Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Atom-economic amide synthesis by iron-substituted polyoxometalate catalyst

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Table of contents

1. General information

The Fe-Anderson POMs catalysts was synthesized by a reported method from our group[1]. All chemicals used for the catalyst preparation were purchased from Sigma-Aldrich and Adamas-beta, and used directly without further purification. FT-IR spectra were recorded on a Thermo Fisher Nicolet 6700. XRD were explored on D/max 2200PC diffractometer (Rigaku, Japan) with Cu - Kα irradiation (λ = 1.5418 Å, 200 kV, 50 mA) in the range of 2θ value between 5° and 80°. GC-MS was implemented in Shimadzu with silicon as reference. GC analyses were performed on Shimadzu GC-2014 with a flame ionization detector equipped with Rtx-1 capillary column (internal diameter=0.25 mm, length=30 m) or a Stabil wax capillary column (internal diameter= 0.25 mm, length=30 m). GCMS-QP2010 with RTX-5MS capillary column(0.25mm×30m). Column chromatography was used 300-400 mesh.

2. Catalyst synthesis

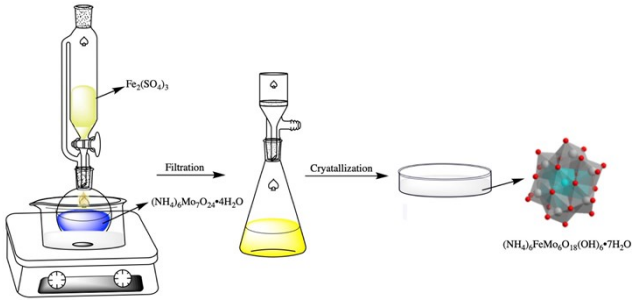


Figure S1. The process of $[NH_4]_3[FeMo_6O_{18}(OH)_6]\cdot 7H_2O$ preparation.

$[NH_4]_3[FeMo_6O_{18}(OH)_6]\cdot 7H_2O$ was synthesized following the previous published works from our group[1]. At first, a certain amount (5.0 g) of $[NH_4]_6Mo_7O_{24}\cdot 4H_2O$ was dissolved in 100 mL water, then the mixture was heated up to 100 ℃ in oil bath under magnetic stirring. Following, $Fe_2(SO_4)_3$ (1.24 g) was dissolved in 20 mL water before being dropped into the above solution. Finally, a slight yellow precipitation formed and was extracted and filtered by Buchner funnel, and the filtrate was placed at room temperature for 72 h for crystallization. Then, the crystallized crude samples were dried to produce white crystals at room temperature for a few days and precipitated the white crystals. After recrystallized, filtered and vacuum dried, the white crystals was deposited and collected. It is worth mentioning that the pH of the above mixed solution was adjusted to 2.5 by nitric acid. IR: 3217(ν_{as}NH, m), 1651 (δOH m), 1397 (δNH, s), 959 (ν Mo=O, ν_s), 860 (ν Mo=O, ν_s), 650 (ν Mo-O-Mo, ν_s), 577 (νM-O-Mo, w) cm^{-1}.

3. Gram-scale reaction process

The whole reaction process was carried with the protection of nitrogen. Typically, $(NH_4)_3[FeMo_6O_{18}(OH)_6]$ (0.05 mol%, 0.006 g) and Benzoic acid (1.0 mmol, 0.10715 g) were placed in a Schlenk tube. Benzylamine (1.0 mmol, 0.12212 g), $PhSiH_3$ (0.0325 g, 0.3 mmol) and toluene (1.0 mL) were sequentially added to the reaction tube. The reaction mixture was stirred at 120 ºC for 36 h. The reaction yield was

determined via gas chromatography-mass spectrometry (GC-MS). The resulting mixture was extracted with ethyl acetate (3×5 mL) and the organic layers were combined, dried over sodium sulfate and concentrated. Finally, the corresponding benzylbenzamide was purified by flashing column chromatography on silica gel (silica; petroleum ether-ethyl acetate mixture).

4. FT-IR spectra of Cat. 1

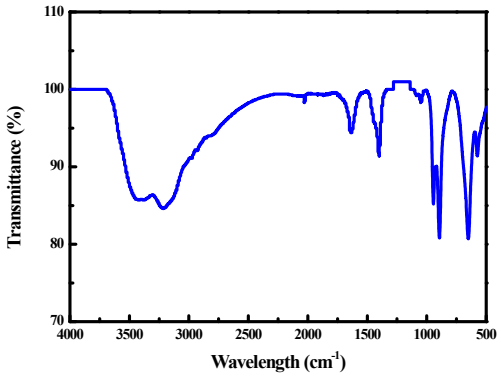


Figure S2. FT-IR spectra of Cat.**1**.

5. XRD spectra of Cat. 1

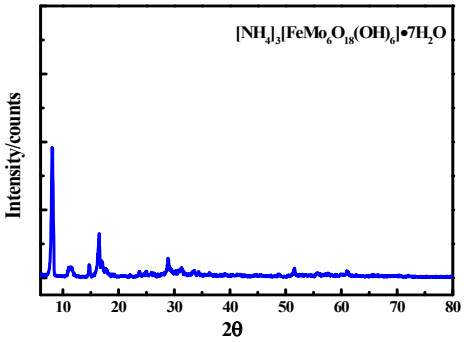


Figure S3. XRD spectra of Cat.**1**.

6. ESI-MS spectra of Cat. 1

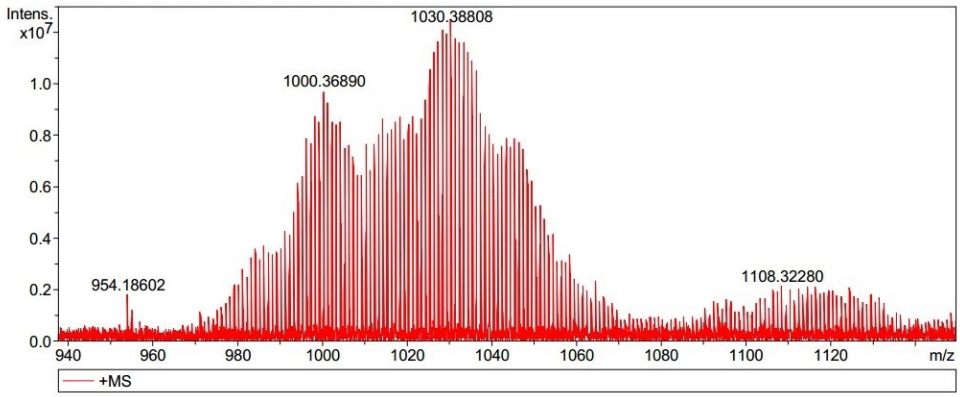
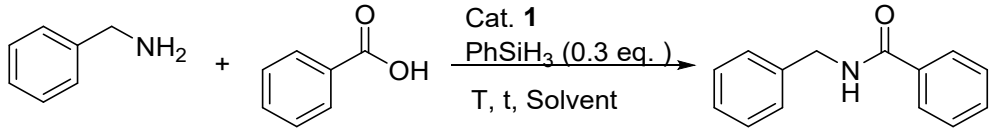


Figure S4. Zoom the area of ESI-MS of $(NH_4)_3[FeMo_6O_{18}(OH)_6]$, (m/z = 1010-1500,

$\{NH_4H[FeMo_6O_{24}H_6]\}^{1-}$ = 1030.388 g/mol).

7. Optimization of reaction conditions and extension of reaction

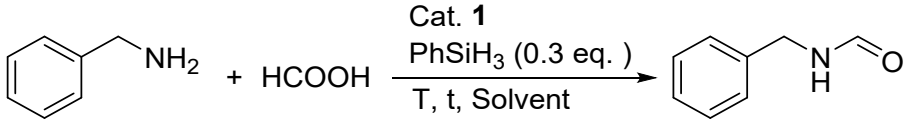
Table S1. Optimization of reaction conditions for Benzylamine and Benzoic acid[a]



Entry	Cat. 1 (mol%)	T (°C)	Solvent (mL)	Time (h)	Yield (%)[b]
1	1.0	80	Toluene (3.0)	24	13
2	1.0	100	Toluene (3.0)	24	25
3	1.0	110	Toluene (3.0)	24	43
4	1.0	120	Toluene (3.0)	24	50
5	1.0	120	Toluene (1.0)	24	56
6	1.0	120	Toluene (2.0)	24	43
7	1.0	120	Toluene (4.0)	24	42
9	1.0	120	1,4-dioxane (3.0)	24	13
10	1.0	120	DMF (3.0)	24	<5
11	1.0	120	m-Xylene (3.0)	24	28
12	1.0	120	Toluene (1.0)	12	48
13	1.0	120	Toluene (1.0)	36	80
14	1.0	120	Toluene (1.0)	48	72
15	0.25	120	Toluene (1.0)	36	70
16	**0.5**	**120**	**Toluene (1.0)**	**36**	**95**

[a]Reaction conditions: Benzylamine (1.0 mmol), Benzoic acid (1.0 mmol), $PhSiH_3$(0.3 equiv). [b]Yields were determined by GC-MS analysis of the crude reaction mixtures.

Table S2. Optimization of the reaction conditions of Benzylamine and Formic acid[a]

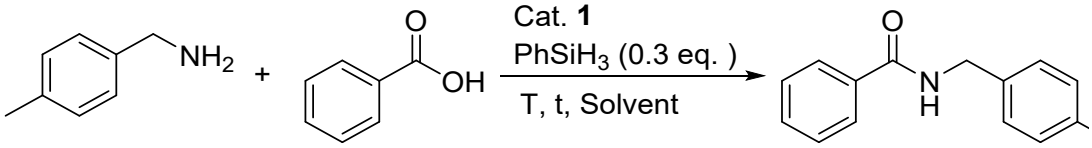


Entry	Cat. 1 (mol%)	T (°C)	Solvent (mL)	Time (h)	Yield (%)[b]
1	1.0	80	Toluene (3.0)	24	20
2	1.0	100	Toluene (3.0)	24	32
3	1.0	110	Toluene (3.0)	24	45
4	1.0	120	Toluene (3.0)	24	50
5	1.0	120	Toluene (1.0)	24	54
6	1.0	120	Toluene (2.0)	24	52
7	1.0	120	Toluene (4.0)	24	49
9	1.0	120	1,4-dioxane (3.0)	24	18
10	1.0	120	DMF (3.0)	24	Trace

11	1.0	120	m-Xylene (3.0)	24	25
12	1.0	120	Toluene (1.0)	12	41
13	1.0	120	Toluene (1.0)	36	82
14	1.0	120	Toluene (1.0)	48	80
15	0.25	120	Toluene (1.0)	36	74
16	**0.5**	**120**	**Toluene (1.0)**	**36**	**94**

[a]Reaction conditions: Benzylamine (1.0 mmol), HCOOH (1.0 mmol), $PhSiH_3$(0.3 equiv). [b]Yields were determined by GC-MS analysis of the crude reaction mixtures.

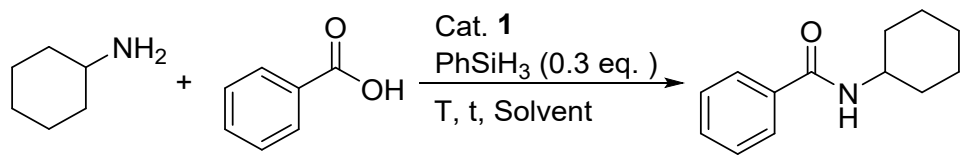
Table S3. Optimization of the reaction conditions of 4-Methylbenzylamine and Benzoic acid[a]



Entry	Cat. 1 (mol%)	T (°C)	Solvent (mL)	Time (h)	Yield (%)[b]
1	1.0	80	Toluene (3.0)	24	10
2	1.0	100	Toluene (3.0)	24	32
3	1.0	110	Toluene (3.0)	24	45
4	1.0	120	Toluene (3.0)	24	50
5	1.0	120	Toluene (1.0)	24	51
6	1.0	120	Toluene (2.0)	24	49
7	1.0	120	Toluene (4.0)	24	48
9	1.0	120	1,4-dioxane (3.0)	24	18
10	1.0	120	DMF (3.0)	24	Trace
11	1.0	120	m-Xylene (3.0)	24	25
12	1.0	120	Toluene (1.0)	12	50
13	1.0	120	Toluene (1.0)	36	82
14	1.0	120	Toluene (1.0)	48	80
15	0.25	120	Toluene (1.0)	36	74
16	**0.5**	**120**	**Toluene (1.0)**	**36**	**88**

[a]Reaction conditions: 4-Methylbenzylamine (1.0 mmol), Benzoic acid (1.0 mmol), $PhSiH_3$(0.3 equiv). [b]Yields were determined by GC-MS analysis of the crude reaction mixtures.

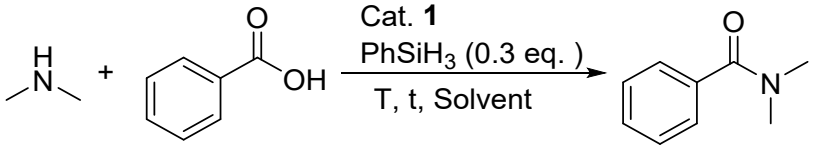
Table S4. Optimization of the reaction conditions of Cyclohexylamine and Benzoic acid[a



Entry	Cat. 1 (mol%)	T (°C)	Solvent (mL)	Time (h)	Yield (%)[b]
1	1.0	80	Toluene (3.0)	24	20
2	1.0	100	Toluene (3.0)	24	28
3	1.0	110	Toluene (3.0)	24	29
4	1.0	120	Toluene (3.0)	24	46
5	1.0	120	Toluene (1.0)	24	52
6	1.0	120	Toluene (2.0)	24	49
7	1.0	120	Toluene (4.0)	24	45
9	1.0	120	1,4-dioxane (3.0)	24	15
10	1.0	120	DMF (3.0)	24	Trace
11	1.0	120	m-Xylene (3.0)	24	20
12	1.0	120	Toluene (1.0)	12	42
13	1.0	120	Toluene (1.0)	36	80
14	1.0	120	Toluene (1.0)	48	78
15	0.25	120	Toluene (1.0)	36	76
16	**0.5**	**120**	**Toluene (1.0)**	**36**	**87**

[a]Reaction conditions: Cyclohexylamine (1.0 mmol), Benzoic acid (1.0 mmol), $PhSiH_3$(0.3 equiv). [b]Yields were determined by GC-MS analysis of the crude reaction mixtures.

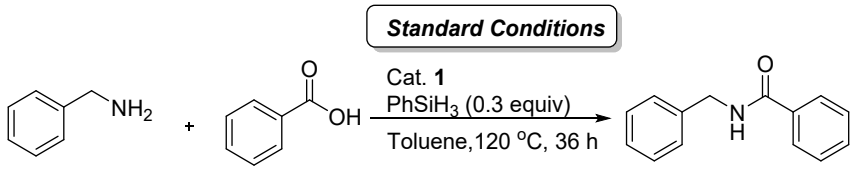
Table S5. Optimization of the reaction conditions of Dimethylamine and Benzoic acid[a]



Entry	Cat. 1 (mol%)	T (°C)	Solvent (mL)	Time (h)	Yield (%)[b]
1	1.0	80	Toluene (3.0)	24	18
2	1.0	100	Toluene (3.0)	24	25
3	1.0	110	Toluene (3.0)	24	46
4	1.0	120	Toluene (3.0)	24	55
5	1.0	120	Toluene (1.0)	24	58
6	1.0	120	Toluene (2.0)	24	56
7	1.0	120	Toluene (4.0)	24	55
9	1.0	120	1,4-dioxane (3.0)	24	10
10	1.0	120	DMF (3.0)	24	Trace
11	1.0	120	m-Xylene (3.0)	24	16
12	1.0	120	Toluene (1.0)	12	40
13	1.0	120	Toluene (1.0)	36	74
14	1.0	120	Toluene (1.0)	48	68
15	0.25	120	Toluene (1.0)	36	70
16	**0.5**	**120**	**Toluene (1.0)**	**36**	**77**

[a]Reaction conditions: Dimethylamine (1.0 mmol), Benzoic acid (1.0 mmol), $PhSiH_3$(0.3 equiv). [b]Yields were determined by GC-MS analysis of the crude reaction mixtures.

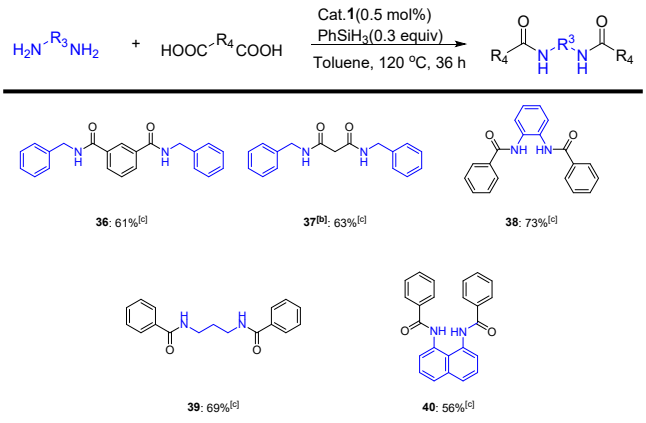
Table S6. Influence of the reaction parameters on the coupling of benzylamine with benzoic acid[a]



Entry	Deviations from standard conditions	Yield (%)[b]
1	None	95(90)
2	Without Cat. **1**	trace
3	$Fe_2(SO_4)_3$ instead of Cat. **1**	26
4	$(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ instead of Cat. **1**	43
5	$Fe_2(SO_4)_3$+$(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ instead of Cat. **1**	59
6	Only $Fe_2(SO_4)_3$	15
7	Only $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$	19
8	Only $Fe_2(SO_4)_3$+$(NH_4)_6Mo_7O_{24}$	21
9	Without $PhSiH_3$	73
10	KCl instead of $PhSiH_3$	25
11	Mo_7 instead of Cat. **1**	20

[a]Reaction conditions: Cat. **1** (0.5 mol%), Benzylamine (1.0 mmol), Benzoic acid (1.0 mmol), Toluene (1.0 mL). [b]Yields were determined by GC-MS analysis of the crude reaction mixtures, value in parentheses is the isolated yields.

Table S7. Catalytic systems for the synthesis of diamides from carboxylic acids and amines[a]



[a]Reaction conditions: Cat. **1** (0.5 mol%),

Benzoic acid (1.0 mmol), Amine (1.0 mmol), Toluene (1.0 mL), $PhSiH_3$ (0.3 eq.), stirring at 120 °C for 36 h. [b]Amine (2.0 mmol). [c]Isolated yields were determined by ^1H NMR.

8. Recycle experiments of catalysts

The Cat. **1** was separated by centrifugation, washed in turn with water, acetone and ethyl acetate. Then, the dried catalyst was reused for more cycles (Figure S5). During the experiments, the amount of reactants were adjusted according to the weight of dried catalysts from last cycle to make sure the ratio between the catalysts and reactants kept the same for all the cycles. The recovered catalyst was characterized by FT-IR (Figure S6) and XRD (Figure S7).

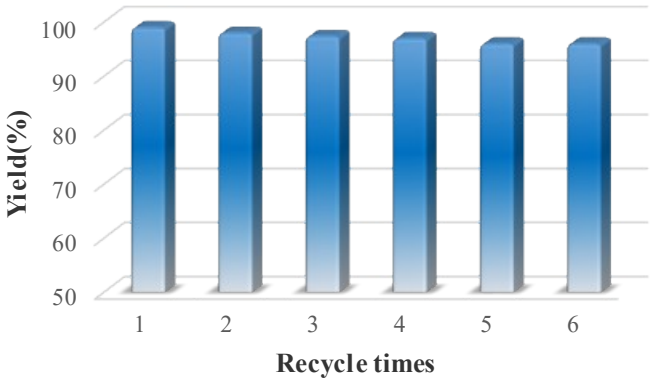


Figure S5. Recycling experiments for the Cat. **1**.

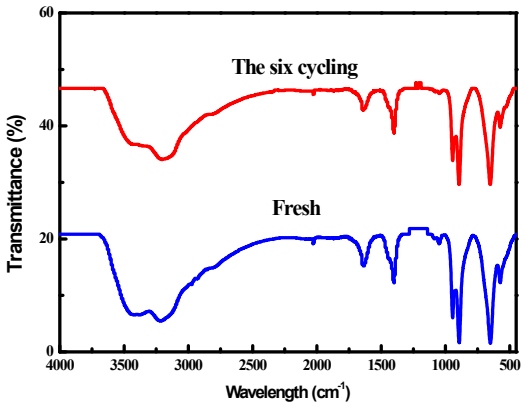


Figure S6. FT-IR spectra of Cat. **1** before and after reaction.

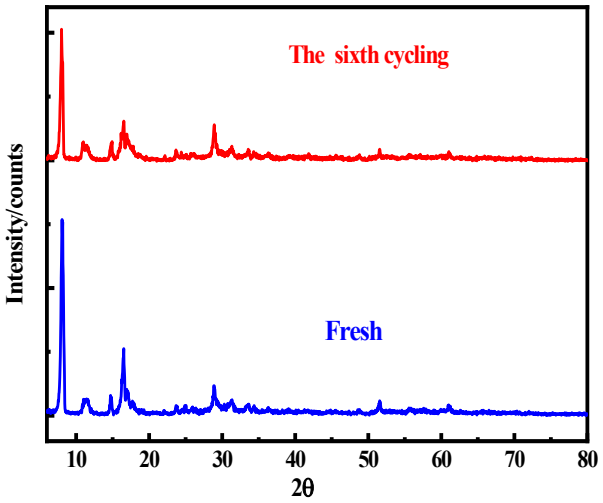


Figure S7. XRD spectra of Cat. **1** before and after reaction.

9. References

[1] Nomiya, K.; Takahashi, T.; Shirai, T.; Miwa, M. Anderson-type heteropolyanions of molybdenum (VI) and tungsten(VI). *Polyhedron*. **1987**, *6*, 213-218.

[2] Ping fan Wu et al. Single-Side Organically Functionalized Anderson-Type Polyoxometalates. *Chem. Eur. J.* **2011**, *17*, 12002-12005.

