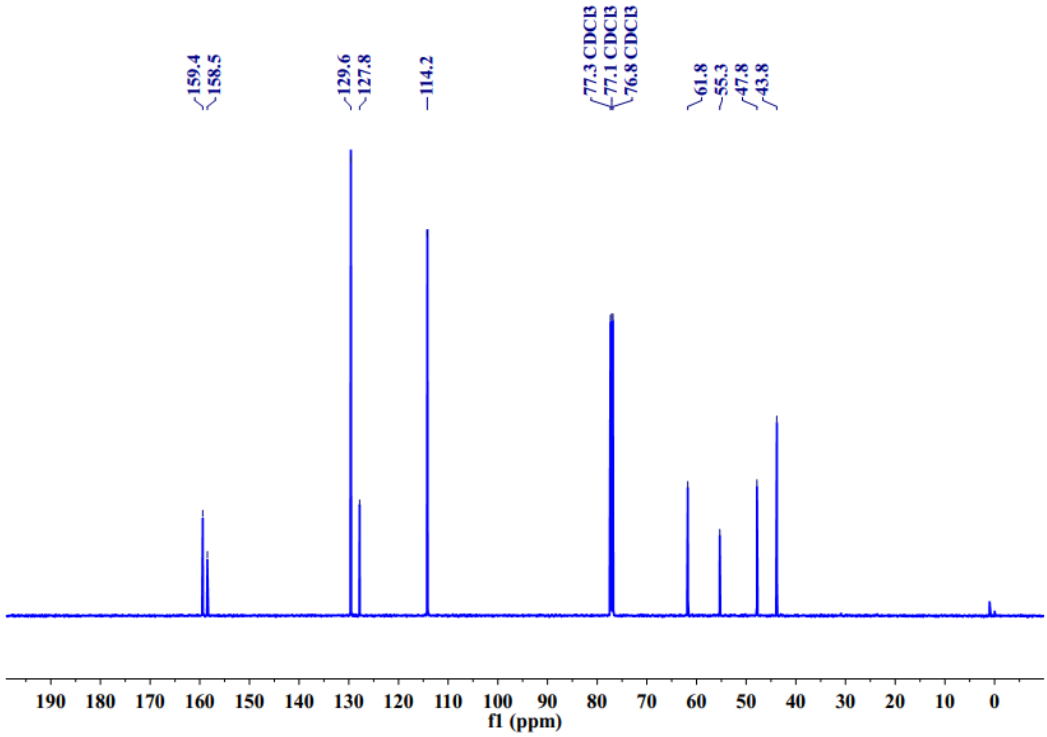


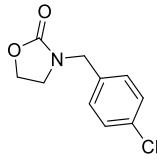
3d ^{1}H NMR (500 MHz, $CDCl_3$)



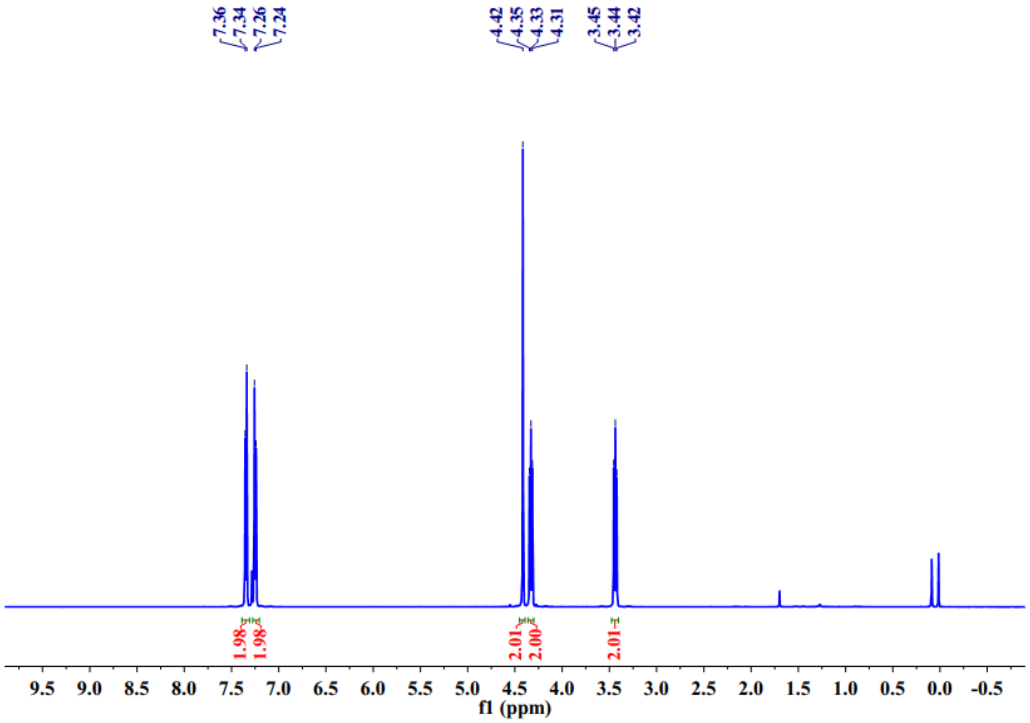


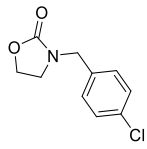
3d ^{13}C NMR (126 MHz, $CDCl_3$)



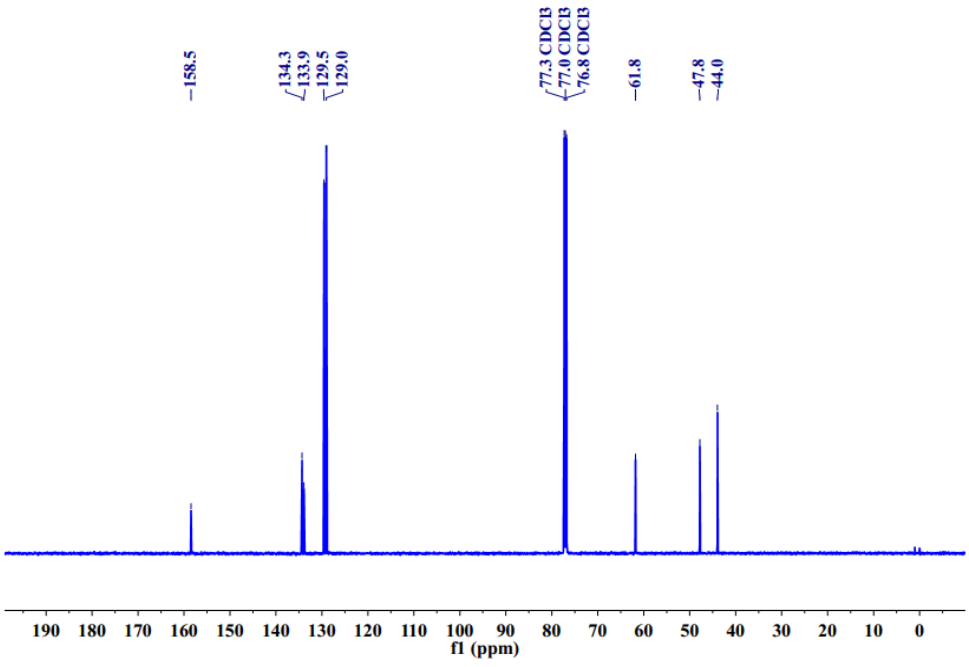


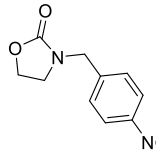
3e ^{1}H NMR (500 MHz, $CDCl_3$)