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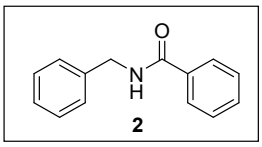
[6] Sawant, D.N.; Bagal, D.B.; Ogawa, S.; Selvam, K.; Saito, S. Diboron-catalyzed dehydrative amidation of aromatic carboxylic acids with amines. *Org. Lett.* **2018**, *20*, 4397-4400.

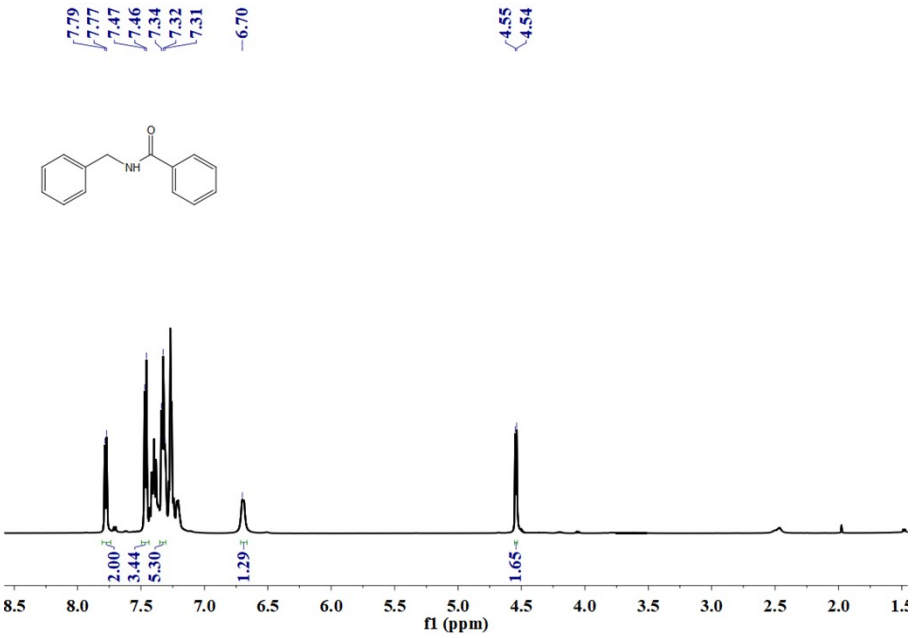
10. NMR data of products



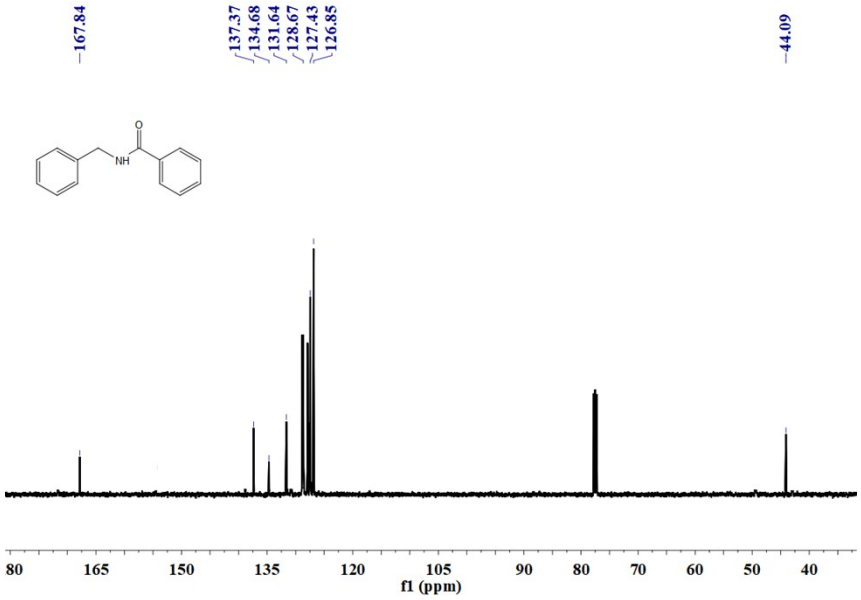
2

***N*-benzylbenzamide[3-6]:** ^{1}H NMR (501 MHz, $CDCl_3$) δ 7.78 (d, *J* = 7.4 Hz, 2H), 7.46 (d, *J* = 7.2 Hz, 3H), 7.32 (t, *J* = 7.4 Hz, 5H), 6.70 (s, 1H), 4.54 (d, *J* = 5.8 Hz, 2H).

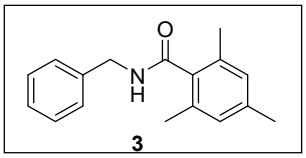
^{13}C NMR (126 MHz, $CDCl_3$) δ 167.84 (s), 137.37 (s), 134.68 (s), 131.64 (s), 128.67 (s), 127.43 (s), 126.85 (s), 44.09 (s).



^{1}H NMR spectra of **2** (501 MHz, $CDCl_3$)

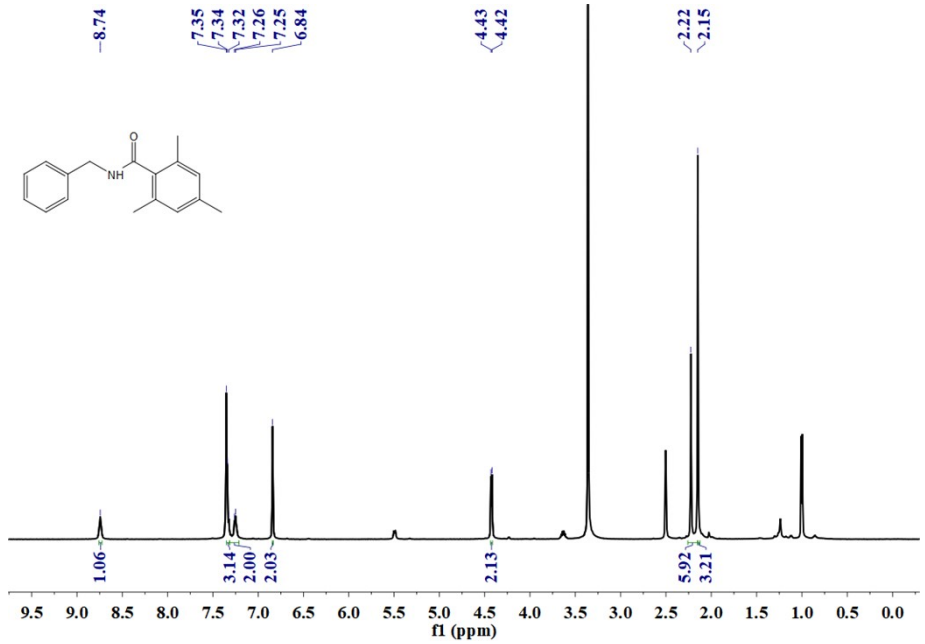


^{13}C NMR spectra of **2** (126 MHz, $CDCl_3$)

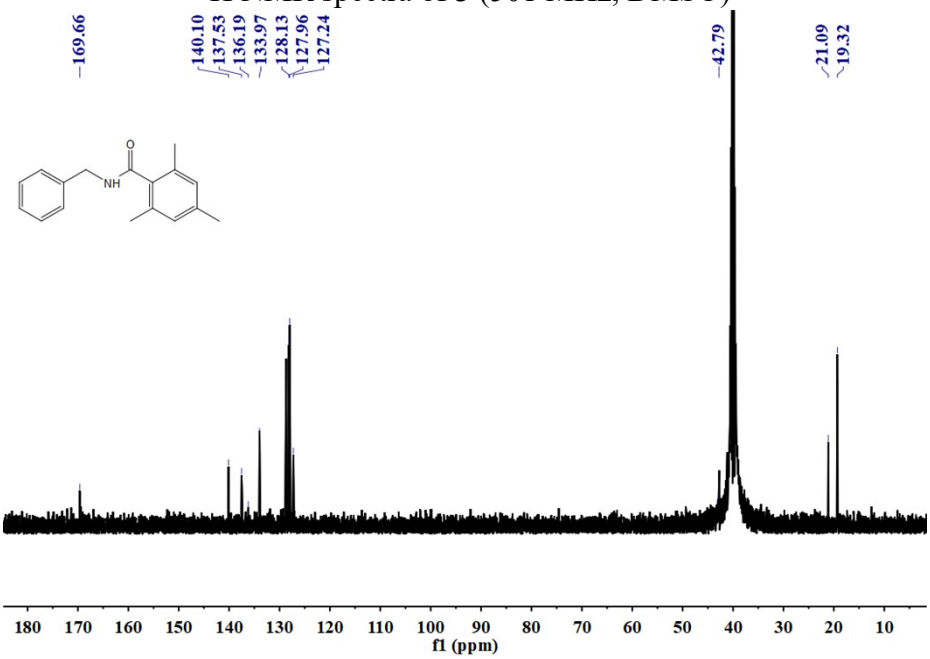


***N*-benzyl-2,4,6-trimethylbenzamide**[3-6]**:** ^{1}H NMR (501 MHz, DMSO) δ 8.74 (s, 1H), 7.33 (d, *J* = 8.0 Hz, 3H), 7.26 (d, *J* = 6.1 Hz, 2H), 6.84 (s, 2H), 4.43 (d, *J* = 6.0 Hz, 2H), 2.22 (s, 6H), 2.15 – 2.13 (m, 3H).

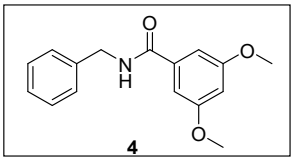
^{13}C NMR (126 MHz, DMSO) δ 169.66 (s), 140.10 (s), 137.53 (s), 136.19 (s), 133.97 (s), 128.13 (s), 127.96 (s), 127.24 (s), 42.79 (s), 21.09 (s), 19.32 (s).



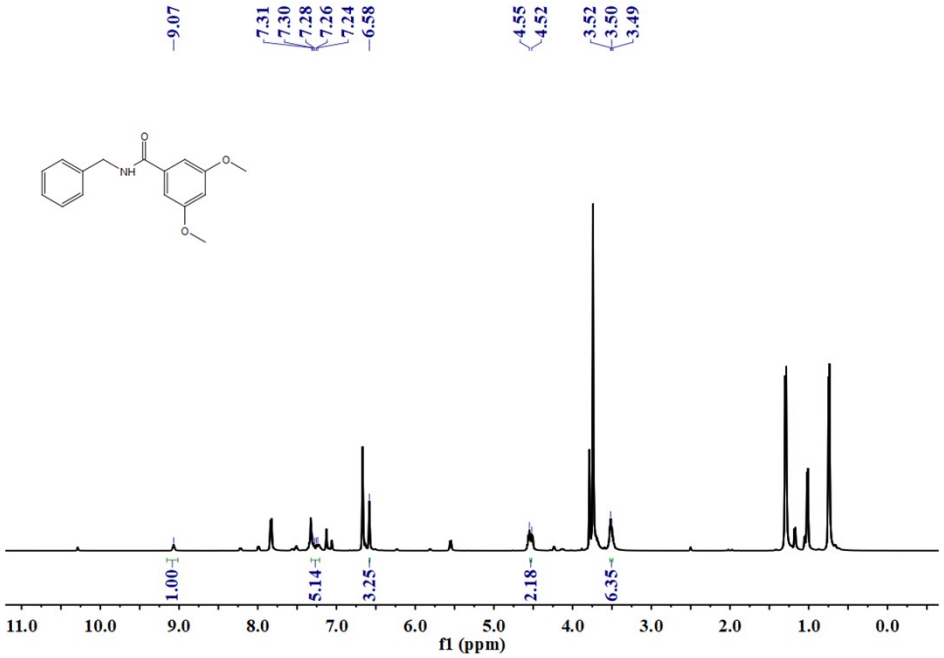
^{1}H NMR spectra of **3** (501 MHz, DMSO)



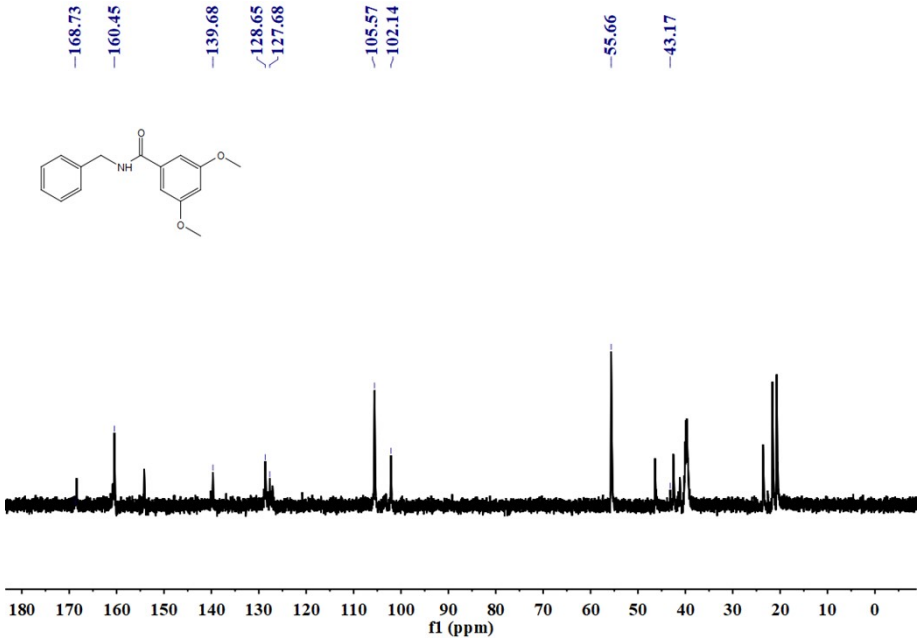
^{13}C NMR spectra of **3** (126 MHz, DMSO)



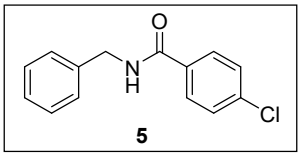
***N*-benzyl-3,5-dimethoxybenzamide**[3-6]**:** ^{1}H NMR (501 MHz, DMSO) δ 9.07 (s, 1H), 7.32 – 7.22 (m, 5H), 6.58 (s, 3H), 4.53 (d, *J* = 7.6 Hz, 2H), 3.51 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (126 MHz, DMSO) δ 168.81 – 168.65 (m), 160.45 (s), 139.68 (s), 128.65 (s), 127.68 (s), 105.57 (s), 102.14 (s), 55.66 (s), 43.17 (s).



^{1}H NMR spectra of **4** (501 MHz, DMSO)

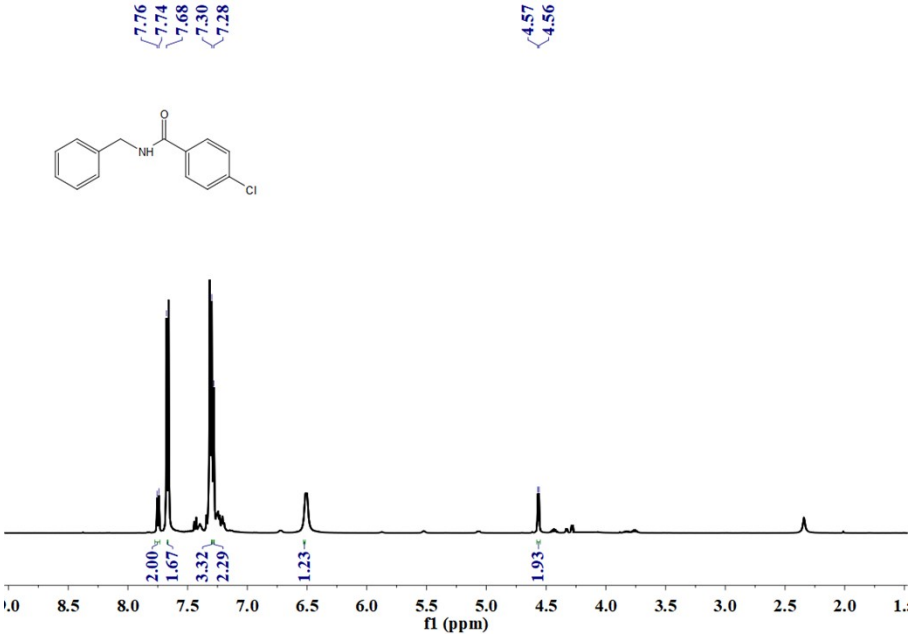


^{13}C NMR spectra of **4** (126 MHz, DMSO)

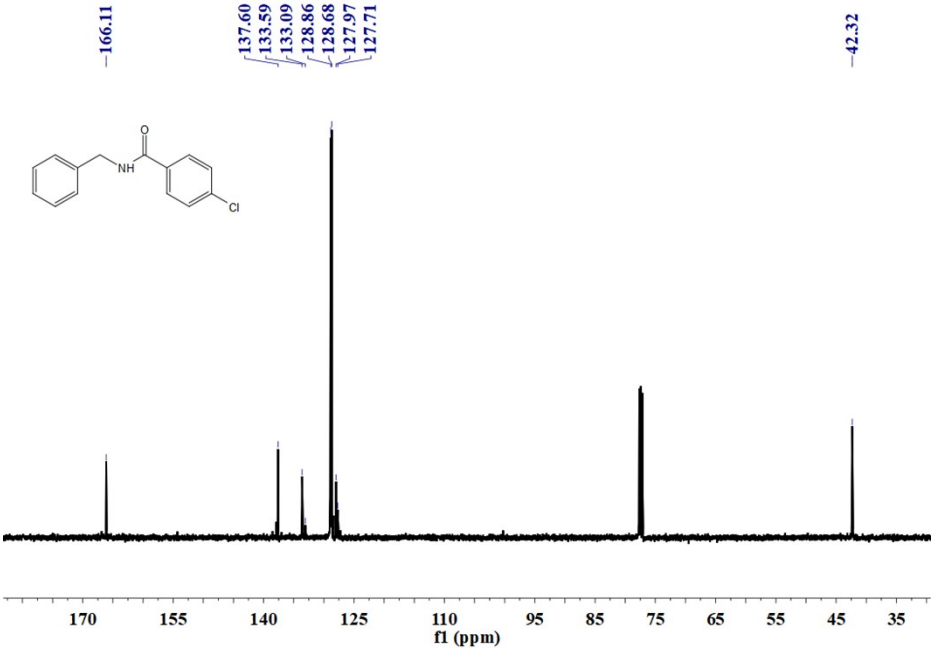


5

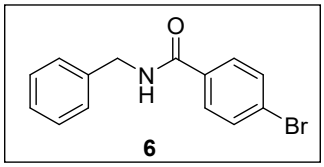
***N*-benzyl-4-chlorobenzamide[3-6]:** ^{1}H NMR (501 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.5 Hz, 2H), 7.67 (s, 2H), 7.30 (s, 3H), 7.28 (s, 2H), 6.52 (s, 1H), 4.56 (d, *J* = 5.8 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.11 (s), 137.60 (s), 133.59 (s), 133.09 (s), 128.86 (s), 128.68 (s), 127.97 (s), 127.71 (s), 44.28 (s).



^{1}H NMR spectra of **5** (501 MHz, $CDCl_3$)

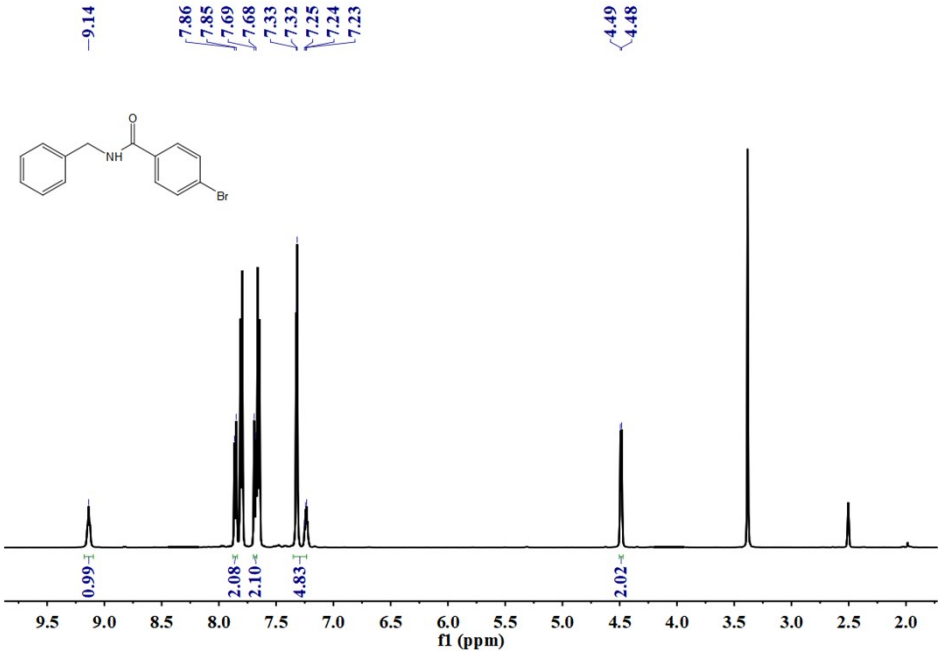


^{13}C NMR spectra of **5** (126 MHz, $CDCl_3$)

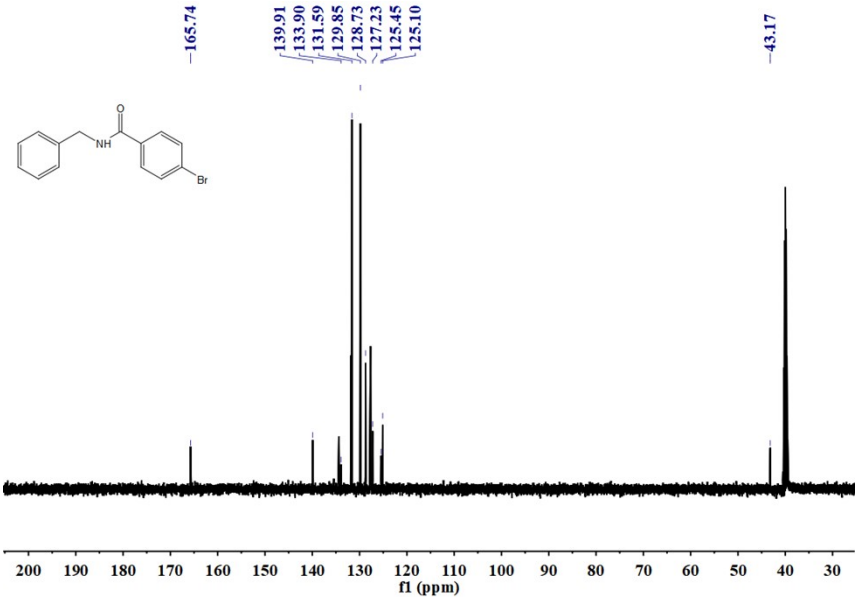


***N*-benzyl-4-bromobenzamide**[3-6]**:** ^{1}H NMR (501 MHz, DMSO) δ 9.14 (s, 1H), 7.86 (d, *J* = 8.4 Hz, 2H), 7.68 (d, *J* = 8.4 Hz, 2H), 7.35 – 7.23 (m, 5H), 4.49 (d, *J* = 5.9 Hz, 2H).