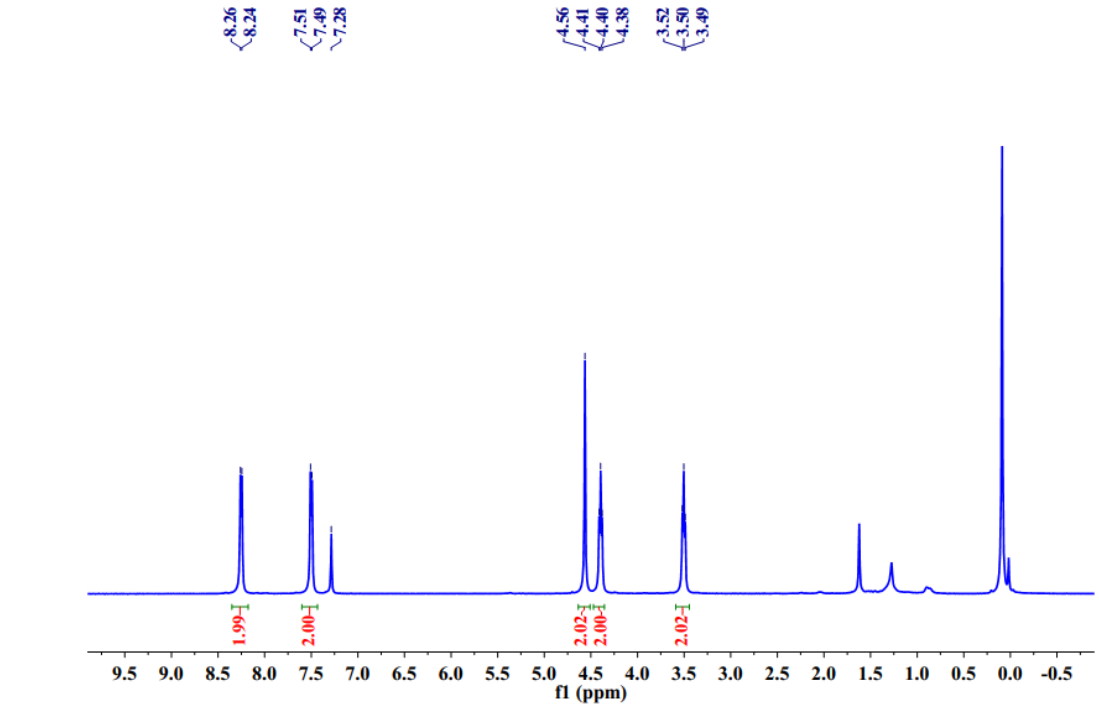


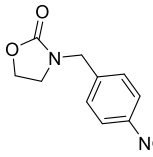
3e ^{13}C NMR (126 MHz, $CDCl_3$)



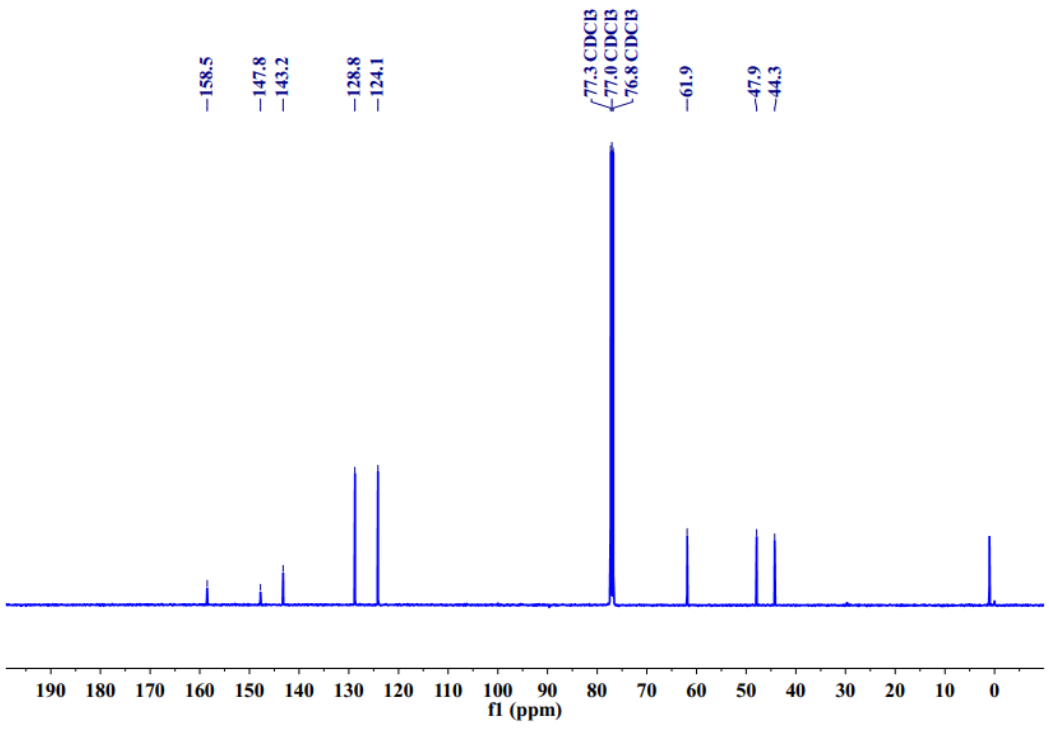


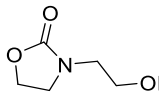
3f ^{1}H NMR (500 MHz, $CDCl_3$)



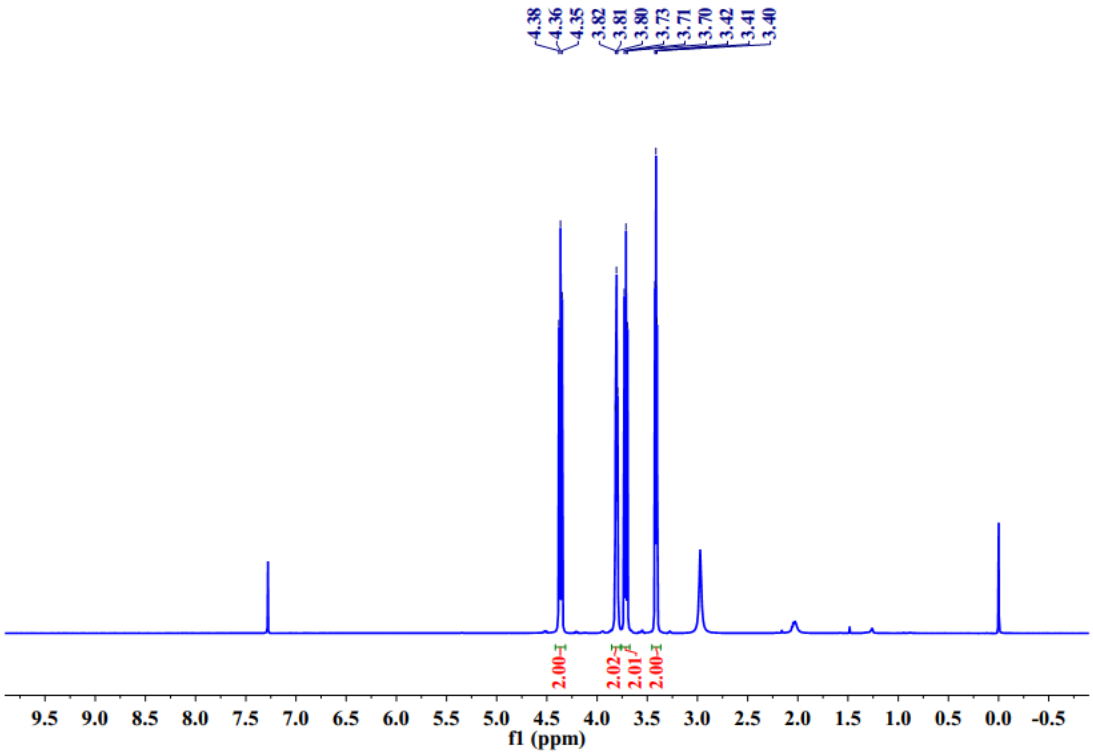


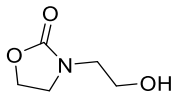
3f ^{13}C NMR (126 MHz, $CDCl_3$)