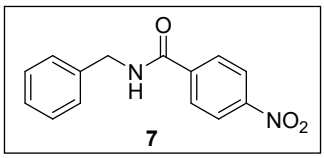
^{13}C NMR (126 MHz, DMSO) δ 165.74 (s), 139.91 (s), 133.90 (s), 131.59 (s), 129.85 (s), 128.73 (s), 127.23 (s), 125.45 (s), 125.10 (s), 43.17 (s).



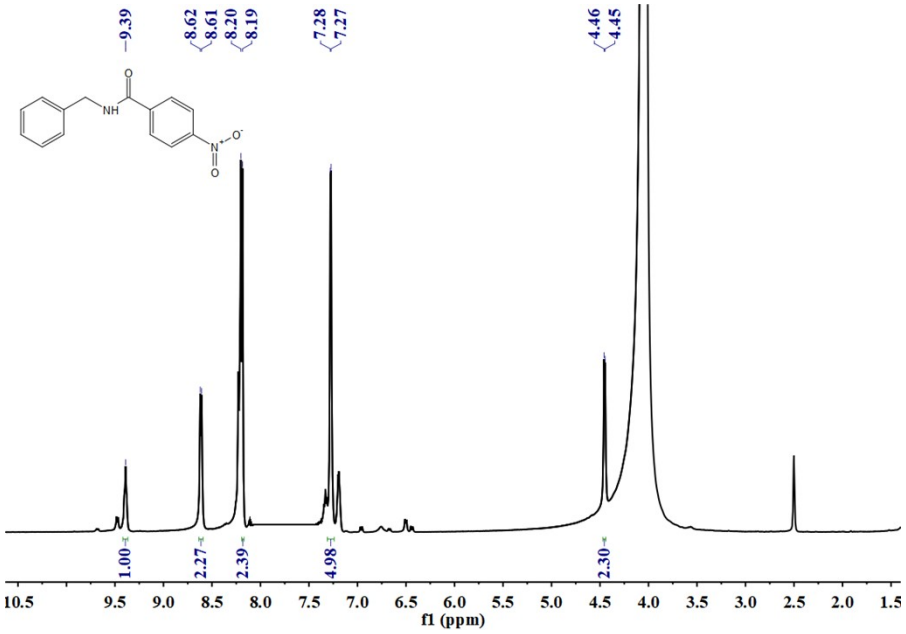
^{1}H NMR spectra of **6** (501 MHz, DMSO)



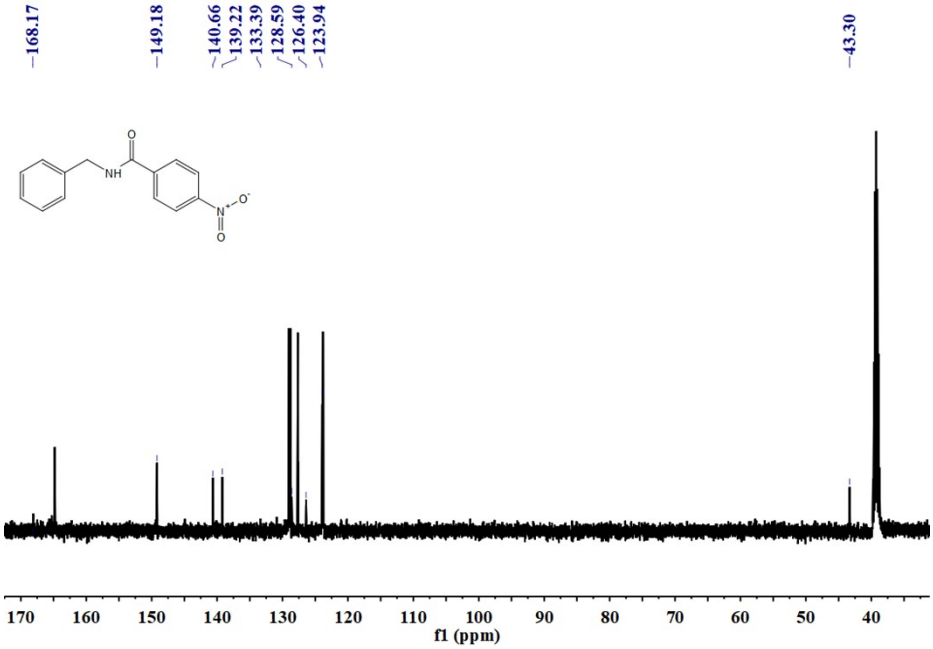
^{13}C NMR spectra of **6** (126 MHz, DMSO)



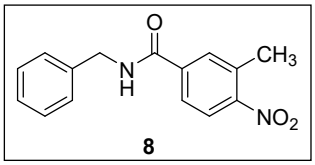
***N*-benzyl-4-nitrobenzamide**[3-6]**:** ^{1}H NMR (501 MHz, DMSO) δ 9.39 (s, 1H), 8.61 (d, *J* = 7.4 Hz, 2H), 8.19 (s, 2H), 7.28 (d, *J* = 4.0 Hz, 5H), 4.45 (d, *J* = 5.6 Hz, 2H).
^{13}C NMR (126 MHz, DMSO) δ 149.18 (s), 140.66 (s), 139.22 (s), 133.39 (s), 128.59 (s), 126.40 (s), 123.94 (s), 43.30 (s).



^{1}H NMR spectra of **7** (501 MHz, DMSO)



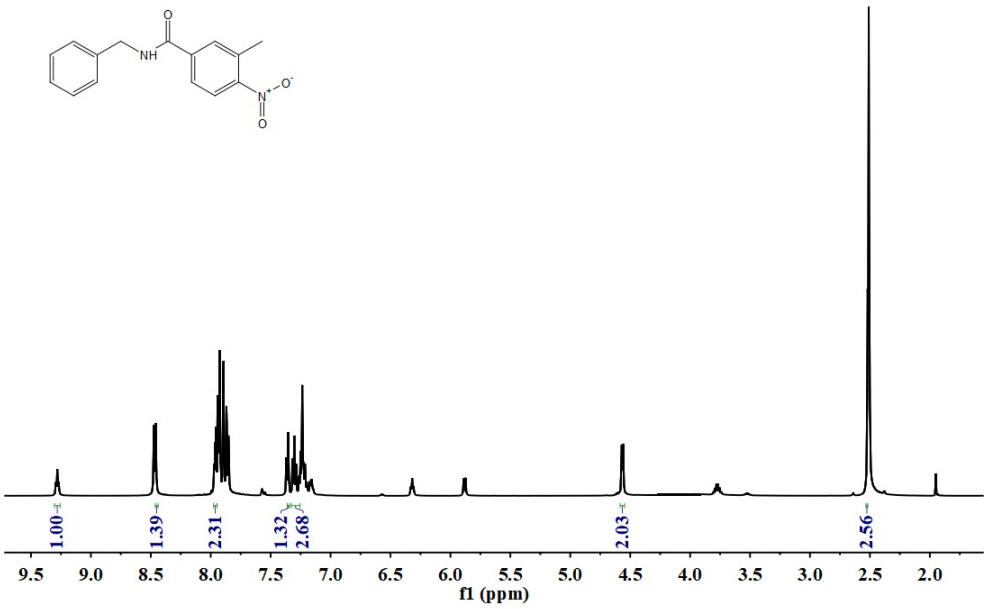
^{13}C NMR spectra of **7** (126 MHz, DMSO)



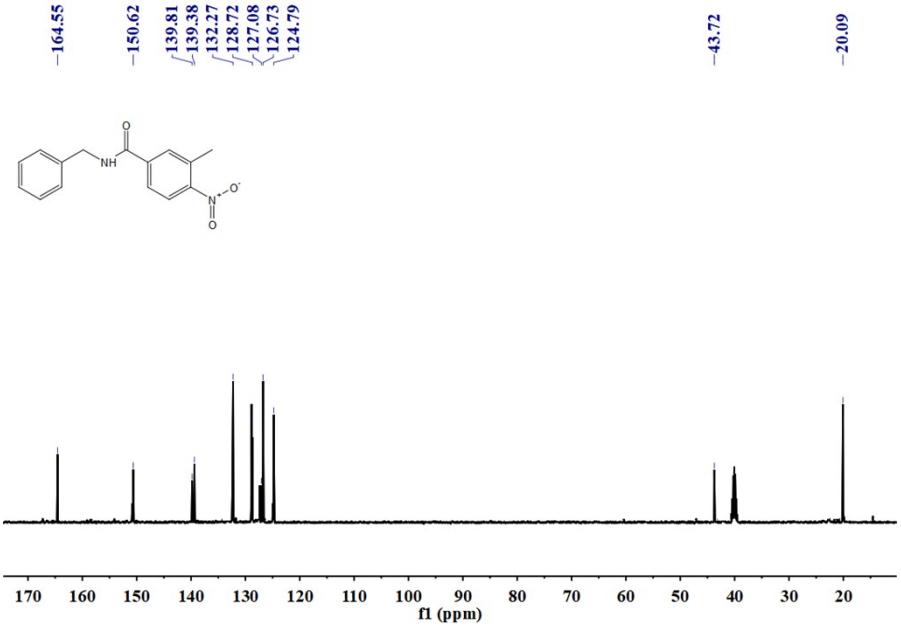
8

***N*-benzyl-3-methyl-4-nitrobenzamide**[3-6]**:** ^{1}H NMR (501 MHz, DMSO) δ 9.28 (s, 1H), 8.46 (s, 1H), 7.97 (d, *J* = 4.9 Hz, 2H), 7.35 (s, 1H), 7.30 (t, *J* = 7.6 Hz, 3H), 4.56 (d, *J* = 5.8 Hz, 2H), 2.52 (s, 3H).

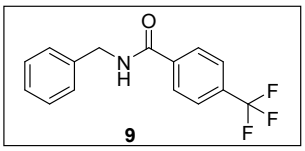
^{13}C NMR (126 MHz, DMSO) δ 164.55 (s), 150.62 (s), 139.81 (s), 139.38 (s), 132.27 (s), 128.72 (s), 127.08 (s), 126.73 (s), 124.79 (s), 43.72 (s), 20.09 (s).



^{1}H NMR spectra of **8** (501 MHz, DMSO)

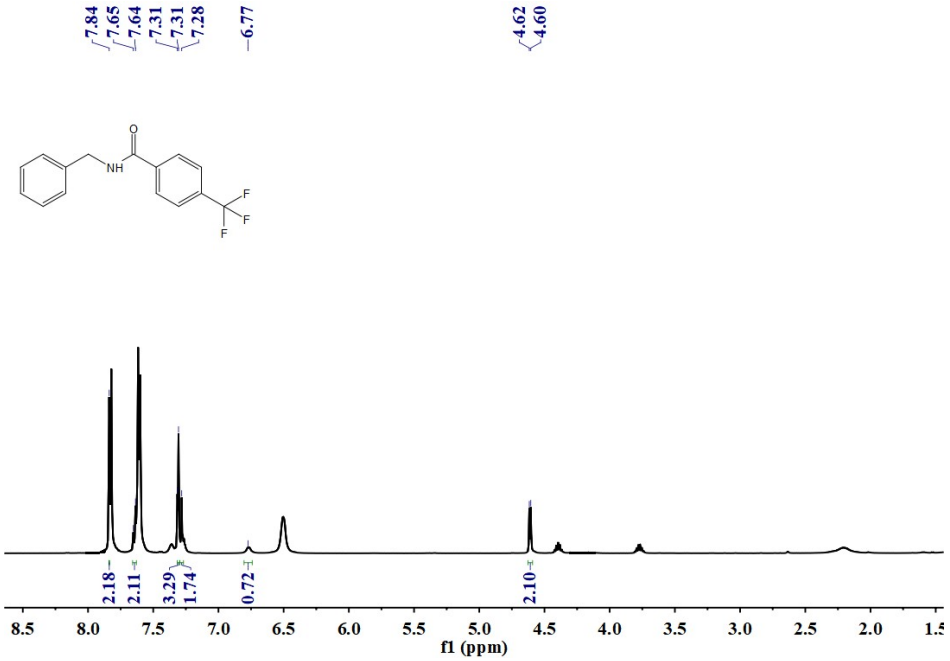


^{13}C NMR spectra of **8** (126 MHz, DMSO)

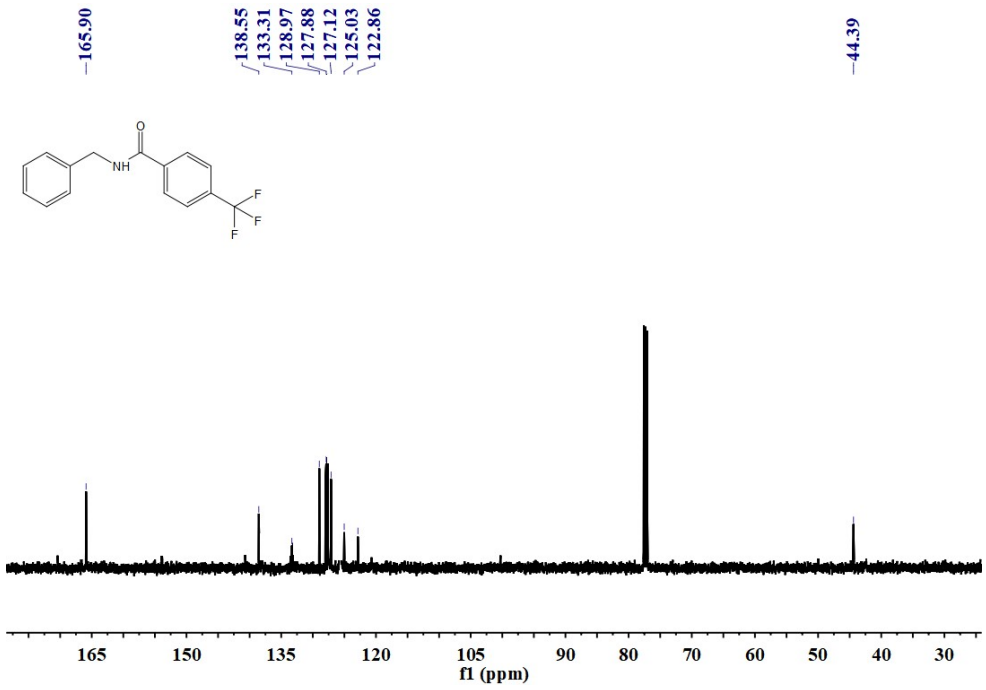


***N*-benzyl-4-(trifluoromethyl)benzamide**[3-6]**:** ^{1}H NMR (501 MHz, $CDCl_3$) δ 7.84 (s, 2H), 7.64 (d, *J* = 8.3 Hz, 2H), 7.31 (d, *J* = 4.2 Hz, 3H), 7.28 (s, 2H), 6.77 (s, 1H), 4.61 (d, *J* = 5.8 Hz, 2H).

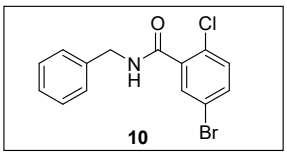
^{13}C NMR (126 MHz, $CDCl_3$) δ 165.90 (s), 138.55 (s), 133.31 (s), 128.97 (s), 127.88 (s), 127.12 (s), 125.03 (s), 122.86 (s), 44.39 (s).



^{1}H NMR spectra of **9** (501 MHz, $CDCl_3$)

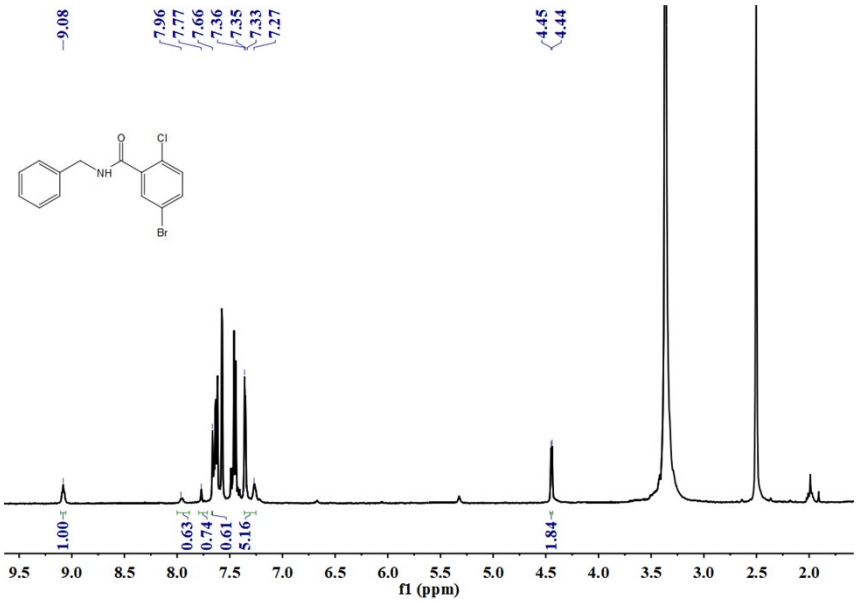


^{13}C NMR spectra of **9** (126 MHz, $CDCl_3$)

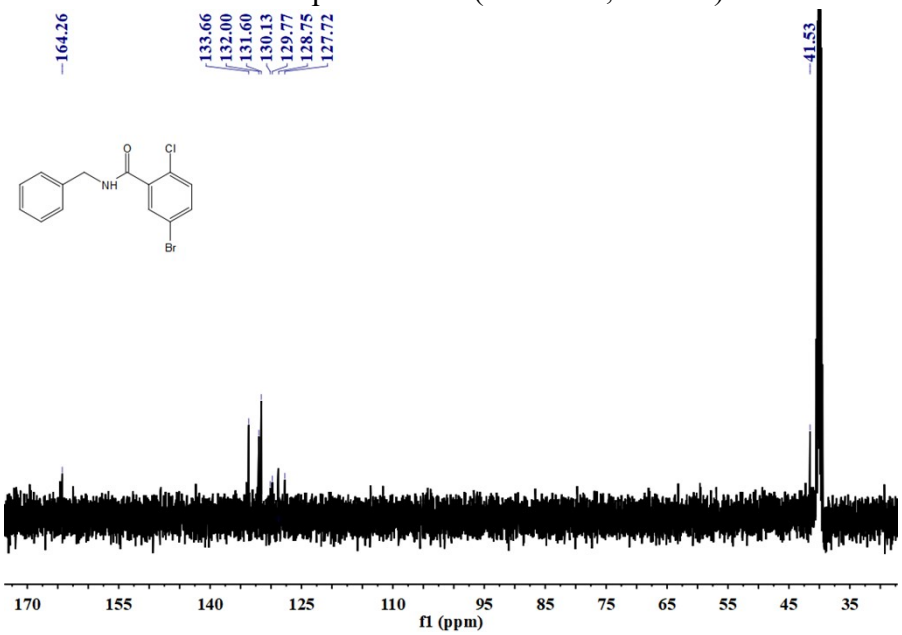


***N*-benzyl-5-bromo-2-chlorobenzamide**[3-6]**:** ^{1}H NMR (501 MHz, DMSO) δ 9.08 (s, 1H), 7.96 (s, 1H), 7.77 (s, 1H), 7.66 (s, 1H), 7.33 (dd, J = 27.3, 17.6 Hz, 5H), 4.45 (d, J = 5.9 Hz, 2H).

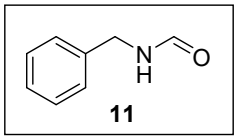
^{13}C NMR (126 MHz, DMSO) δ 164.26 (s), 133.66 (s), 132.00 (s), 131.60 (s), 130.13 (s), 129.77 (s), 128.83 – 128.67 (m), 127.72 (s), 41.53 (s).



^{1}H NMR spectra of **10** (501 MHz, DMSO)

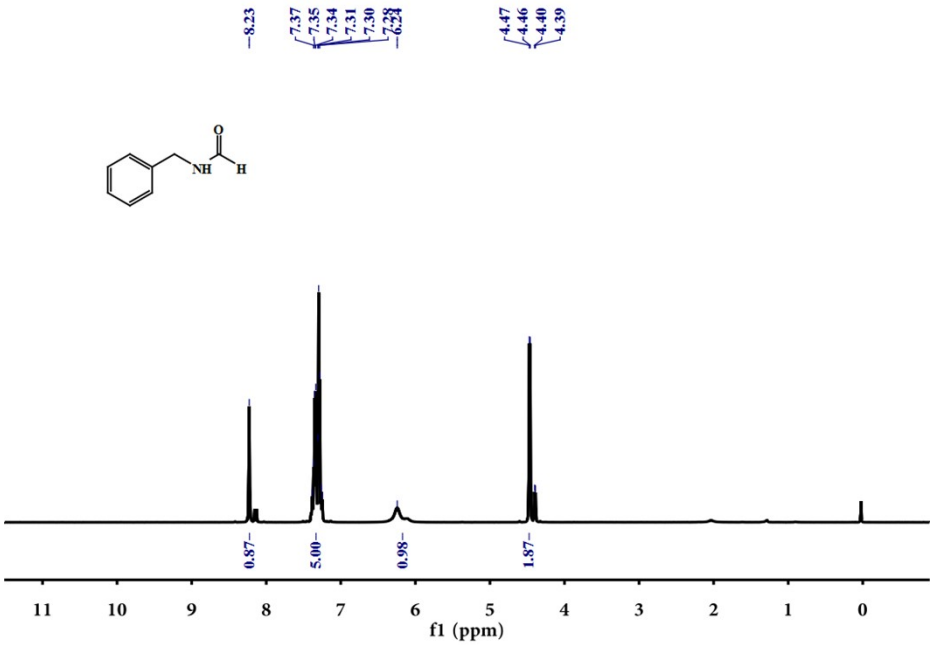


^{13}C NMR spectra of **10** (126 MHz, DMSO)

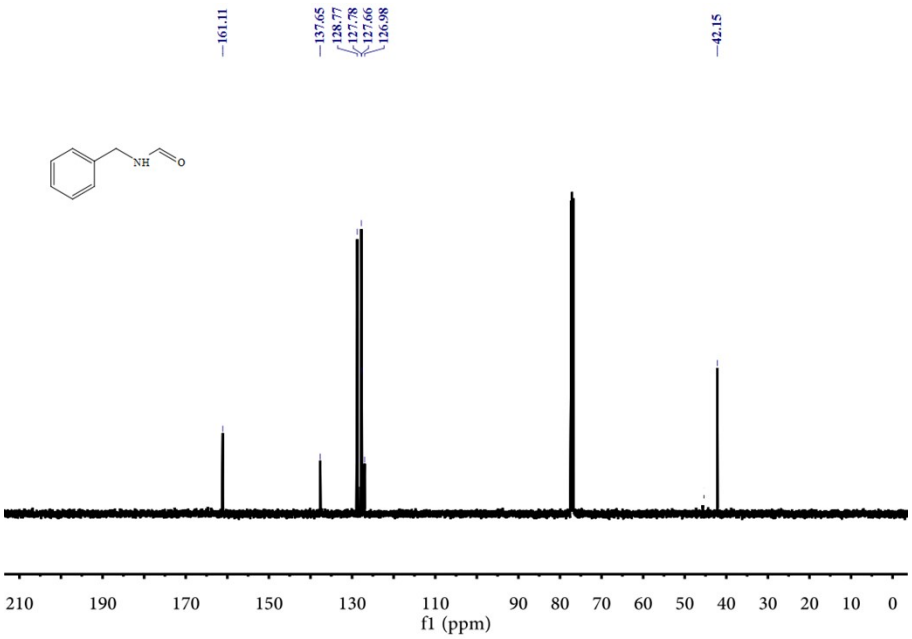


11

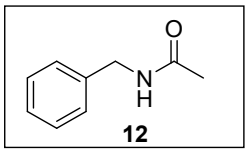
***N*-benzylformamide**[3-6]**:** ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10 (s, 1H), 7.37 – 7.27 (m, 3H), 7.24 (t, *J* = 7.3 Hz, 2H), 6.99 (s, 1H), 4.37 (s, 2H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 160.81 (s), 137.74 (s), 129.35 (s), 128.08 (s), 42.37 (s).



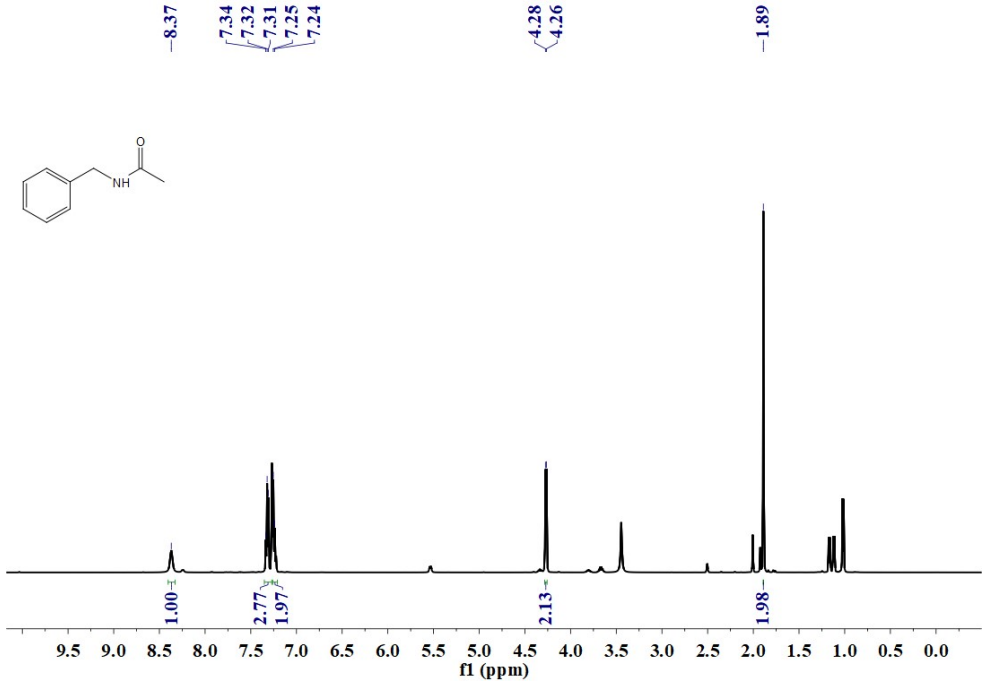
^{1}H NMR spectra of **11** (500 MHz, $CDCl_3$)



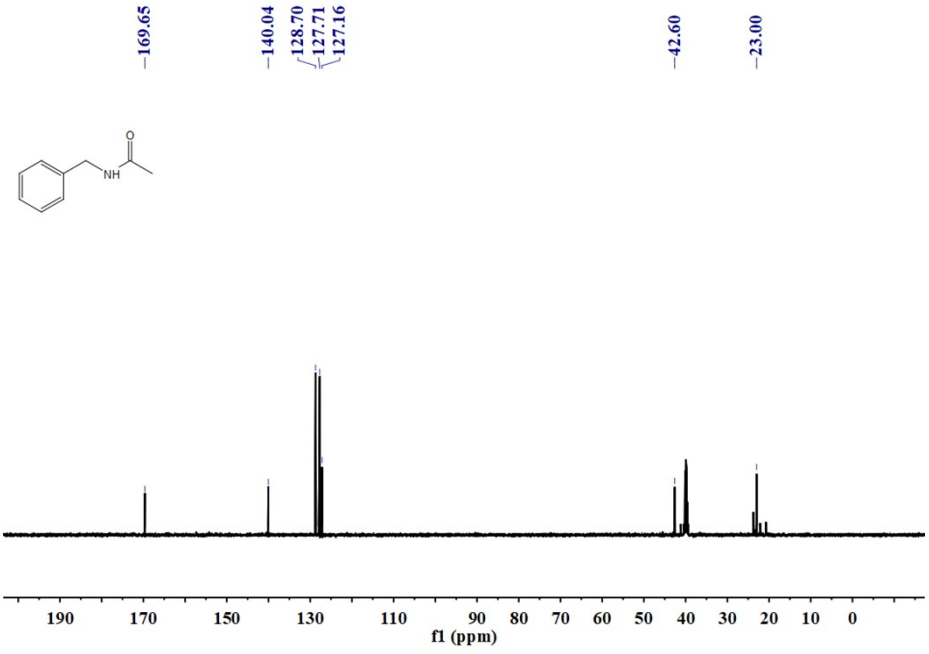
^{13}C NMR spectra of **11** (125 MHz, $CDCl_3$)



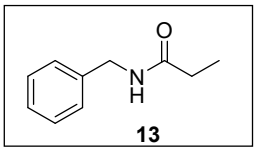
***N*-benzylacetamide**[3-6]**:** ^{1}H NMR (501 MHz, DMSO) δ 8.37 (s, 1H), 7.32 (t, *J* = 7.4 Hz, 3H), 7.25 (d, *J* = 8.0 Hz, 2H), 4.27 (d, *J* = 5.9 Hz, 2H), 1.89 (s, 2H).
^{13}C NMR (126 MHz, DMSO) δ 169.65 (s), 140.04 (s), 128.70 (s), 127.71 (s), 127.16 (s), 42.60 (s), 23.00 (s).



^{1}H NMR spectra of **12** (501 MHz, DMSO)

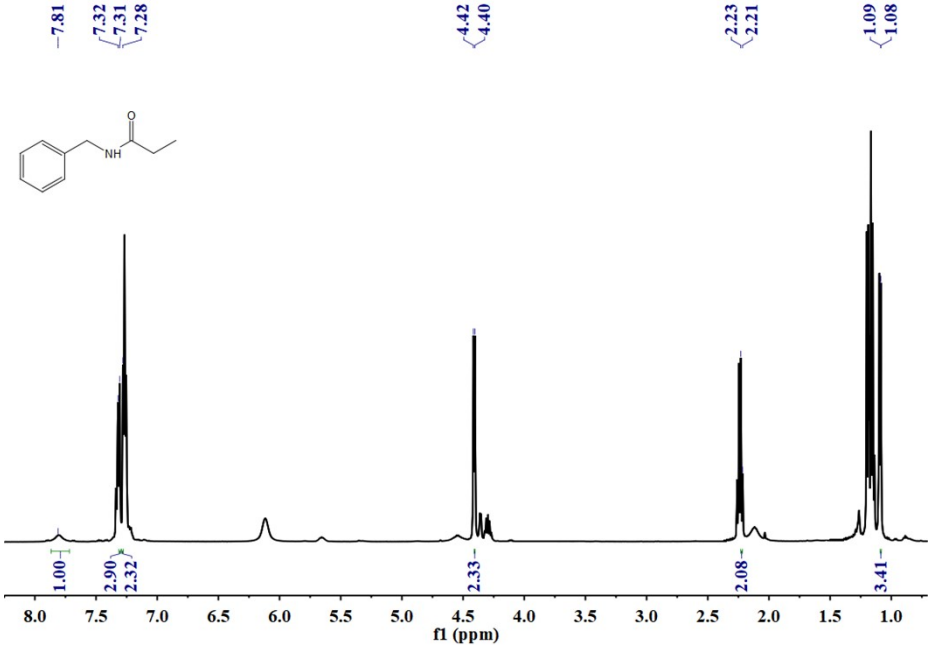


^{13}C NMR spectra of **12** (126 MHz, DMSO)

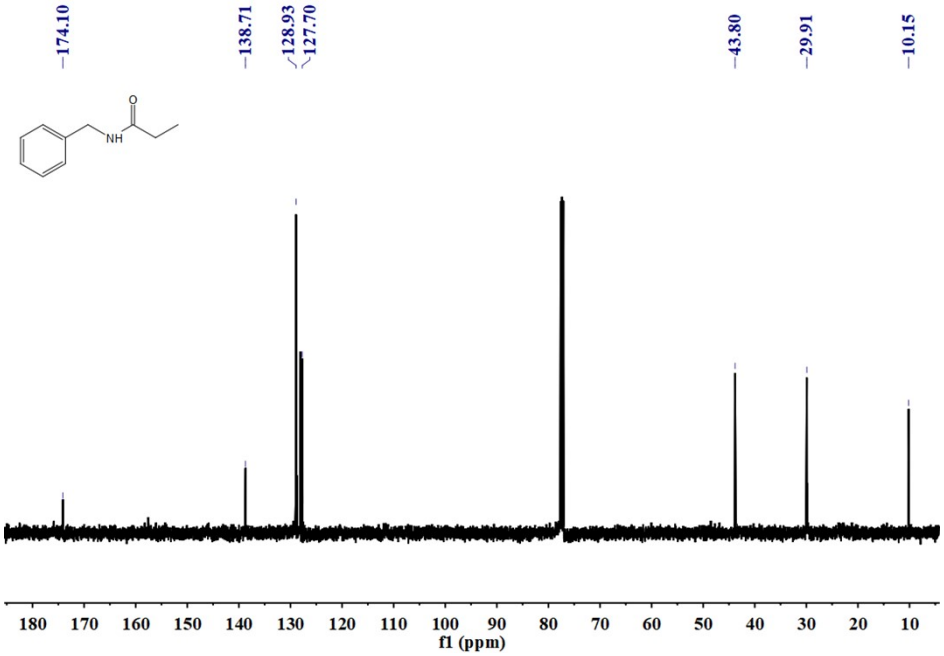


13

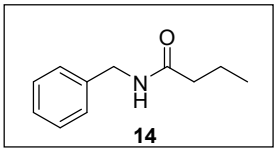
***N*-benzylpropionamide**[3-6]**:** ^{1}H NMR (501 MHz, $CDCl_3$) δ 7.81 (s, 1H), 7.31 (s, 3H), 7.28 (s, 2H), 4.40 (s, 2H), 2.23 (s, 2H), 1.09 (d, J = 1.3 Hz, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 174.10 (s), 138.71 (s), 128.93 (s), 127.70 (s), 43.80 (s), 29.91 (s), 10.15 (s).



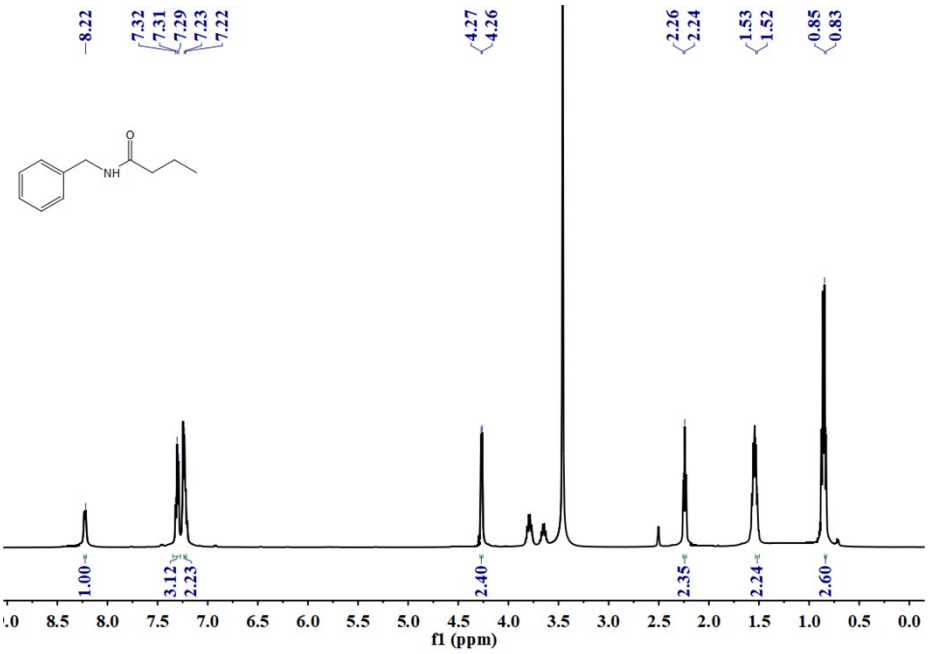
^{1}H NMR spectra of **13** (501 MHz, $CDCl_3$)



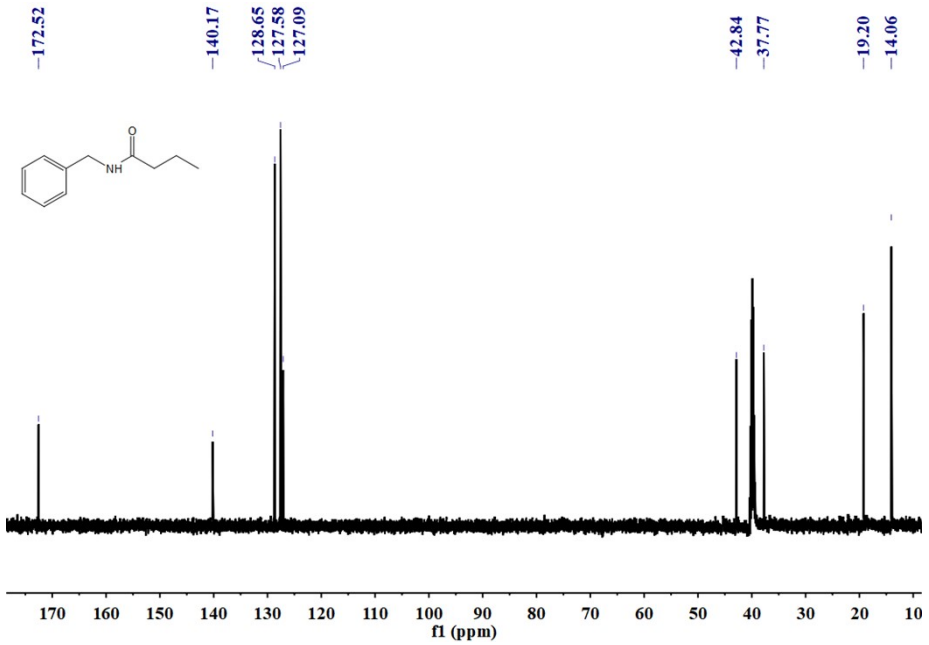
^{13}C NMR spectra of **13** (126 MHz, $CDCl_3$)



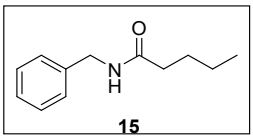
***N*-benzylbutyramide**[3-6]**:** ^{1}H NMR (501 MHz, DMSO) δ 8.22 (s, 1H), 7.31 (t, *J* = 7.4 Hz, 3H), 7.23 (d, *J* = 6.1 Hz, 2H), 4.27 (d, *J* = 5.9 Hz, 2H), 2.25 (d, *J* = 7.3 Hz, 2H), 1.52 (d, *J* = 7.2 Hz, 2H), 0.84 (d, *J* = 7.5 Hz, 3H).
^{13}C NMR (126 MHz, DMSO) δ 172.52 (s), 140.17 (s), 128.65 (s), 127.58 (s), 127.09 (s), 42.84 (s), 37.77 (s), 19.20 (s), 14.06 (s).



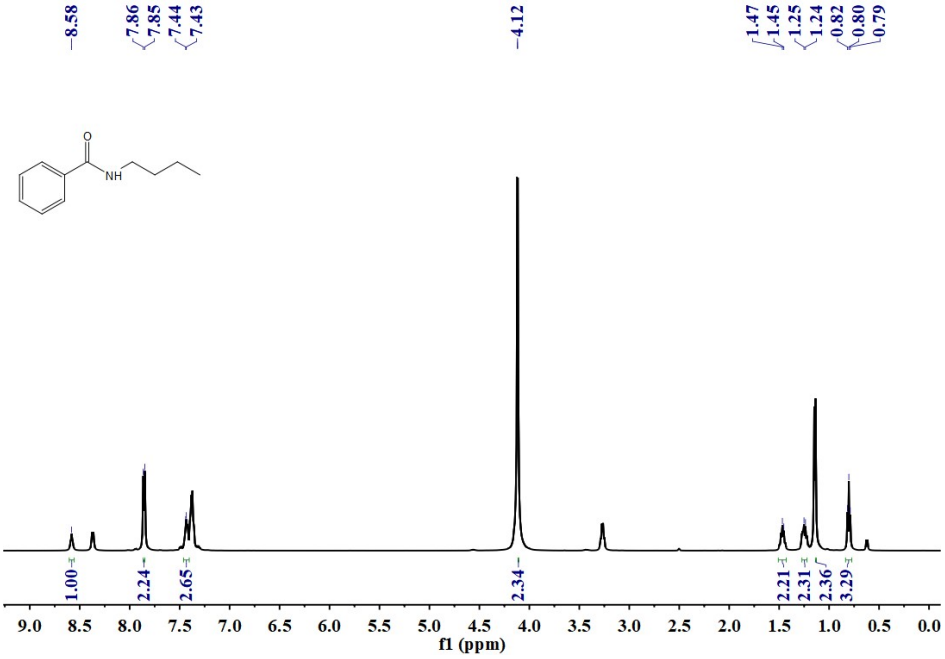
^{1}H NMR spectra of **14** (501 MHz, DMSO)



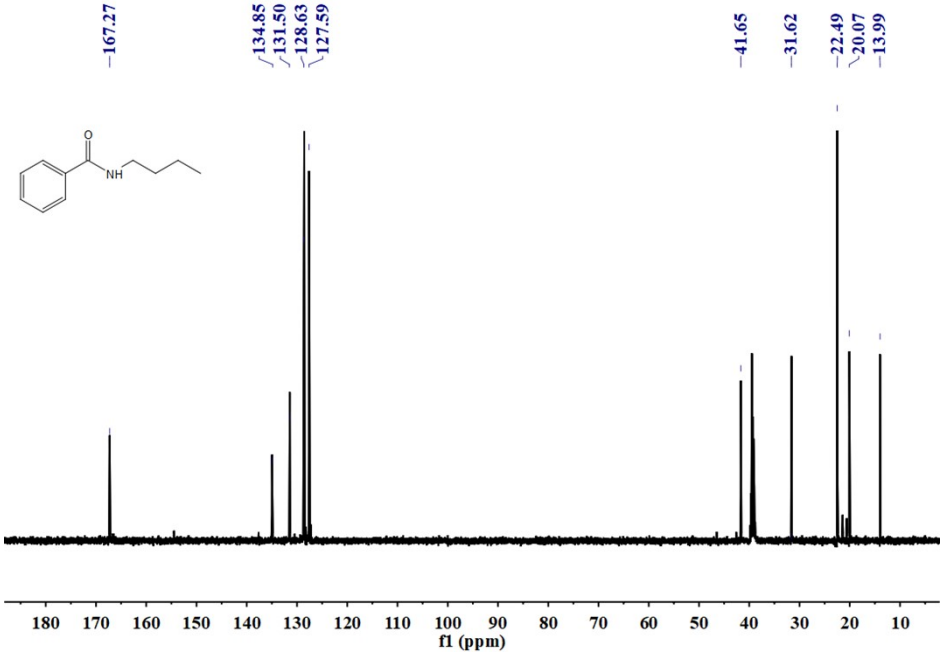
^{13}C NMR spectra of **14** (126 MHz, DMSO)



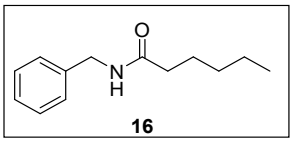
***N*-benzylpentanamide**[3-6]**:** ^{1}H NMR (501 MHz, DMSO) δ 8.58 (s, 1H), 7.86 (d, J = 7.4 Hz, 2H), 7.44 (d, J = 3.2 Hz, 3H), 4.11 – 4.11 (m, 2H), 1.46 (d, J = 7.2 Hz, 2H), 1.24 (d, J = 7.6 Hz, 2H), 1.14 (s, 2H), 0.80 (t, J = 7.3 Hz, 3H).
^{13}C NMR (126 MHz, DMSO) δ 167.27 (s), 134.85 (s), 131.50 (s), 127.59 (s), 41.65 (s), 31.58 (s), 22.49 (s), 20.07 (s), 13.99 (s).