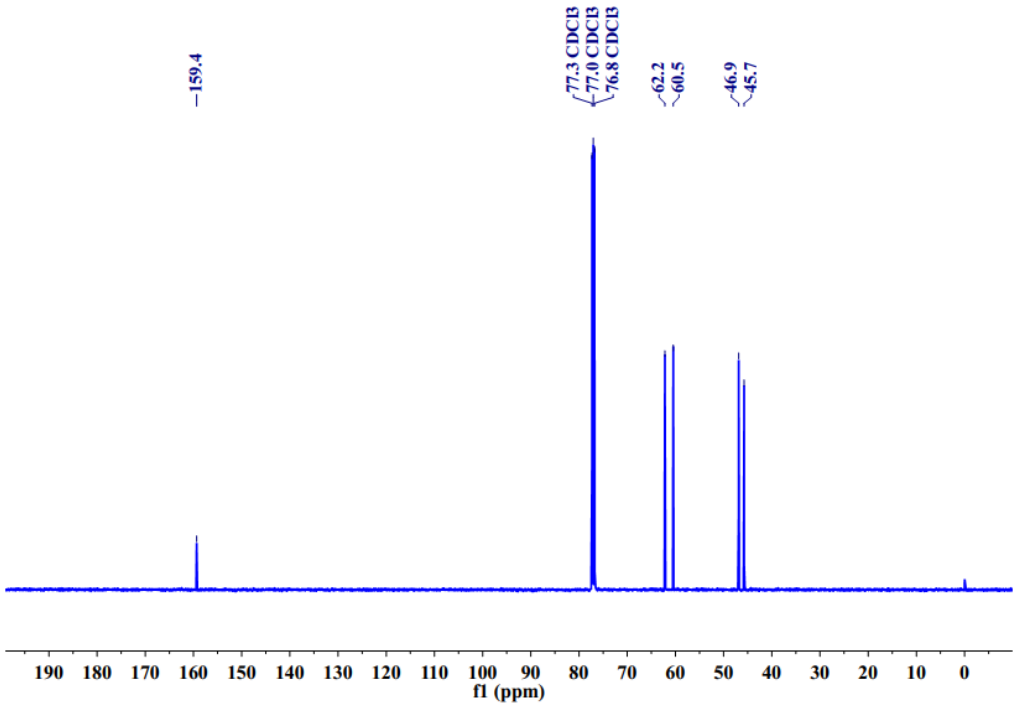


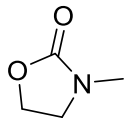
3g ^{1}H NMR (500 MHz, $CDCl_3$)



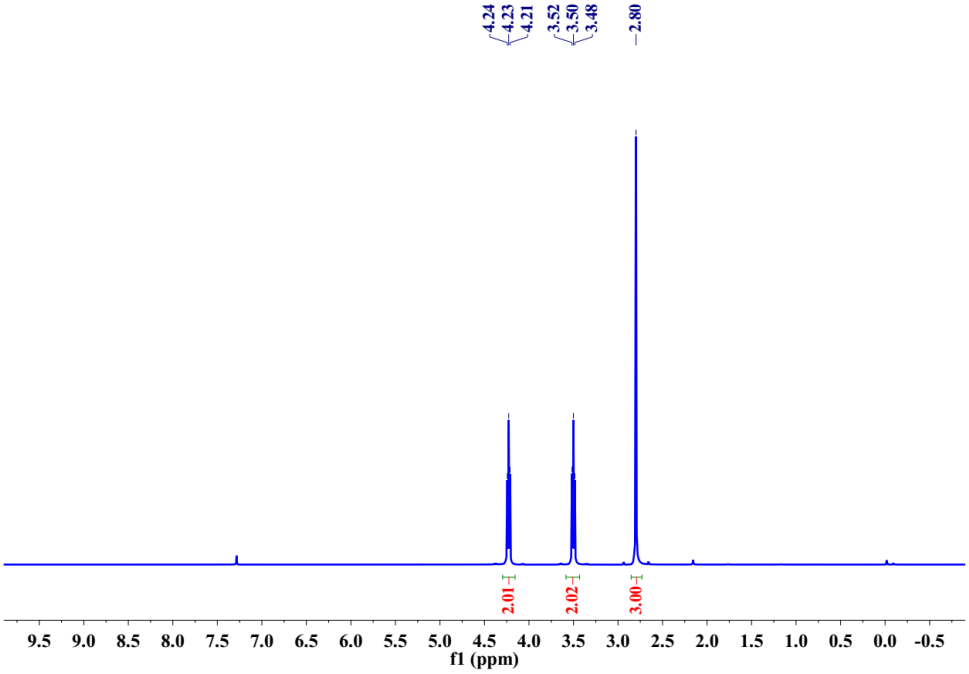


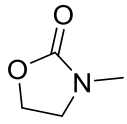
3g ^{13}C NMR (126 MHz, $CDCl_3$)



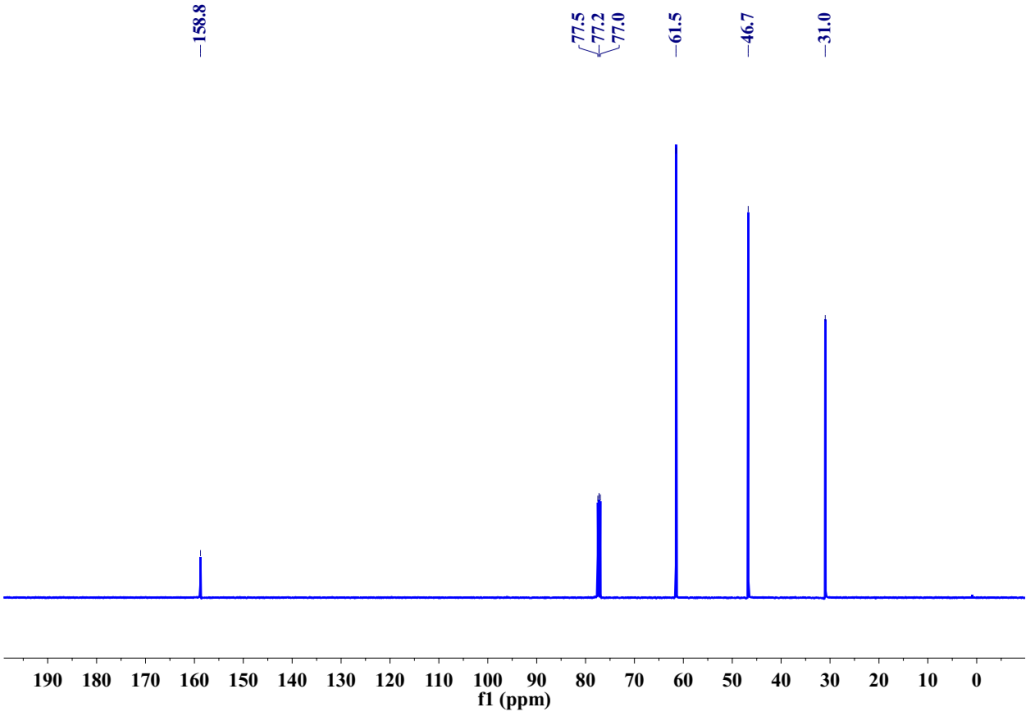


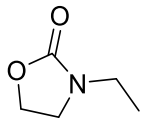
3h ^{1}H NMR (500 MHz, $CDCl_3$)