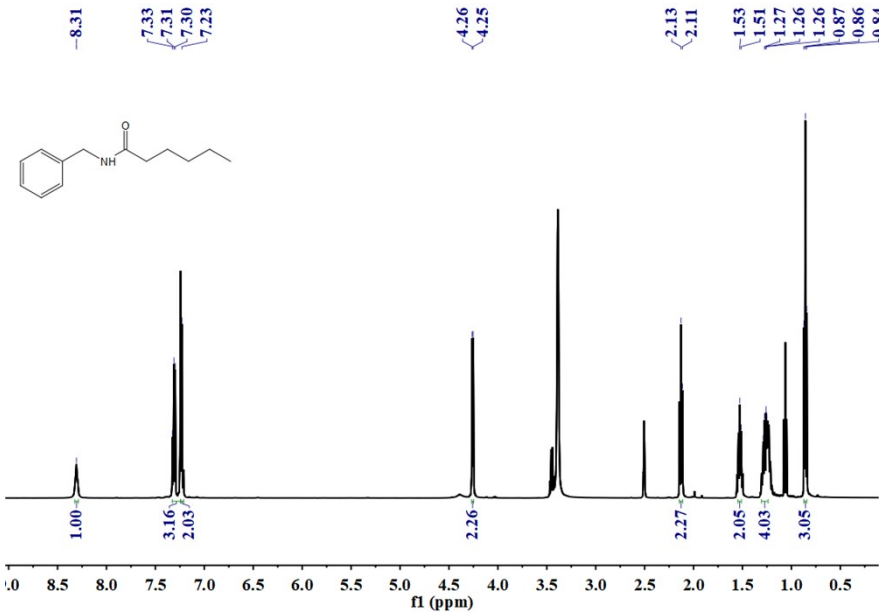
^{1}H NMR spectra of **15** (501 MHz, DMSO)



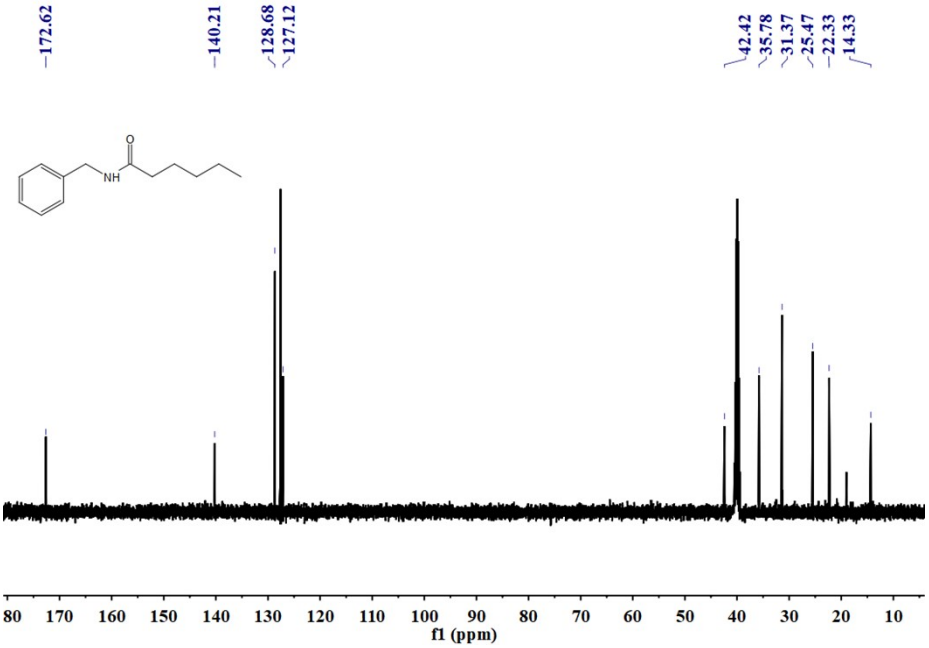
^{13}C NMR spectra of **15** (126 MHz, DMSO)



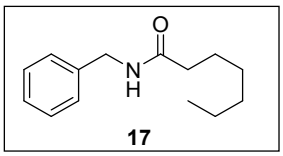
***N*-benzylhexanamide**[3-6]**:** ^{1}H NMR (501 MHz, DMSO) δ 8.31 (s, 1H), 7.33 – 7.24 (m, 3H), 7.23 (s, 2H), 4.26 (d, *J* = 6.0 Hz, 2H), 2.12 (d, *J* = 7.5 Hz, 2H), 1.52 (d, *J* = 7.3 Hz, 2H), 1.27 (dd, *J* = 10.7, 3.9 Hz, 4H), 0.86 (d, *J* = 7.0 Hz, 3H).
^{13}C NMR (126 MHz, DMSO) δ 172.62 (s), 140.21 (s), 128.68 (s), 127.12 (s), 42.42 (s), 35.78 (s), 31.37 (s), 25.47 (s), 22.33 (s), 14.33 (s).



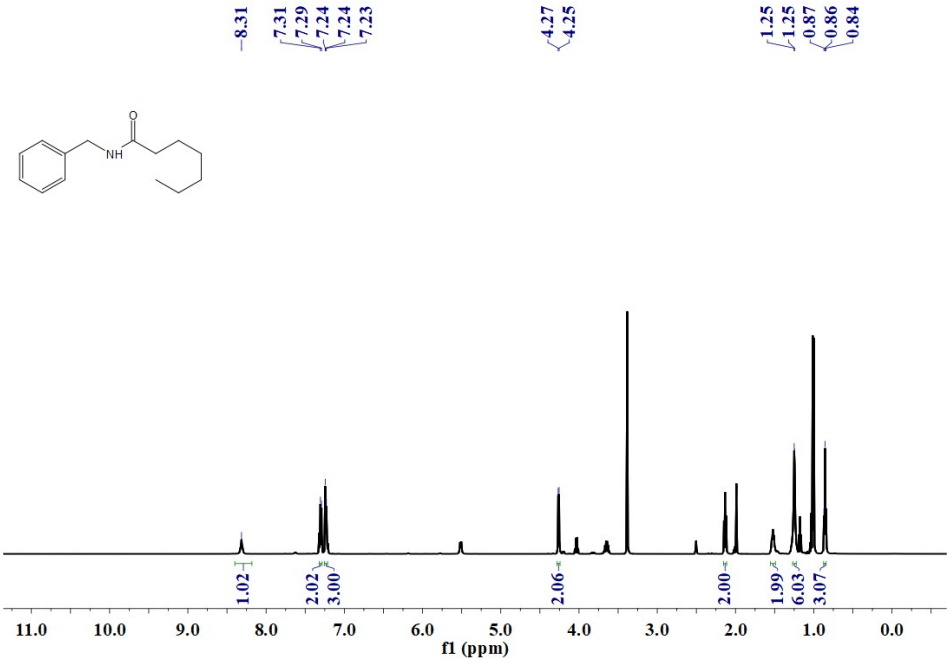
^{1}H NMR spectra of **16** (501 MHz, DMSO)



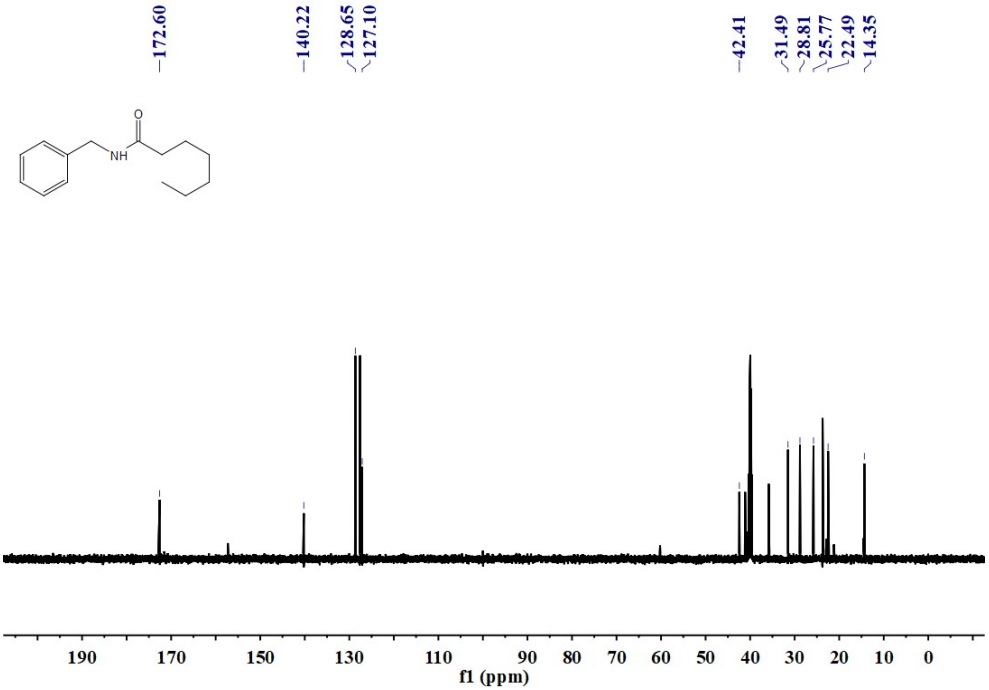
^{13}C NMR spectra of **16** (126 MHz, DMSO)



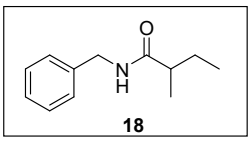
***N*-benzylheptanamide**[3-6]**:** ^{1}H NMR (501 MHz, DMSO) δ 8.31 (s, 1H), 7.30 (d, *J* = 7.2 Hz, 2H), 7.25 – 7.21 (m, 3H), 4.26 (d, *J* = 6.0 Hz, 2H), 2.13 (t, *J* = 7.4 Hz, 2H), 1.51 (dd, *J* = 14.3, 7.1 Hz, 2H), 1.25 (d, *J* = 1.9 Hz, 6H), 0.86 (t, *J* = 6.8 Hz, 3H).
^{13}C NMR (126 MHz, DMSO) δ 172.60 (s), 140.22 (s), 128.65 (s), 127.10 (s), 42.41 (s), 31.49 (s), 28.81 (s), 25.77 (s), 22.49 (s), 14.35 (s).



^{1}H NMR spectra of **17** (501 MHz, DMSO)

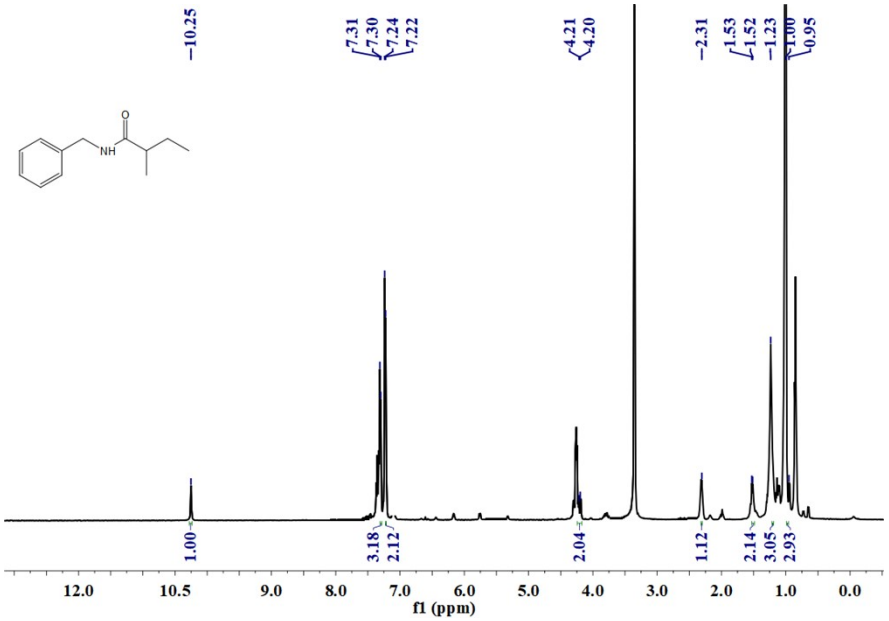


^{13}C NMR spectra of **17** (126 MHz, DMSO)

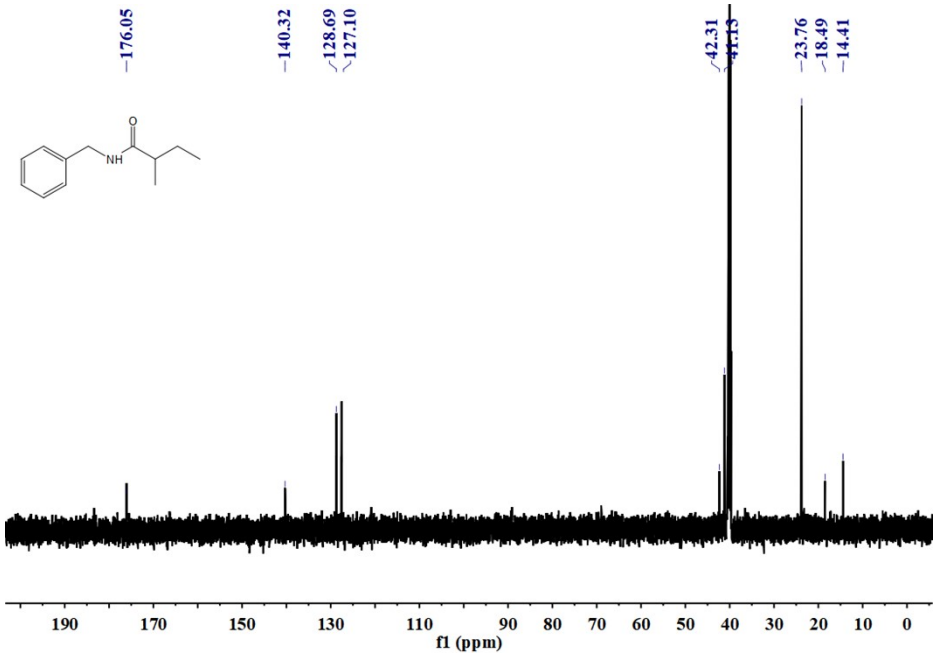


***N*-benzyl-2-methylbutanamide**[3-6]**:** ^{1}H NMR (501 MHz, DMSO) δ 10.25 (s, 1H), 7.30 (s, 3H), 7.22 – 7.22 (m, 2H), 4.21 (d, *J* = 8.9 Hz, 2H), 2.31 (s, 1H), 1.52 (d, *J* = 6.8 Hz, 2H), 1.21 – 1.19 (m, 3H), 0.96 (s, 3H).

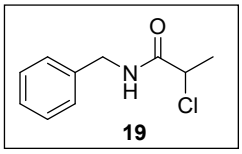
^{13}C NMR (126 MHz, DMSO) δ 176.05 (s), 140.32 (s), 128.69 (s), 127.10 (s), 42.31 (s), 41.13 (s), 23.76 (s), 18.49 (s), 14.41 (s).



^{1}H NMR spectra of **18** (501 MHz, DMSO)

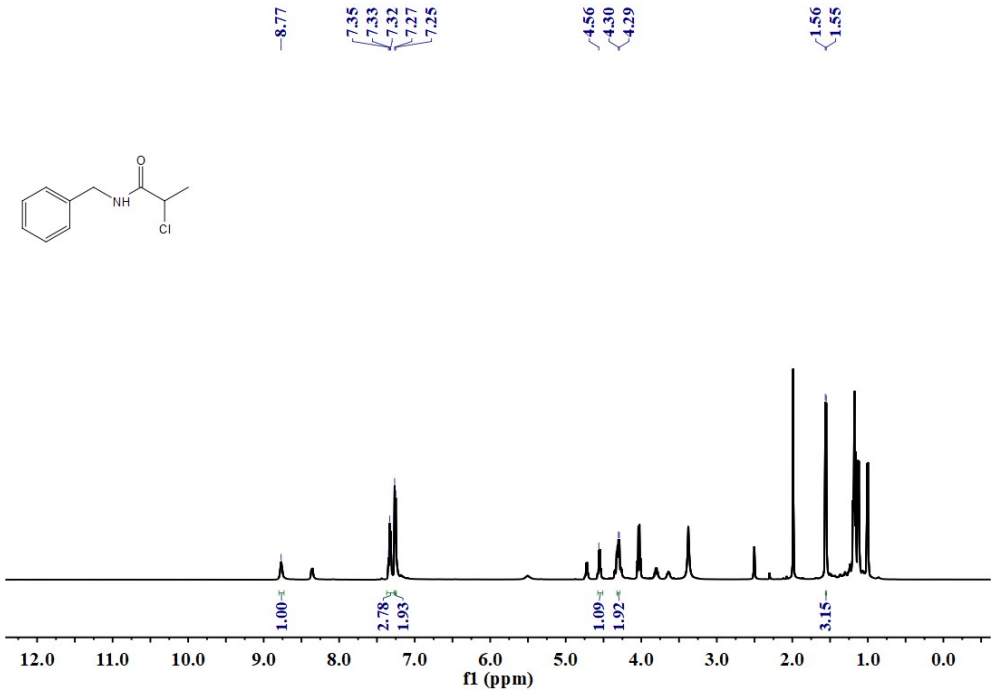


^{13}C NMR spectra of **18** (126 MHz, DMSO)

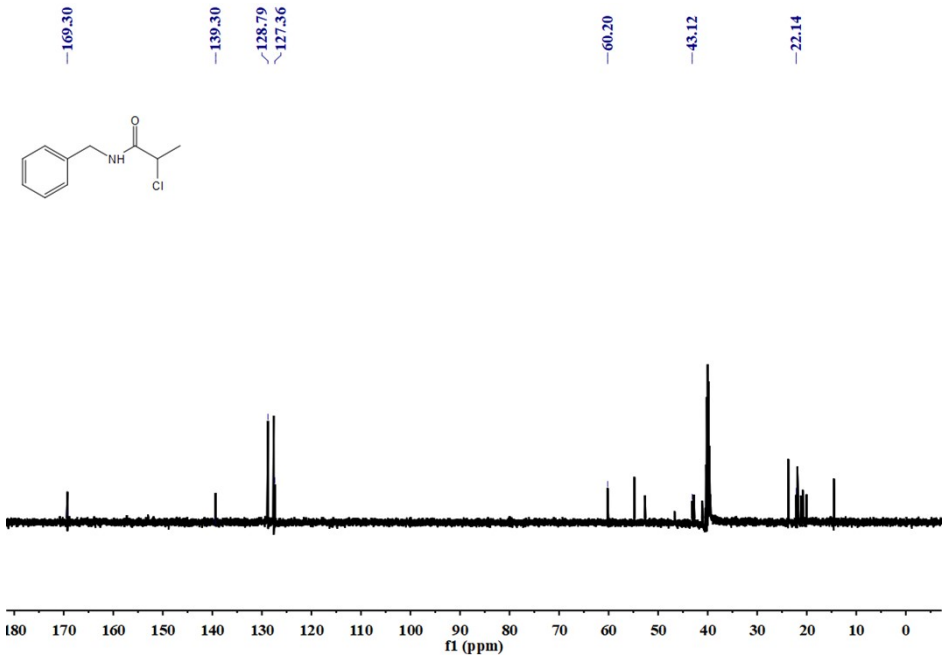


***N*-benzyl-2-chloropropanamide[3-6]:** ^{1}H NMR (501 MHz, DMSO) δ 8.77 (s, 1H), 7.33 (t, *J* = 7.5 Hz, 3H), 7.25 (s, 2H), 4.56 (s, 1H), 4.30 (d, *J* = 6.1 Hz, 2H), 1.55 (s, 3H).

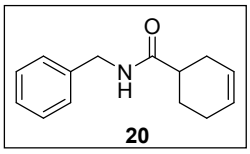
^{13}C NMR (126 MHz, DMSO) δ 169.30 (s), 139.33 (s), 128.79 (s), 127.36 (s), 60.20 (s), 43.12 (s), 22.14 (s).