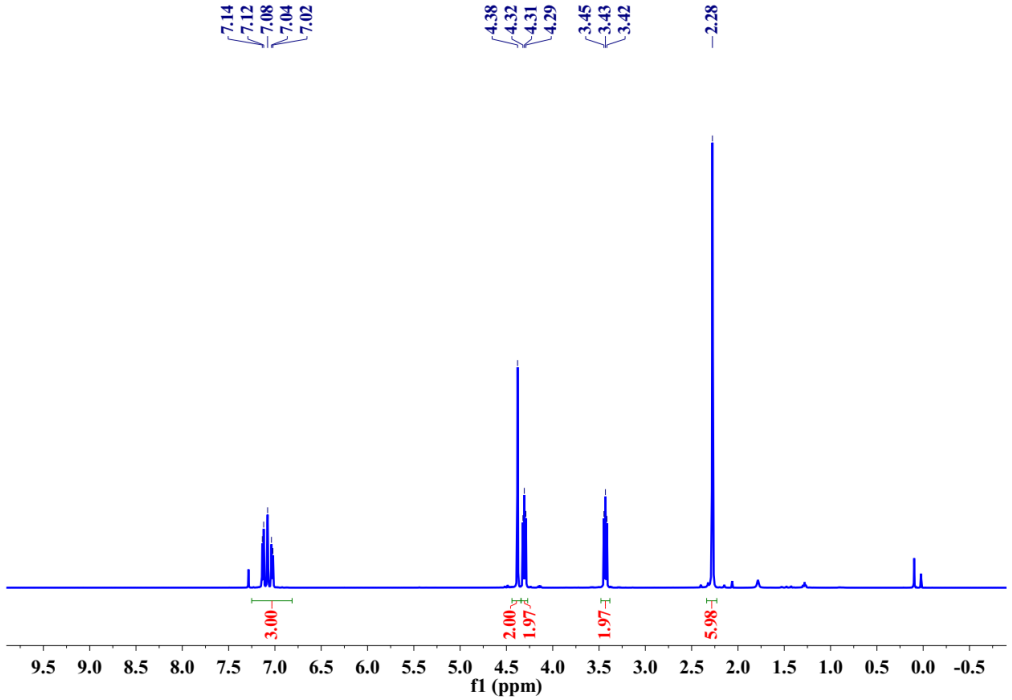


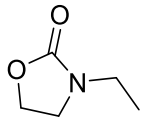
3h ^{13}C NMR (126 MHz, $CDCl_3$)



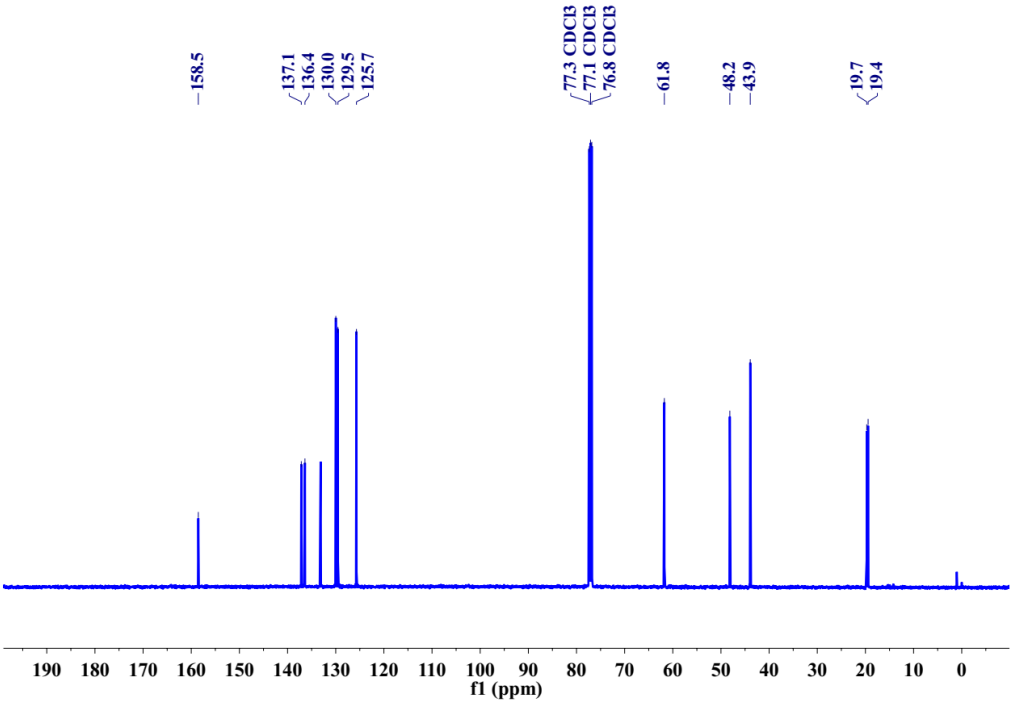


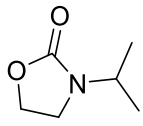
3i ^{1}H NMR (500 MHz, $CDCl_3$)



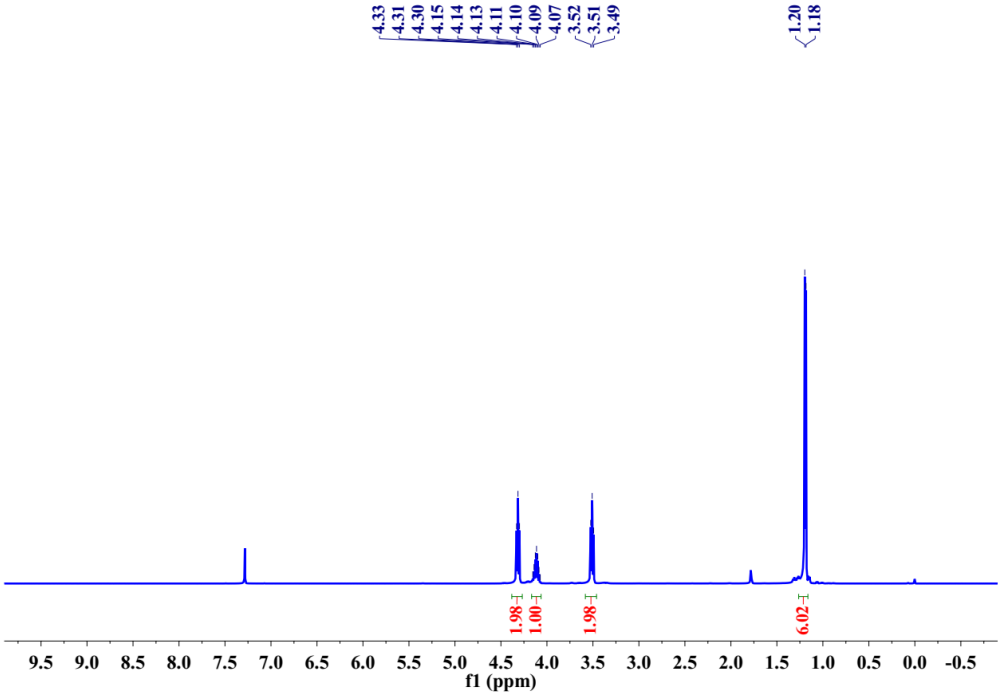


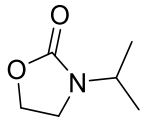
3i ^{13}C NMR (126 MHz, $CDCl_3$)



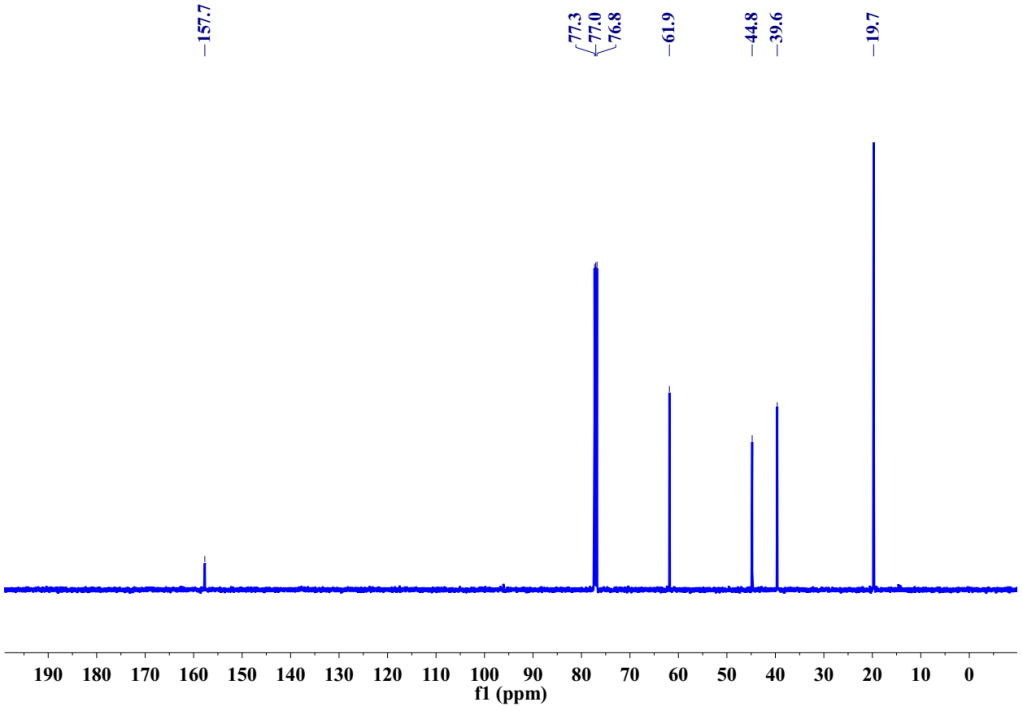


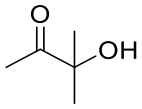
3j ^{1}H NMR (500 MHz, $CDCl_3$)



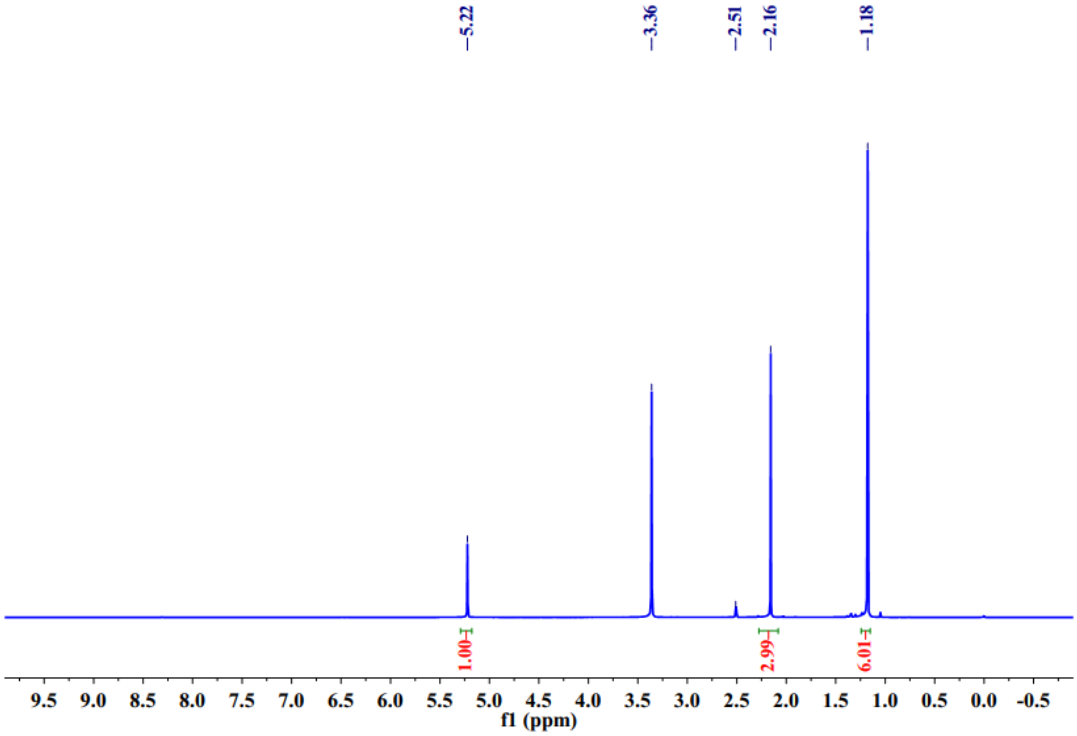


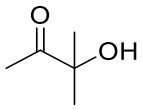
3j ^{13}C NMR (126 MHz, $CDCl_3$)