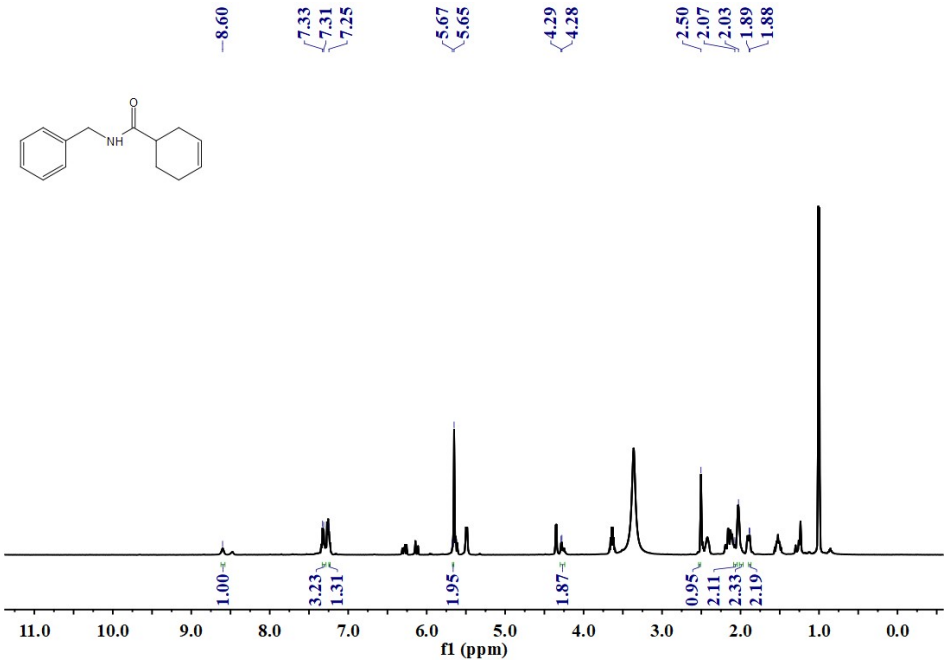
^{1}H NMR spectra of **19** (501 MHz, DMSO)



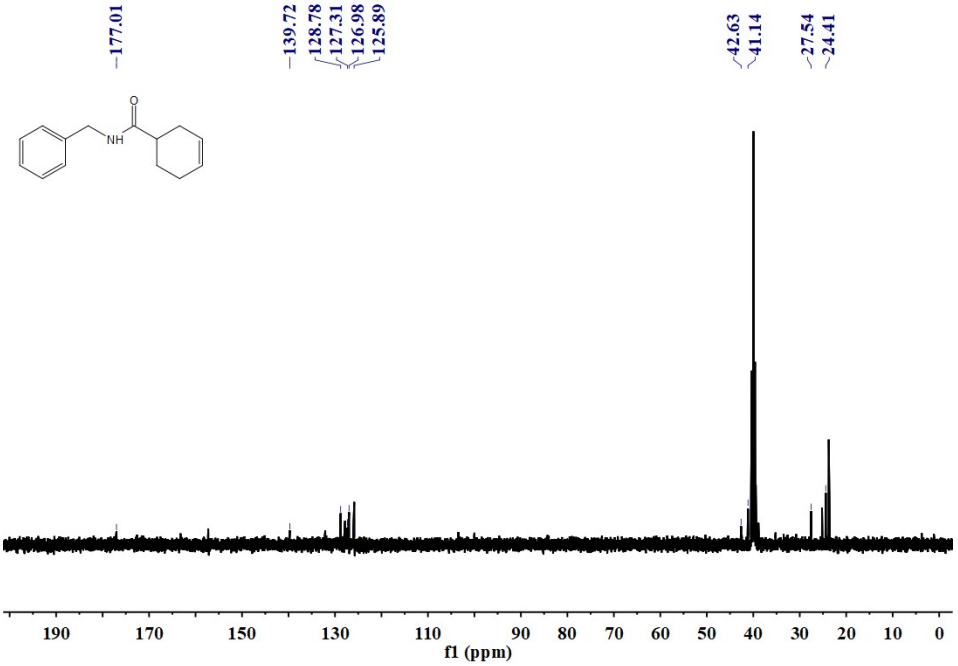
^{13}C NMR spectra of **19** (126 MHz, DMSO)



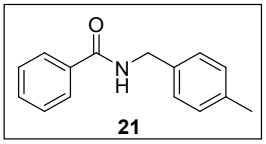
***N*-benzylcyclohex-3-ene-1-carboxamide[3-6]:** ^{1}H NMR (501 MHz, DMSO) δ 8.60 (s, 1H), 7.32 (d, *J* = 7.3 Hz, 3H), 7.25 (s, 1H), 5.65 (s, 2H), 4.28 (d, *J* = 5.9 Hz, 2H), 2.51 (s, 1H), 2.07 (s, 2H), 1.99 (s, 2H), 1.89 (d, *J* = 2.9 Hz, 2H).
^{13}C NMR (126 MHz, DMSO) δ 177.01 (s), 139.72 (s), 128.78 (s), 127.31 (s), 126.98 (s), 125.89 (s), 42.63 (s), 41.14 (s), 27.54 (s), 24.41 (s).



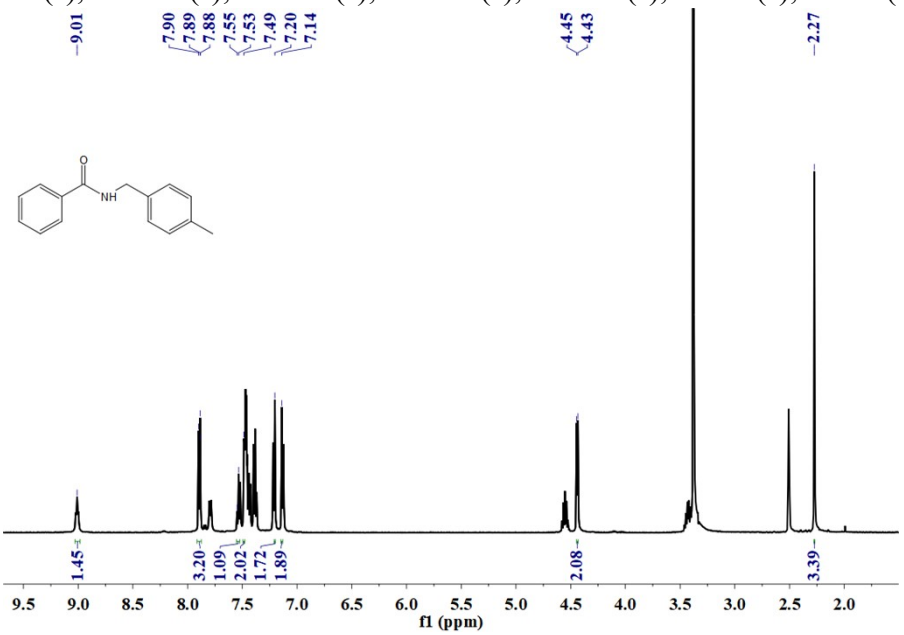
^{1}H NMR spectra of **20** (501 MHz, DMSO)



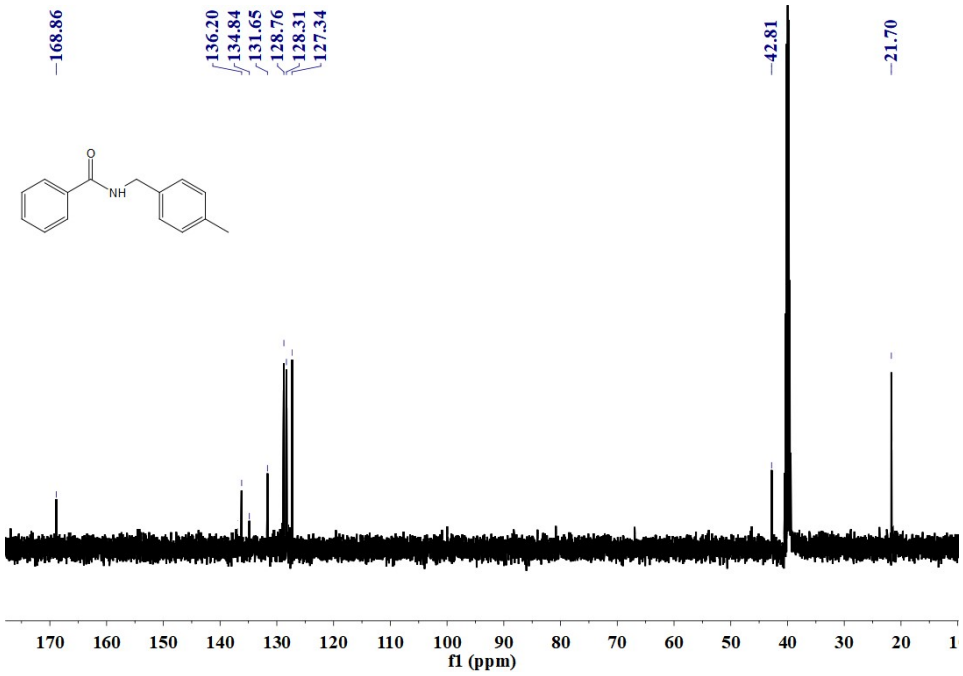
^{13}C NMR spectra of **20** (126 MHz, DMSO)



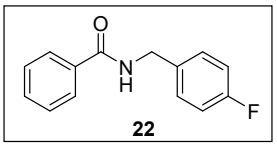
***N*-(4-methylbenzyl)benzamide[3-6]:** ^{1}H NMR (501 MHz, DMSO) δ 9.01 (s, 1H), 7.91 – 7.87 (m, 3H), 7.54 (d, *J* = 7.2 Hz, 1H), 7.49 (s, 2H), 7.20 (s, 2H), 7.14 (s, 2H), 4.44 (d, *J* = 6.0 Hz, 2H), 2.27 (s, 3H). ^{13}C NMR (126 MHz, DMSO) δ 168.86 (s), 136.20 (s), 134.84 (s), 131.65 (s), 128.76 (s), 128.31 (s), 127.34 (s), 42.81 (s), 21.70 (s).



^{1}H NMR spectra of **21** (501 MHz, DMSO)

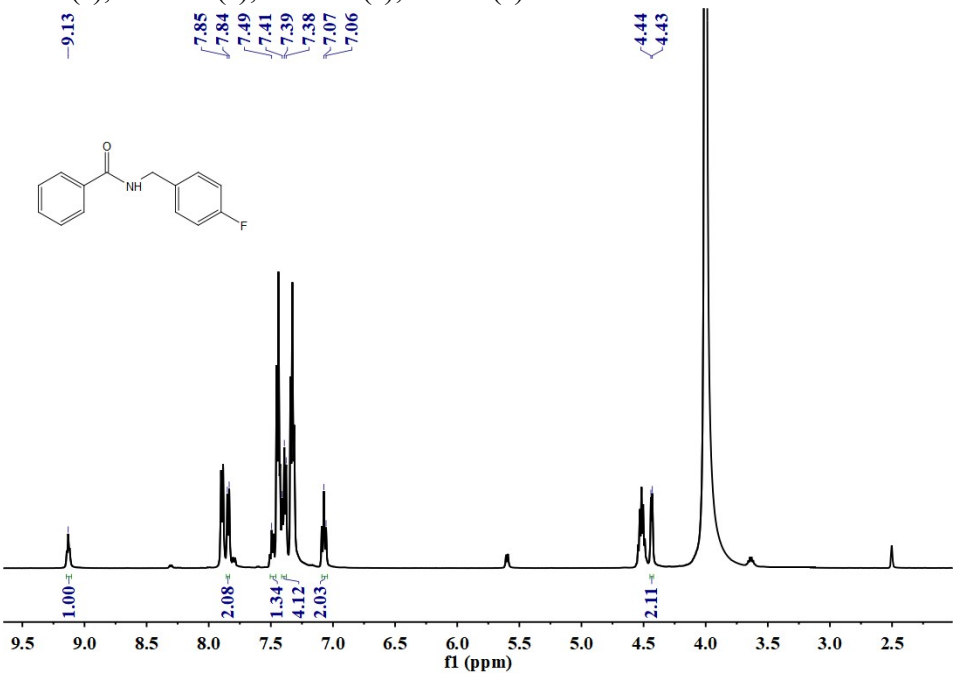


^{13}C NMR spectra of **21** (126 MHz, DMSO)

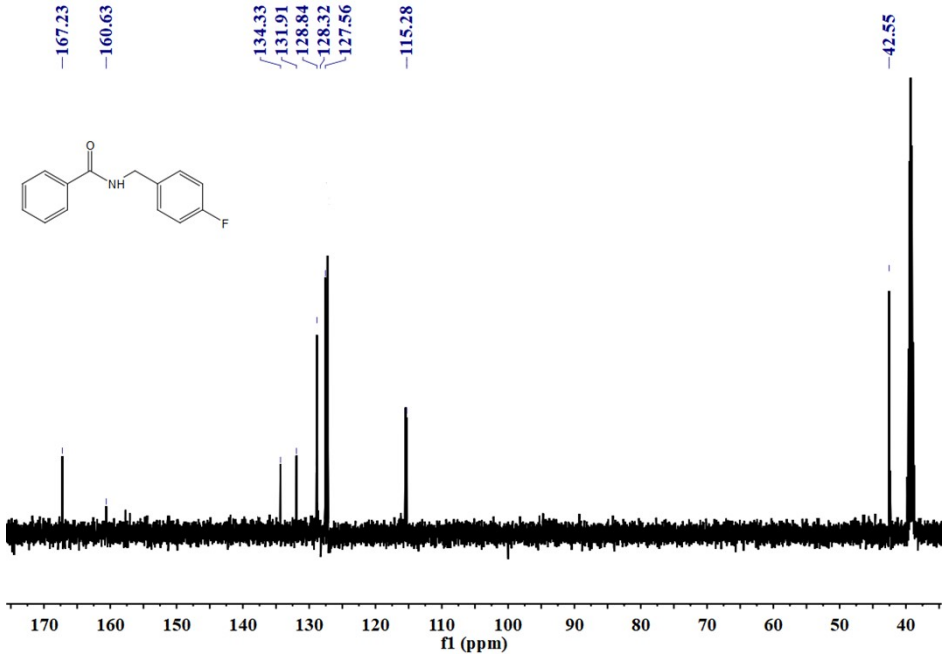


***N*-(4-fluorobenzyl)benzamide**[3-6]**:** ^{1}H NMR (501 MHz, DMSO) δ 9.13 (s, 1H), 7.84 (d, *J* = 7.4 Hz, 2H), 7.49 (s, 1H), 7.41 – 7.38 (m, 4H), 7.06 (d, *J* = 8.7 Hz, 2H), 4.44 (d, *J* = 5.8 Hz, 2H).

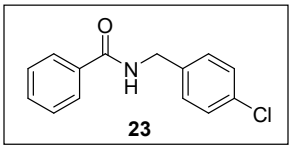
^{13}C NMR (126 MHz, DMSO) δ 167.23 (s), 160.63 (s), 134.33 (s), 131.91 (s), 128.84 (s), 128.32 (s), 127.56 (s), 115.28 (s), 42.55 (s).



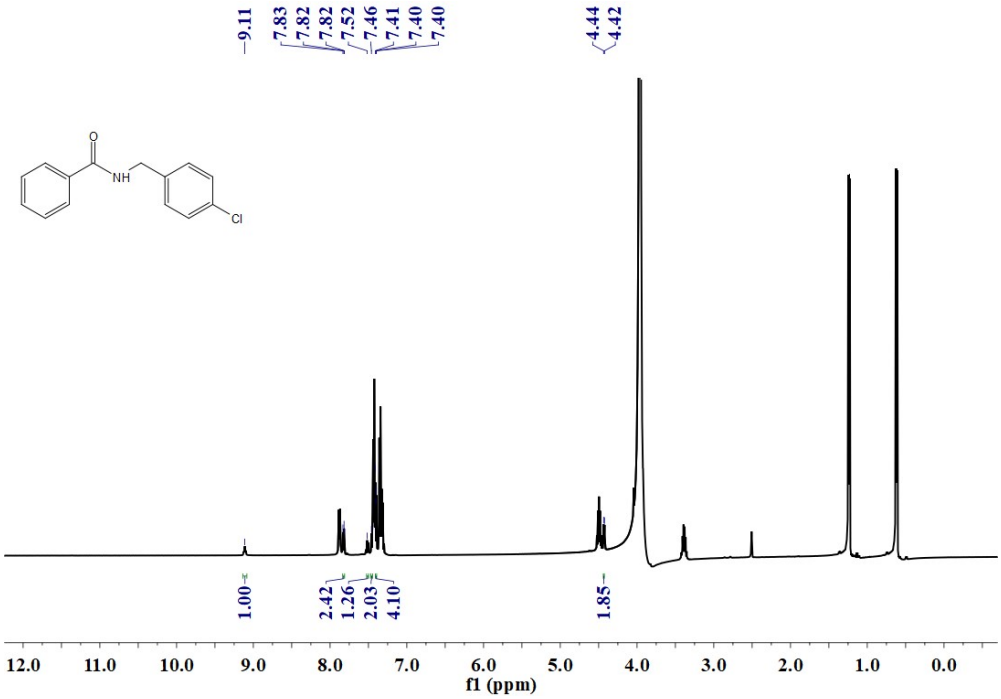
^{1}H NMR spectra of **22** (501 MHz, DMSO)



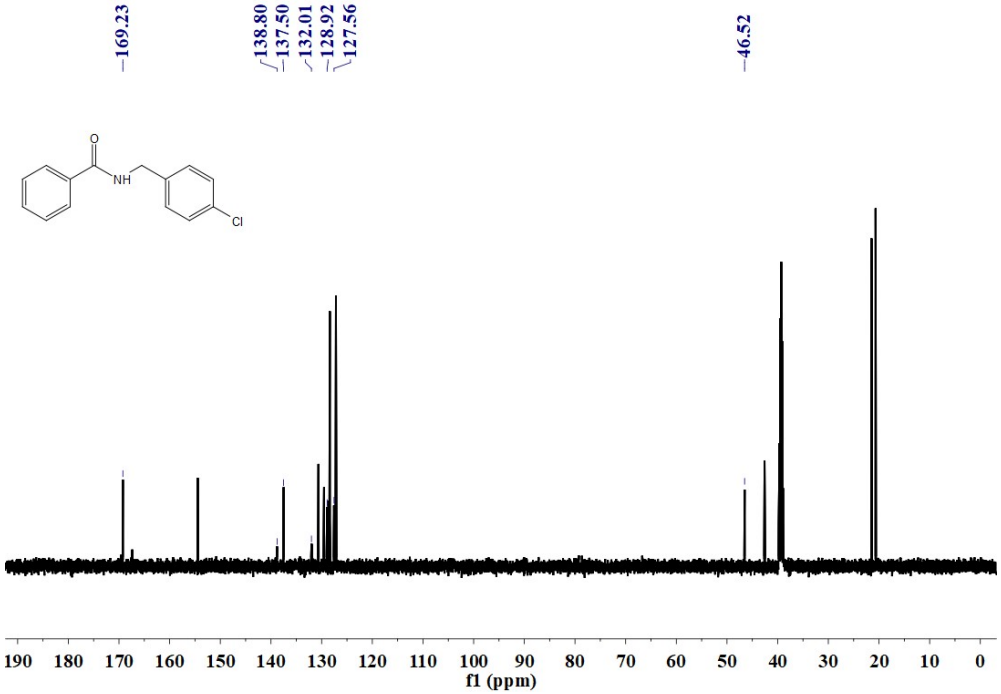
^{13}C NMR spectra of **22** (126 MHz, DMSO)



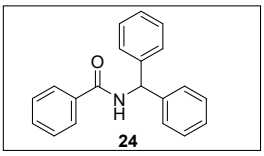
***N*-(4-chlorobenzyl)benzamide**[3-6]**:** ^{1}H NMR (501 MHz, DMSO) δ 9.11 (s, 1H), 7.84 – 7.81 (m, 2H), 7.52 (s, 1H), 7.46 (s, 2H), 7.40 (d, *J* = 1.3 Hz, 4H), 4.44 (s, 2H). ^{13}C NMR (126 MHz, DMSO) δ 169.23 (s), 138.80 (s), 137.50 (s), 132.01 (s), 128.92 (s), 127.56 (s), 46.52 (s).



^{1}H NMR spectra of **23** (501 MHz, DMSO)

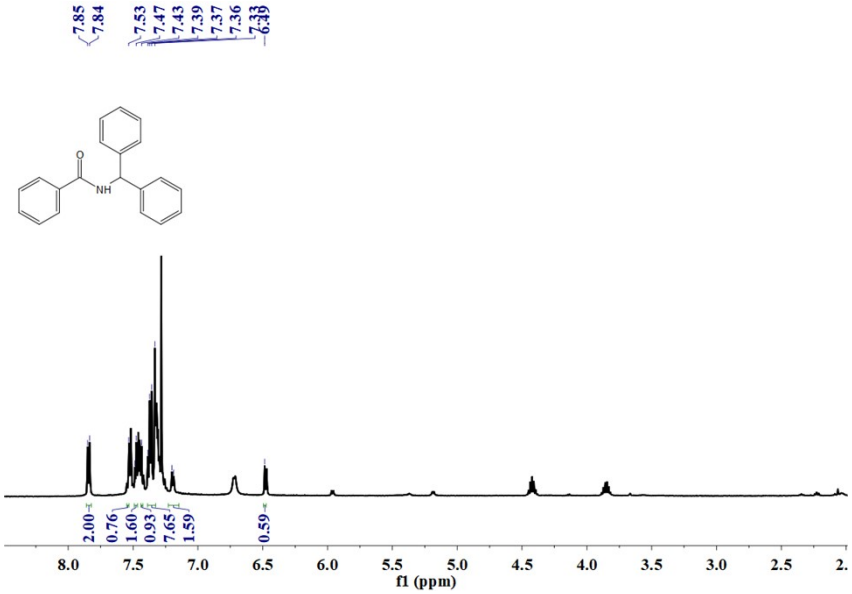


^{13}C NMR spectra of **23** (126 MHz, DMSO)

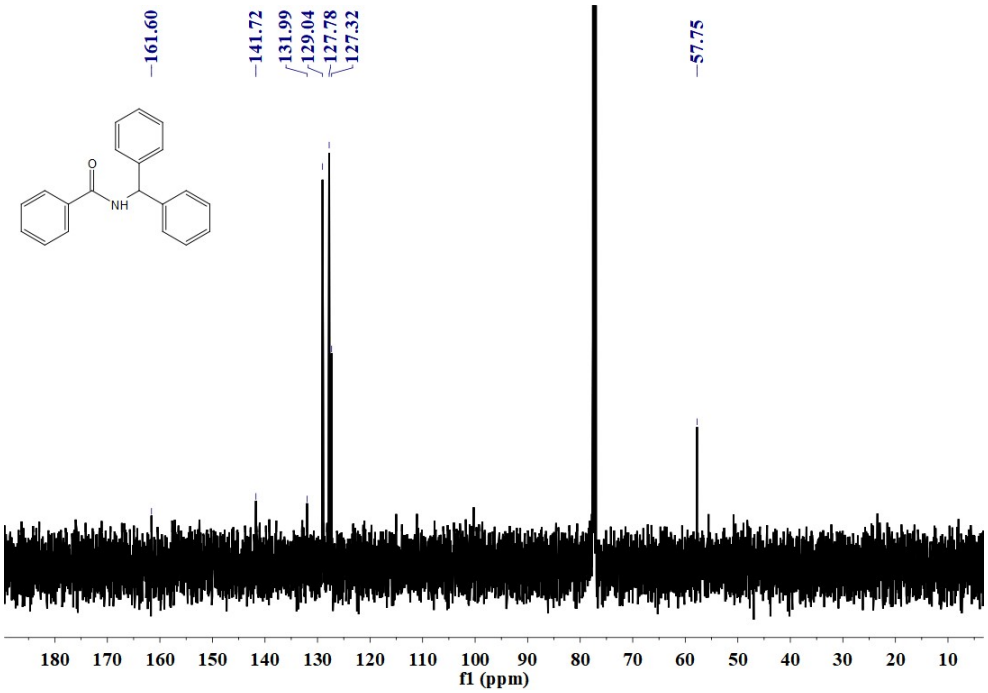


***N*-benzhydrylbenzamide**[3-6]**:** ^{1}H NMR (501 MHz, $CDCl_3$) δ 7.84 (d, *J* = 7.1 Hz, 2H), 7.53 (s, 1H), 7.48 (d, *J* = 5.5 Hz, 2H), 7.43 (s, 1H), 7.36 (dd, *J* = 17.3, 10.0 Hz, 8H), 7.19 (d, *J* = 7.0 Hz, 2H), 6.49 (s, 1H).