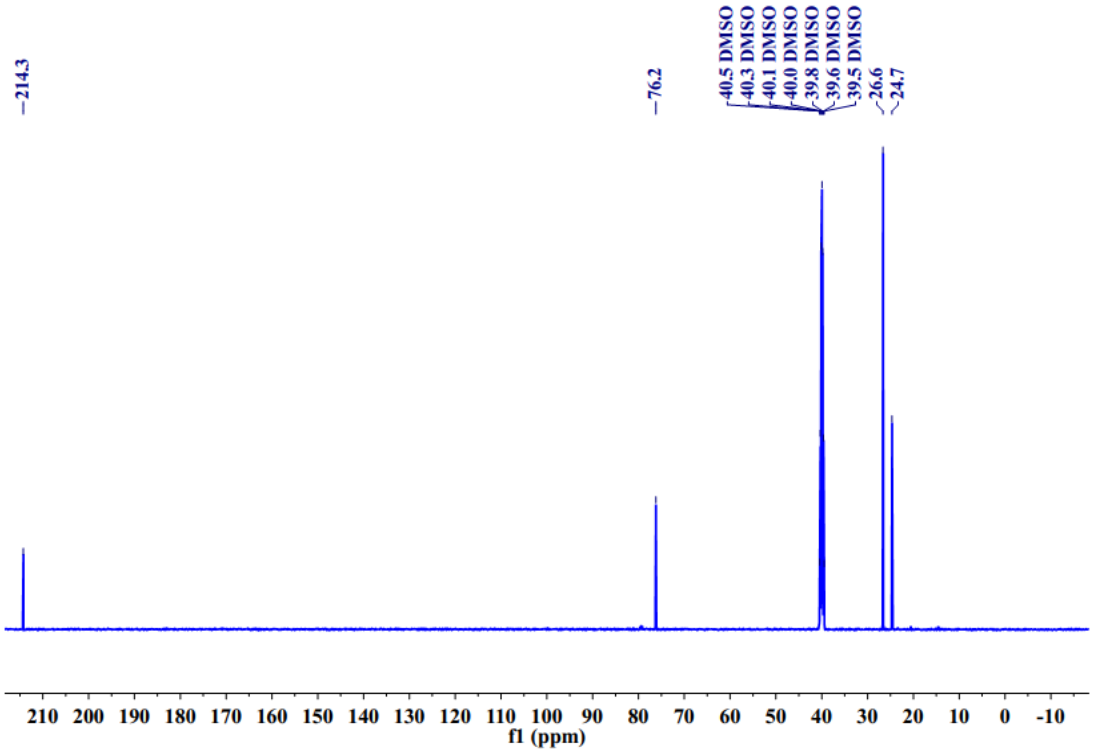


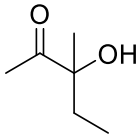
4a ^{1}H NMR (500 MHz, DMSO-*d*$_6$)



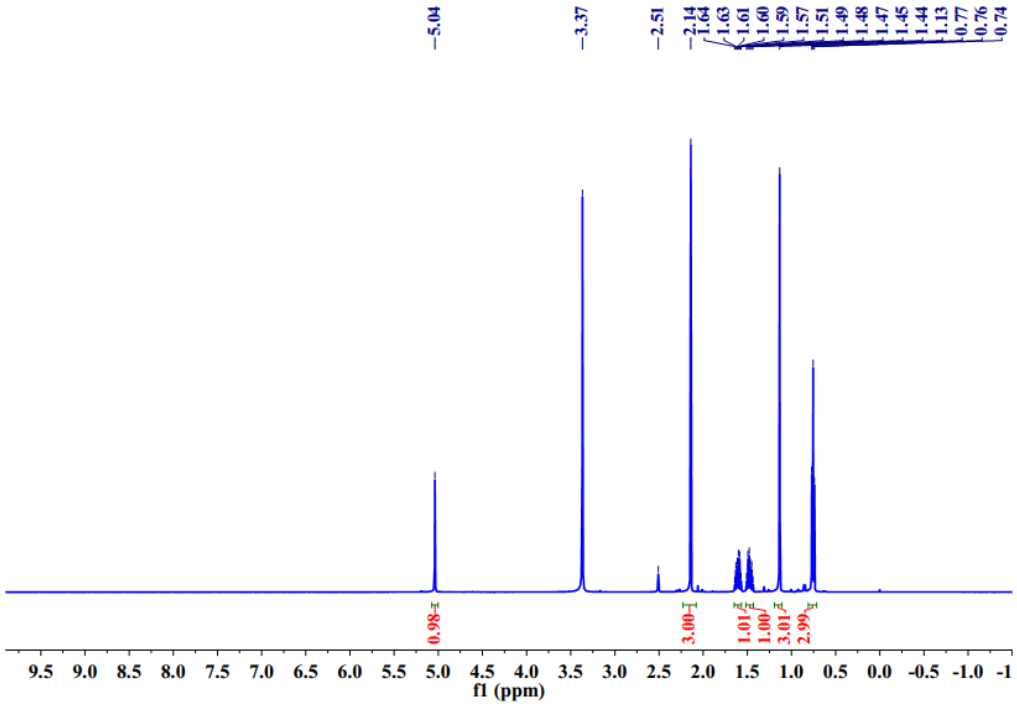


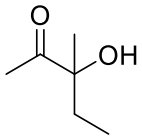
4a ^{13}C NMR (126 MHz, DMSO-*d*$_6$)



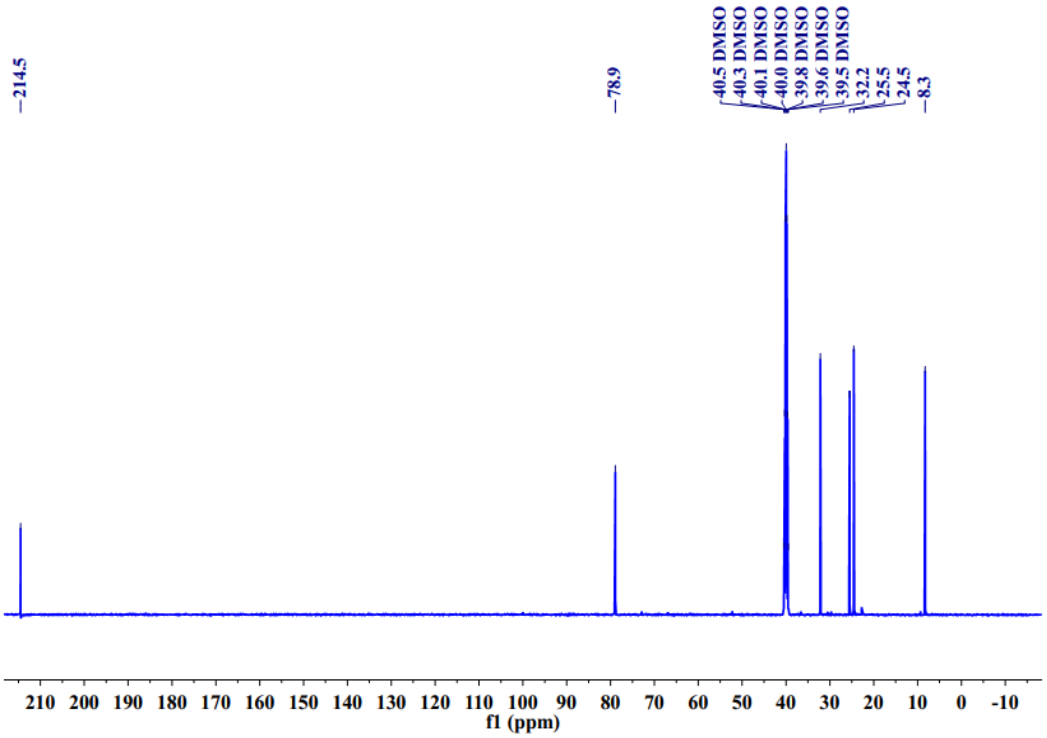


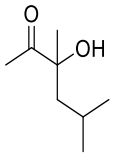
4b ^{1}H NMR (500 MHz, DMSO-d_6)



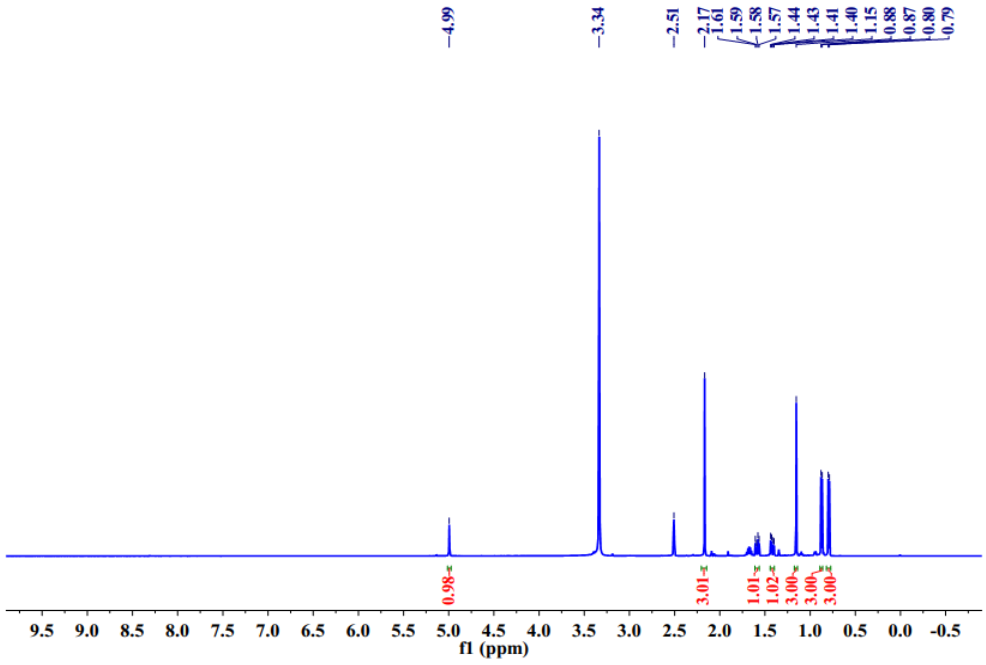


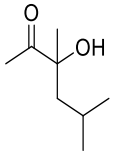
4b ^{13}C NMR (126 MHz, DMSO-d_6)



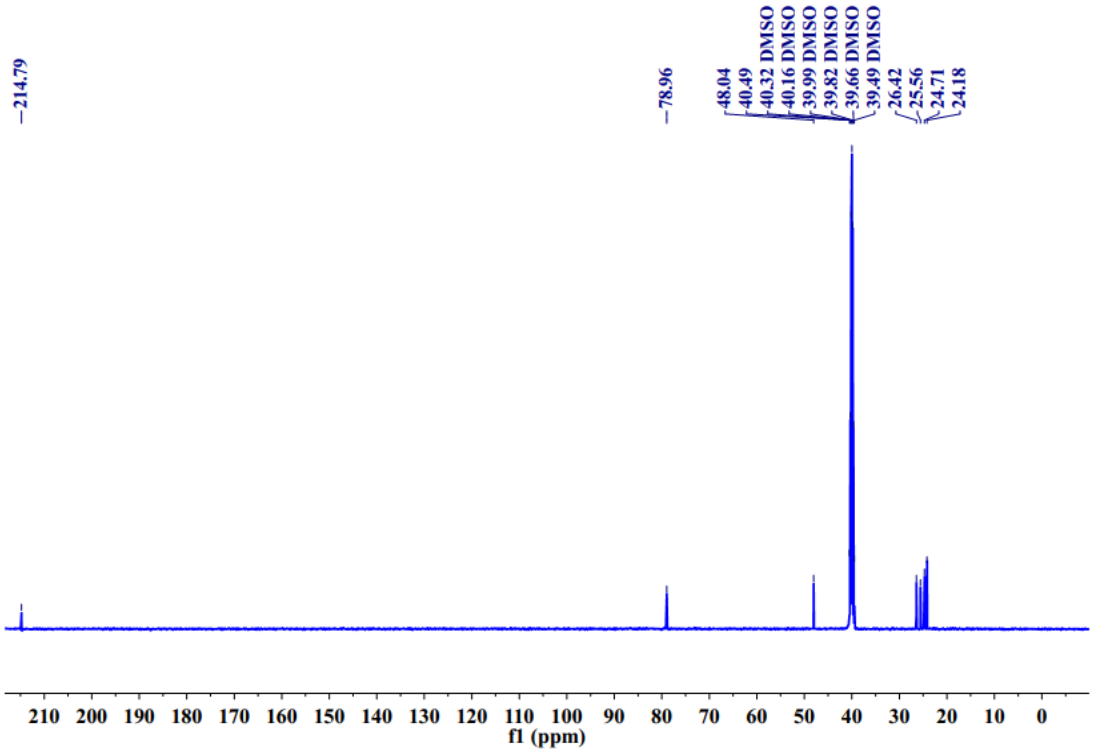


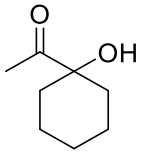
4c ^{1}H NMR (500 MHz, DMSO-*d*$_6$)



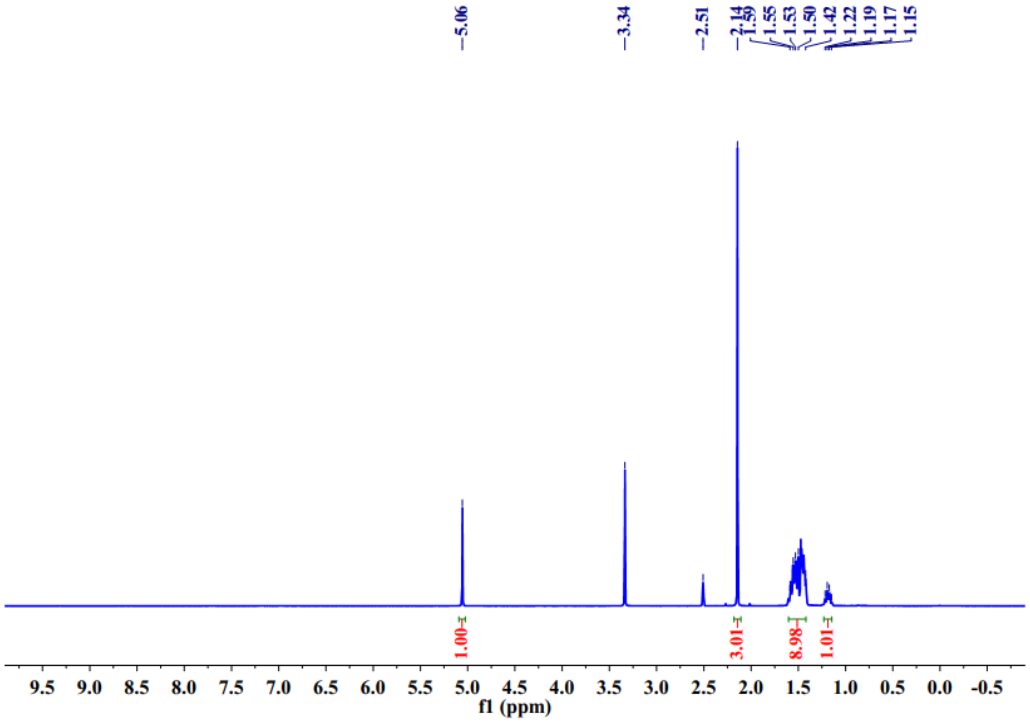


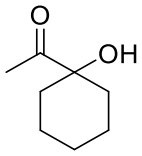
4c ^{13}C NMR (126 MHz, DMSO-*d*$_6$)