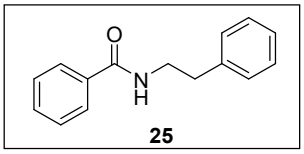
^{13}C NMR (126 MHz, $CDCl_3$) δ 161.60 (s), 141.72 (s), 131.99 (s), 129.04 (s), 127.78 (s), 127.32 (s), 57.75 (s).



^{1}H NMR spectra of **24** (501 MHz, $CDCl_3$)

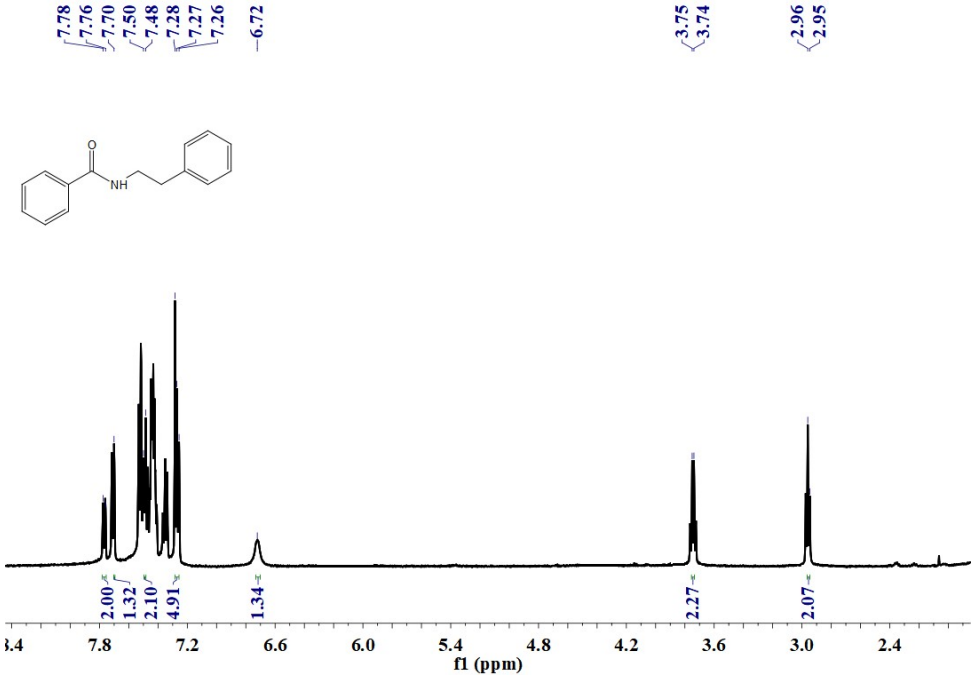


^{13}C NMR spectra of **24** (126 MHz, $CDCl_3$)

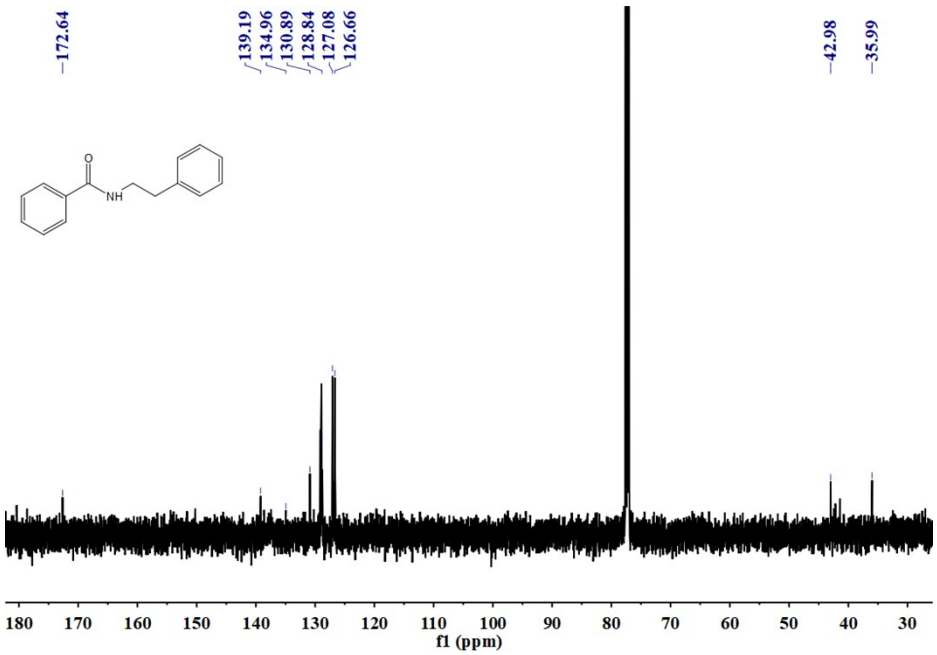


***N*-phenethylbenzamide**[3-6]**:** ^{1}H NMR (501 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.4 Hz, 2H), 7.70 (s, 1H), 7.48 (s, 2H), 7.28 (d, *J* = 6.4 Hz, 5H), 6.72 (s, 1H), 3.74 (d, *J* = 6.0 Hz, 2H), 2.95 (d, *J* = 6.9 Hz, 2H).

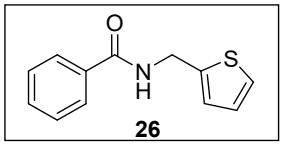
^{13}C NMR (126 MHz, $CDCl_3$) δ 172.64 (s), 139.19 (s), 134.96 (s), 130.89 (s), 128.84 (s), 127.08 (s), 126.66 (s), 42.98 (s), 35.99 (s).



^{1}H NMR spectra of **25** (501 MHz, $CDCl_3$)



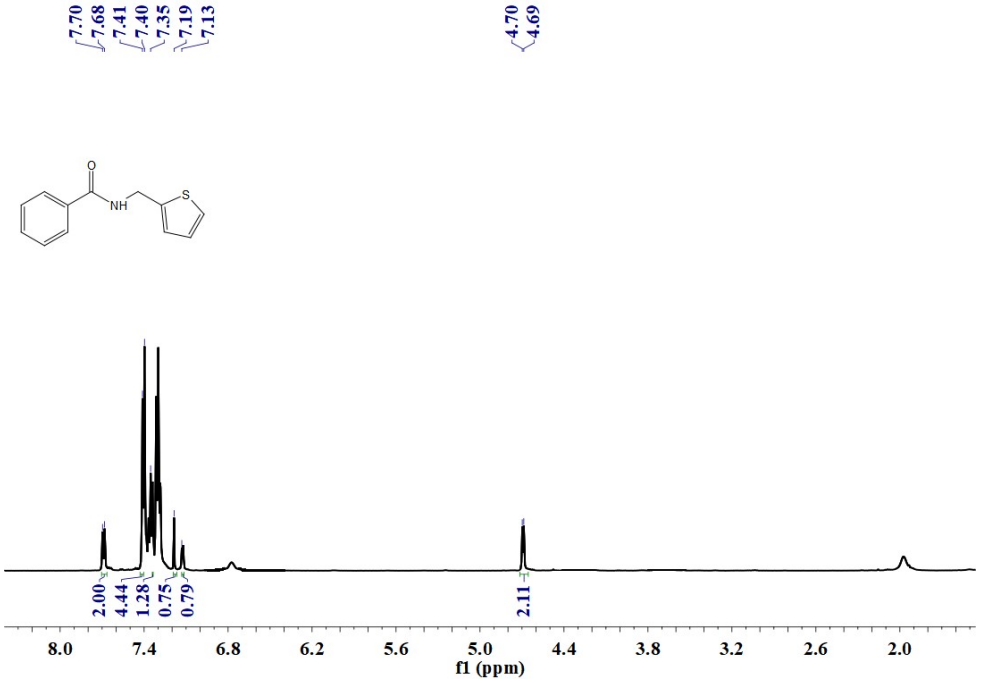
^{13}C NMR spectra of **25** (126 MHz, $CDCl_3$)



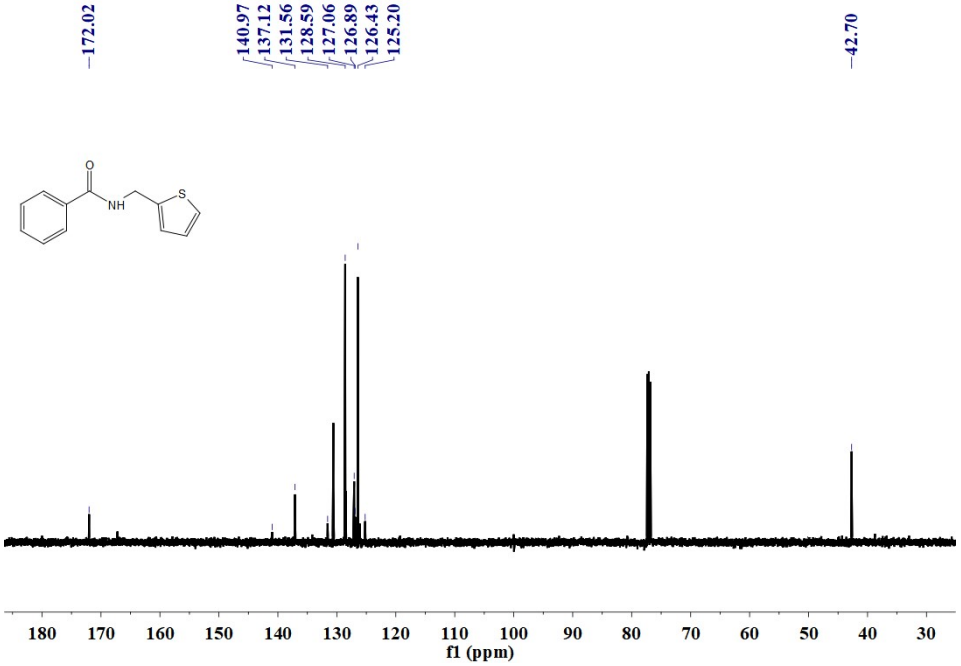
26

***N*-(thiophen-2-ylmethyl)benzamide[3-6]:** ^{1}H NMR (501 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.4 Hz, 2H), 7.41 (s, 4H), 7.34 (s, 1H), 7.19 (s, 1H), 7.13 (s, 1H), 4.69 (d, *J* = 5.5 Hz, 2H).

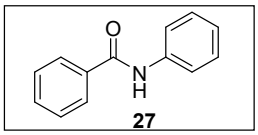
^{13}C NMR (126 MHz, $CDCl_3$) δ 172.02 (s), 140.97 (s), 137.12 (s), 131.56 (s), 128.59 (s), 127.06 (s), 126.89 (s), 126.43 (s), 125.20 (s), 42.70 (s).



^{1}H NMR spectra of **26** (501 MHz, $CDCl_3$)



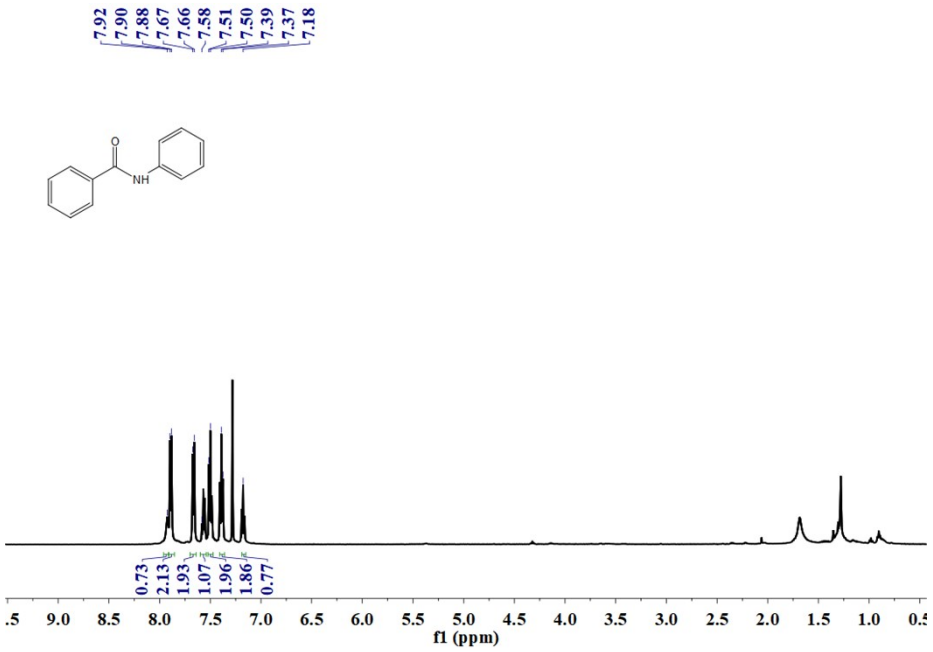
^{13}C NMR spectra of **26** (126 MHz, $CDCl_3$)



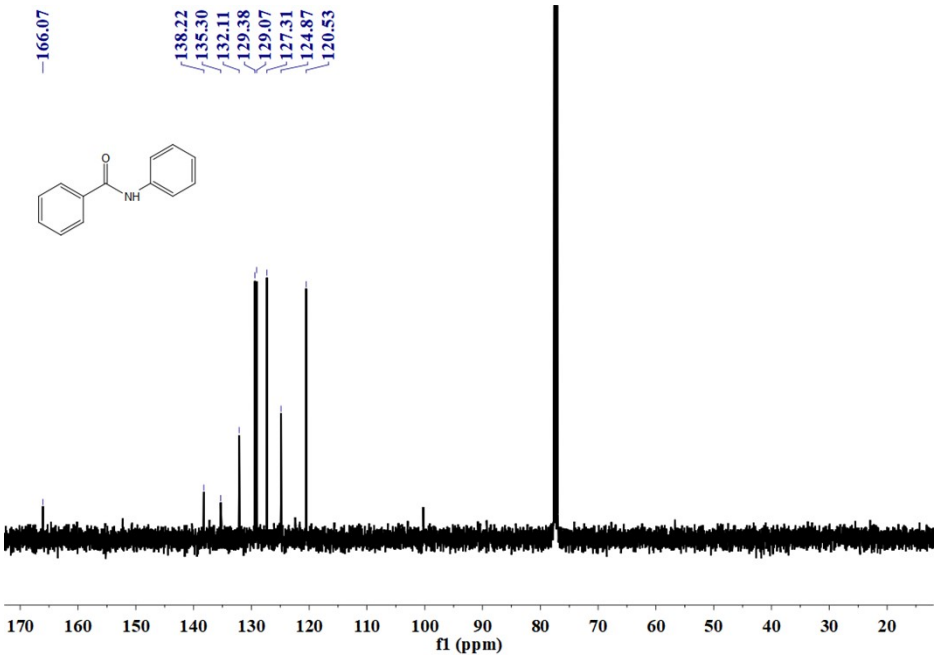
27

***N*-phenylbenzamide**[3-6]**:** ^{1}H NMR (501 MHz, $CDCl_3$) δ 7.92 (s, 1H), 7.89 (d, *J* = 7.3 Hz, 2H), 7.67 (d, *J* = 7.9 Hz, 2H), 7.58 (s, 1H), 7.51 (d, *J* = 7.7 Hz, 2H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.18 (s, 1H).

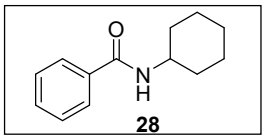
^{13}C NMR (126 MHz, $CDCl_3$) δ 166.07 (s), 138.22 (s), 135.30 (s), 132.11 (s), 129.38 (s), 129.07 (s), 127.31 (s), 124.87 (s), 120.53 (s).



^{1}H NMR spectra of **27** (501 MHz, $CDCl_3$)



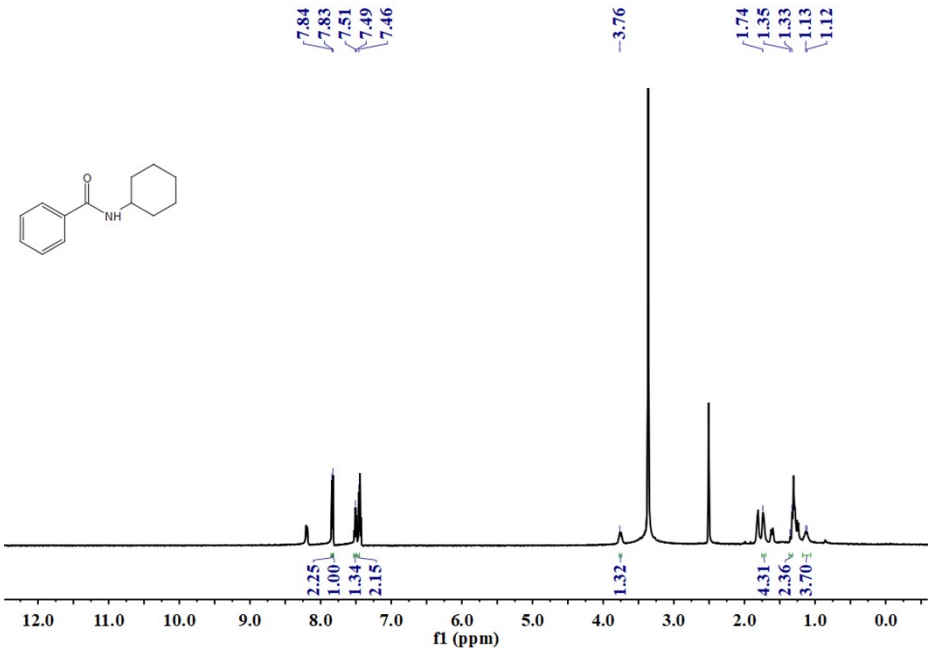
^{13}C NMR spectra of **27** (126 MHz, $CDCl_3$)



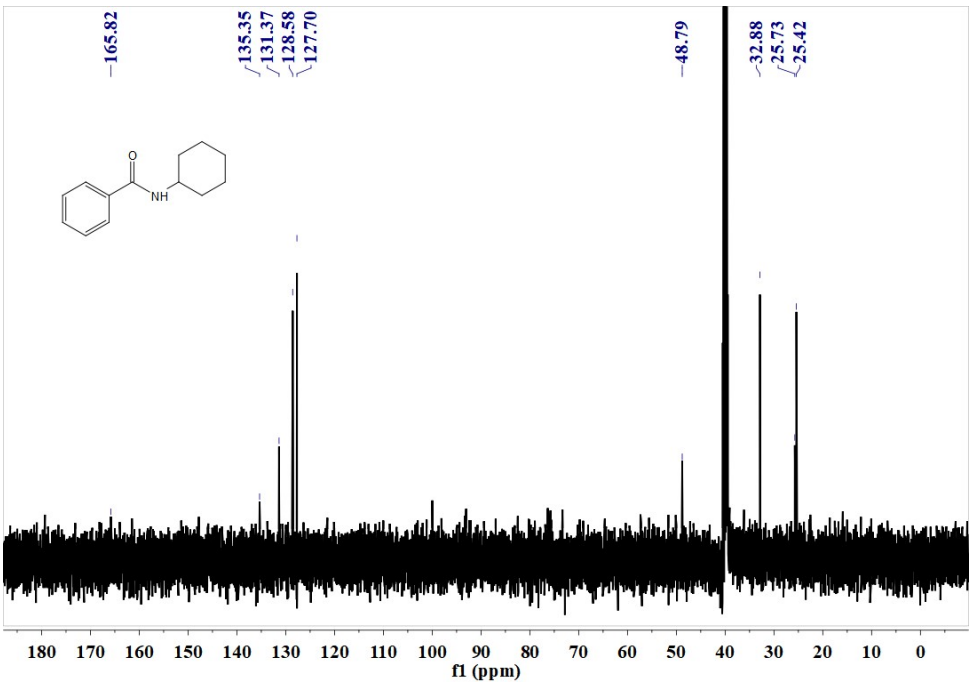
28

***N*-cyclohexylbenzamide**[3-6]**:** ^{1}H NMR (501 MHz, DMSO) δ 7.84 (s, 2H), 7.82 (s, 1H), 7.51 (s, 1H), 7.48 (d, *J* = 16.9 Hz, 2H), 3.76 (s, 1H), 1.74 (s, 4H), 1.34 (d, *J* = 13.2 Hz, 2H), 1.12 (d, *J* = 9.1 Hz, 4H).

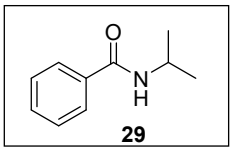
^{13}C NMR (126 MHz, DMSO) δ 165.82 (s), 135.35 (s), 131.37 (s), 128.58 (s), 127.70 (s), 48.79 (s), 32.88 (s), 25.73 (s), 25.42 (s).