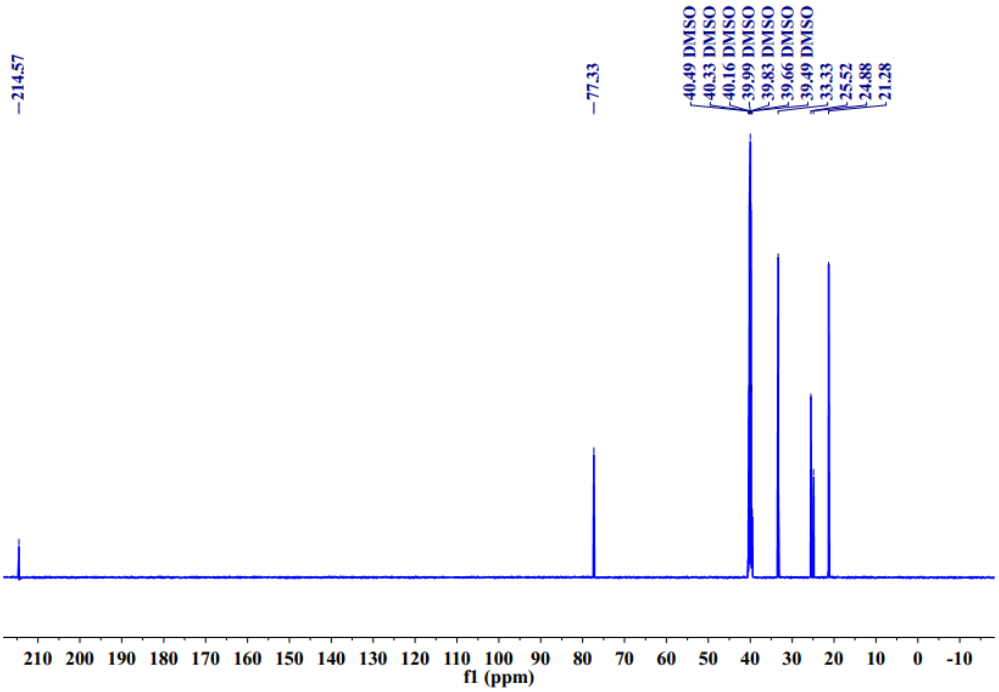


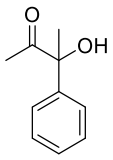
4d ^{1}H NMR (500 MHz, DMSO-d_6)





4d ^{13}C NMR (126 MHz, DMSO-*d*$_6$)





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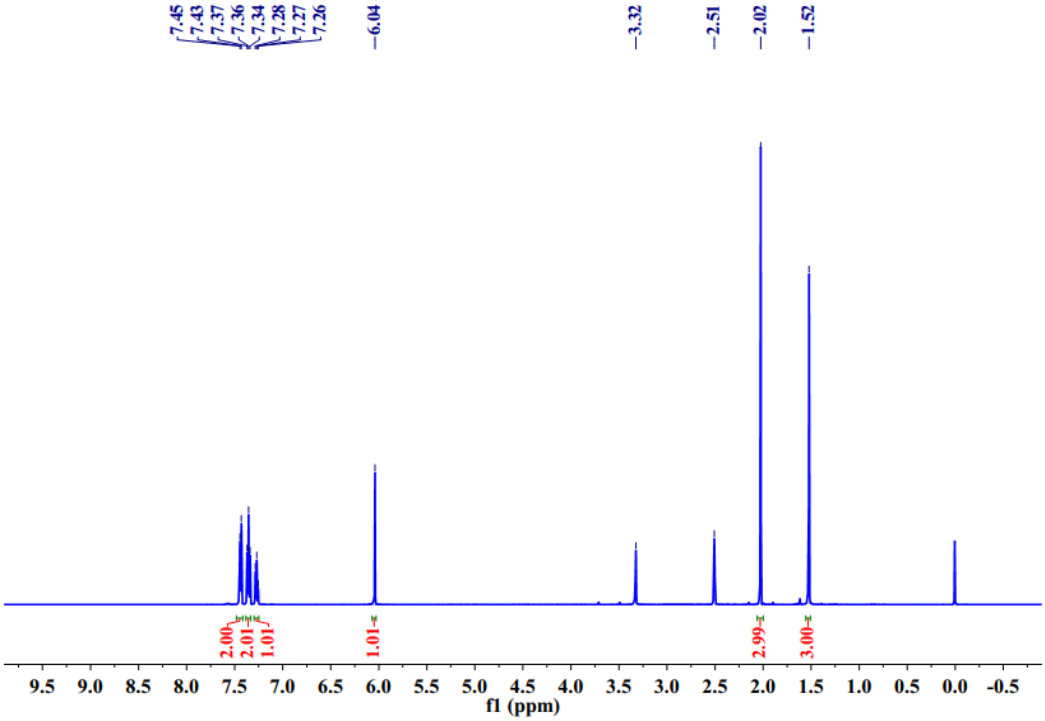

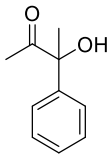

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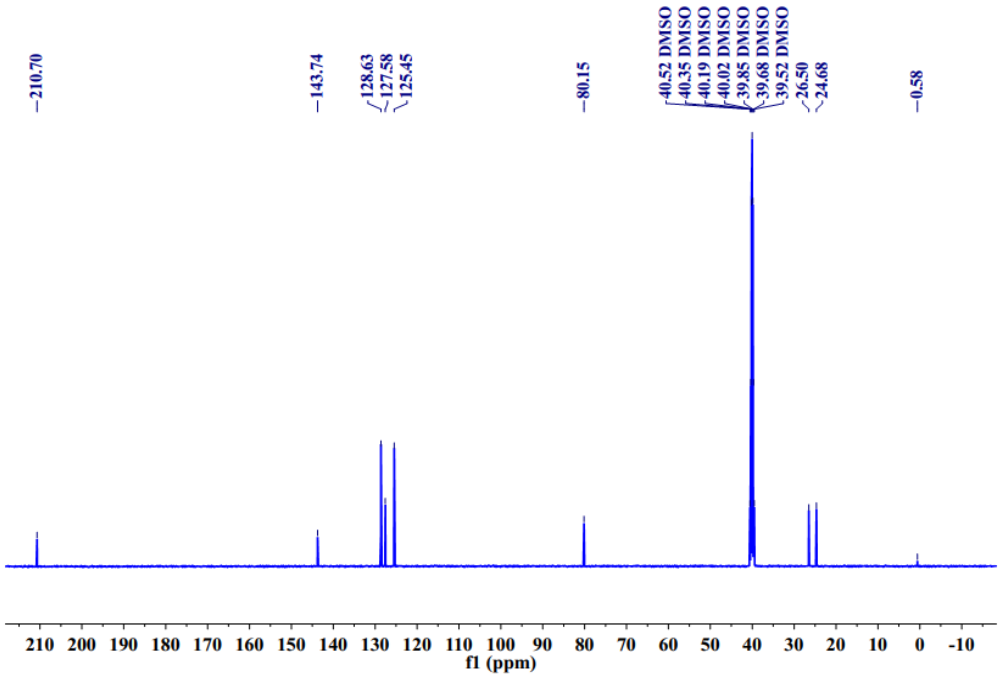

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