^{1}H NMR spectra of **28** (501 MHz, DMSO)

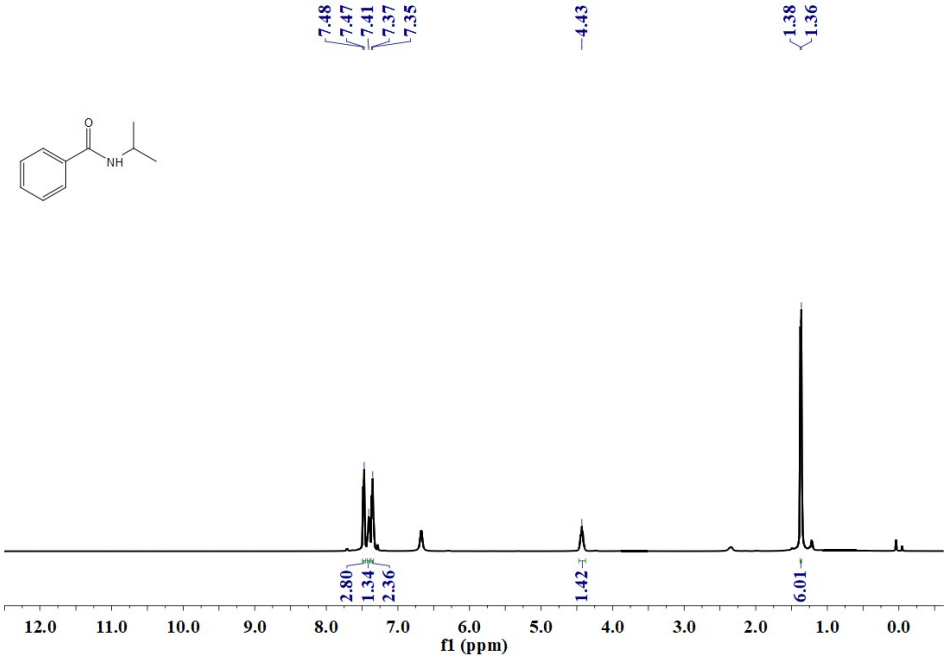


^{13}C NMR spectra of **28** (126 MHz, DMSO)

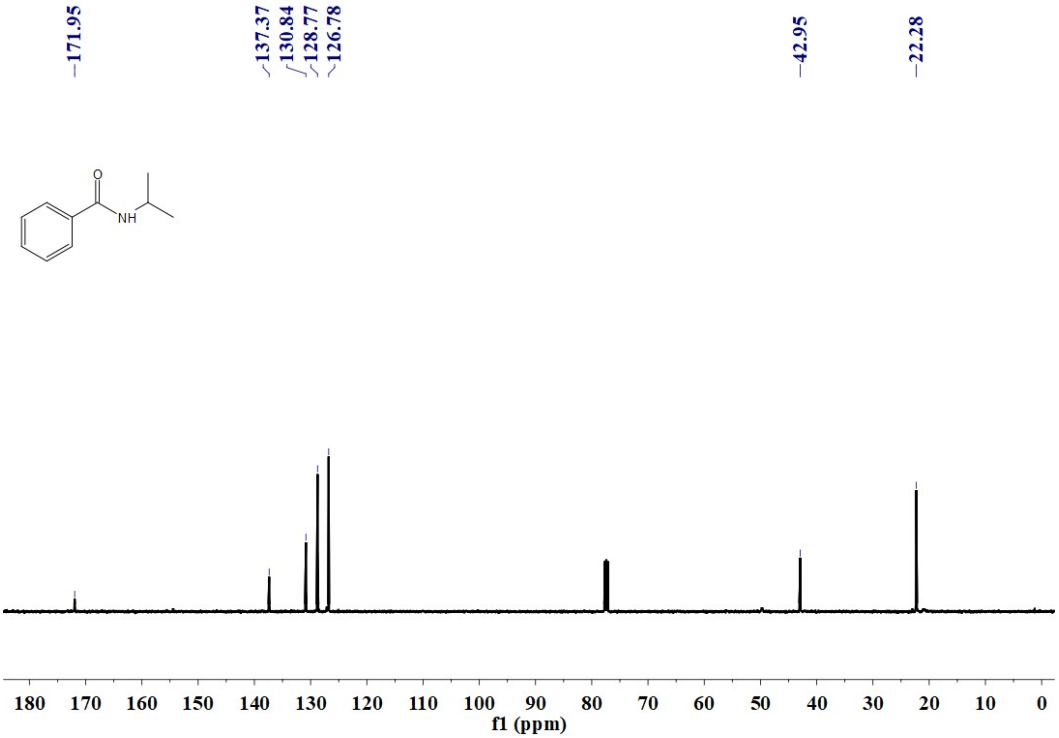


29

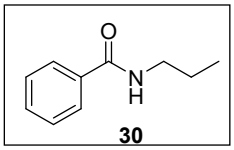
***N*-isopropylbenzamide**[3-6]**:** ^{1}H NMR (501 MHz, $CDCl_3$) δ 7.48 (d, *J* = 7.1 Hz, 3H), 7.41 (s, 1H), 7.36 (d, *J* = 7.4 Hz, 2H), 4.43 (s, 1H), 1.37 (d, *J* = 6.7 Hz, 6H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 171.95 (s), 137.37 (s), 130.84 (s), 128.77 (s), 126.78 (s), 42.95 (s), 22.28 (s).



^{1}H NMR spectra of **29** (501 MHz, $CDCl_3$)

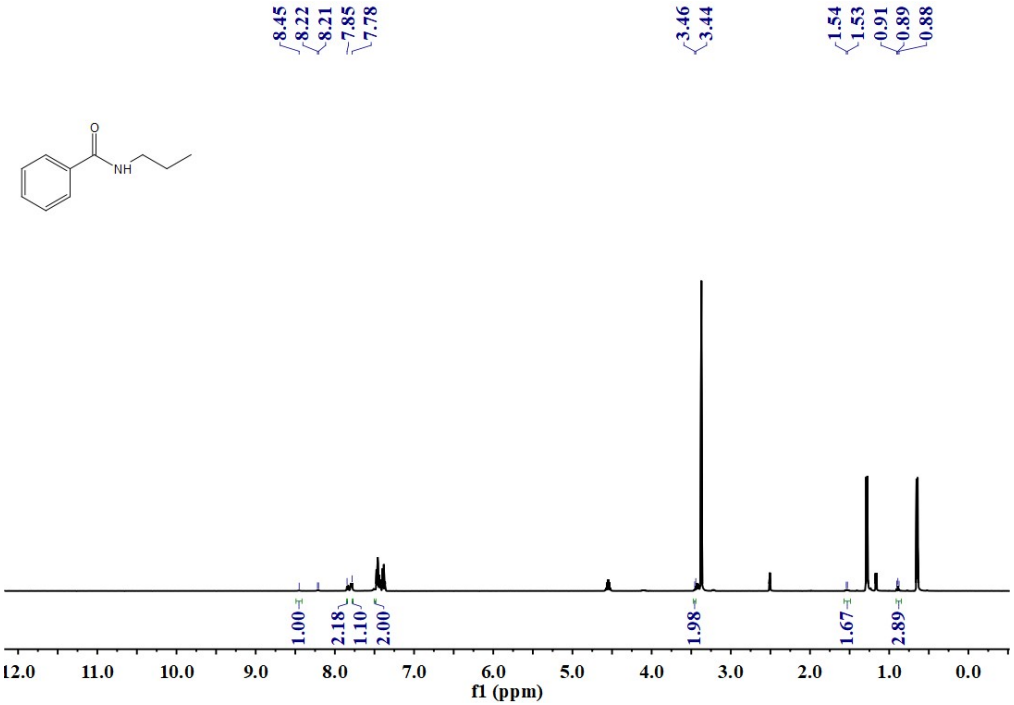


^{13}C NMR spectra of **29** (126 MHz, $CDCl_3$)

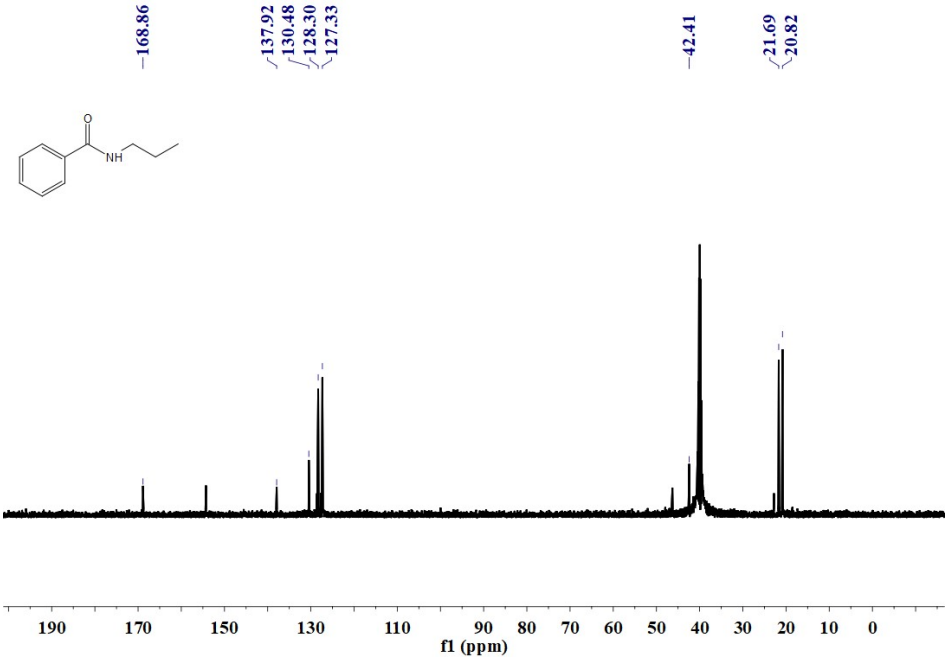


***N*-propylbenzamide**[3-6]**:** ^{1}H NMR (501 MHz, DMSO) δ 8.45 (s, 1H), 7.85 (s, 2H), 7.78 – 7.77 (m, 1H), 7.49 (d, *J* = 5.2 Hz, 2H), 3.45 (d, *J* = 6.7 Hz, 2H), 1.53 (d, *J* = 7.2 Hz, 2H), 0.89 (t, *J* = 7.4 Hz, 3H).

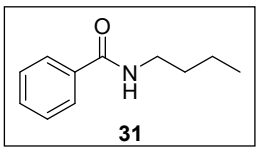
^{13}C NMR (126 MHz, DMSO) δ 168.86 (s), 137.92 (s), 130.48 (s), 128.30 (s), 127.33 (s), 42.41 (s), 21.69 (s), 20.82 (s).



^{1}H NMR spectra of **30** (501 MHz, DMSO)

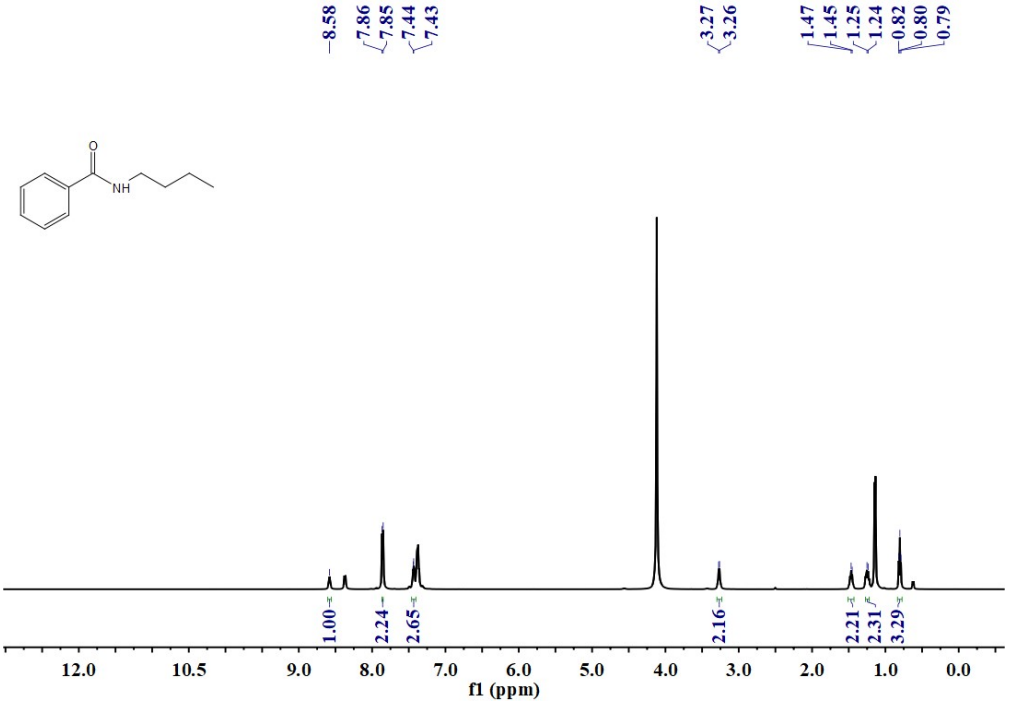


^{13}C NMR spectra of **30**(126 MHz, DMSO)

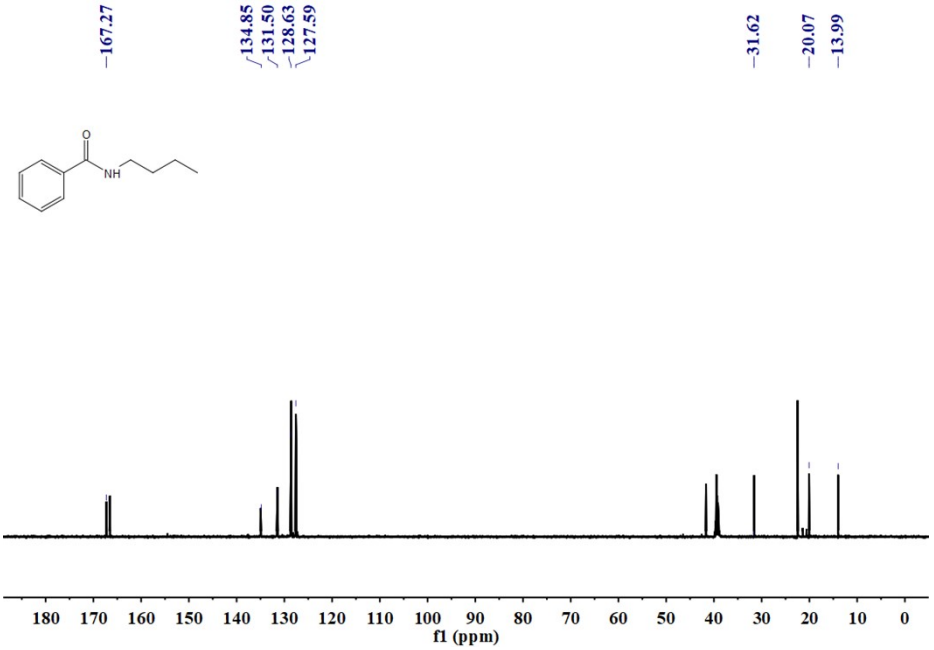


***N*-butylbenzamide**[3-6]**:** ^{1}H NMR (501 MHz, DMSO) δ 8.58 (s, 1H), 7.86 (d, *J* = 7.4 Hz, 2H), 7.44 (d, *J* = 3.2 Hz, 3H), 3.27 (d, *J* = 6.2 Hz, 2H), 1.46 (d, *J* = 7.2 Hz, 2H), 1.24 (d, *J* = 7.6 Hz, 2H), 0.80 (t, *J* = 7.3 Hz, 3H).

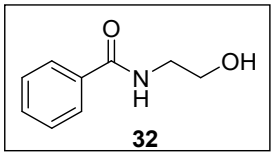
^{13}C NMR (126 MHz, DMSO) δ 167.27 (s), 134.85 (s), 131.50 (s), 128.63 (s), 127.59 (s), 31.58 (s), 20.07 (s), 13.99 (s).



^{1}H NMR spectra of **31** (501 MHz, DMSO)



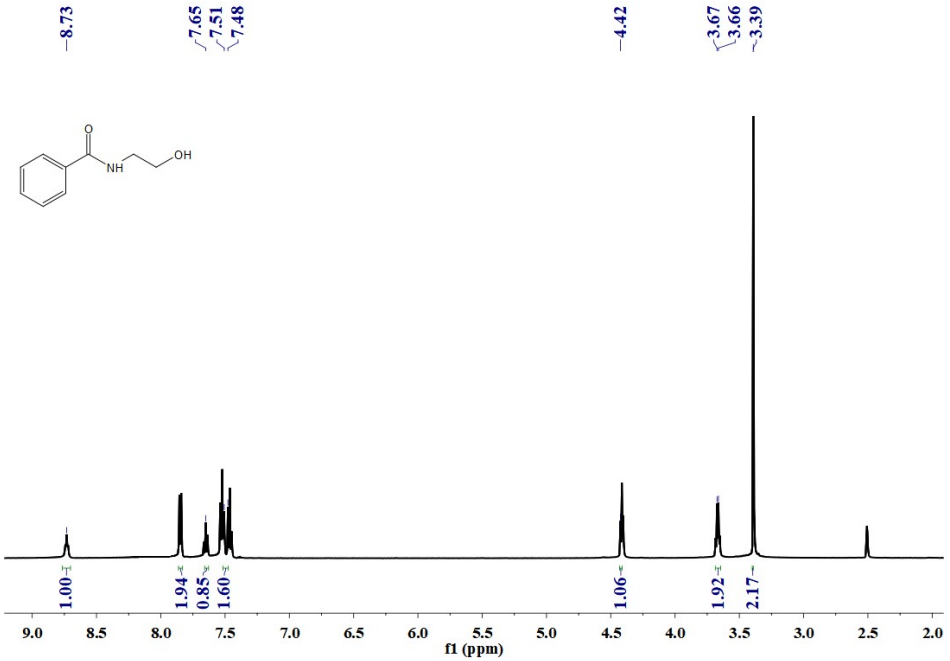
^{13}C NMR spectra of **31** (126 MHz, DMSO)



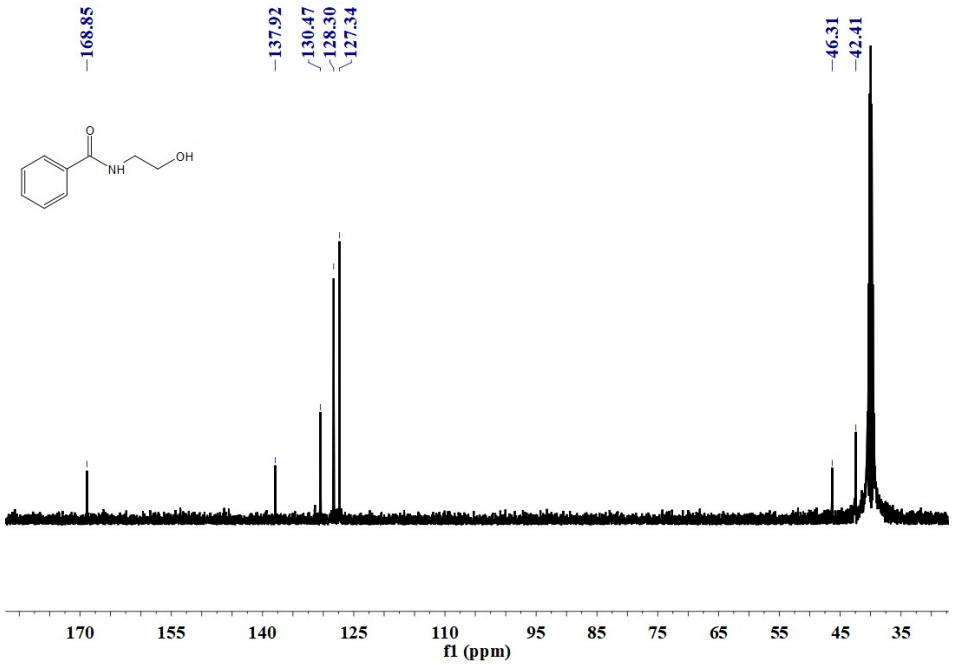
32

***N*-(2-hydroxyethyl)benzamide[3-6]:** ^{1}H NMR (501 MHz, DMSO) δ 8.73 (s, 1H), 7.85 (d, *J* = 7.5 Hz, 2H), 7.65 (s, 1H), 7.49 (d, *J* = 14.3 Hz, 2H), 4.42 (s, 1H), 3.67 (d, *J* = 5.6 Hz, 2H), 3.39 (s, 2H).

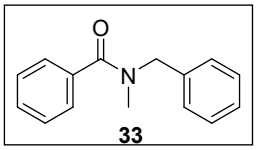
^{13}C NMR (126 MHz, DMSO) δ 168.85 (s), 137.92 (s), 130.47 (s), 128.30 (s), 127.34 (s), 46.31 (s), 42.41 (s).



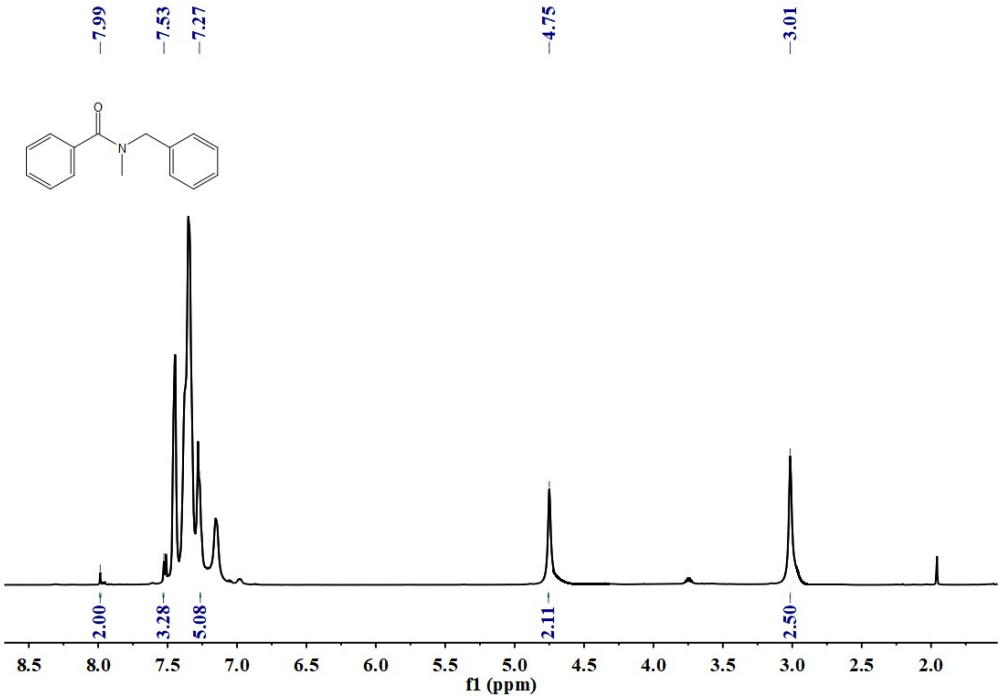
^{1}H NMR spectra of **32** (501 MHz, DMSO)



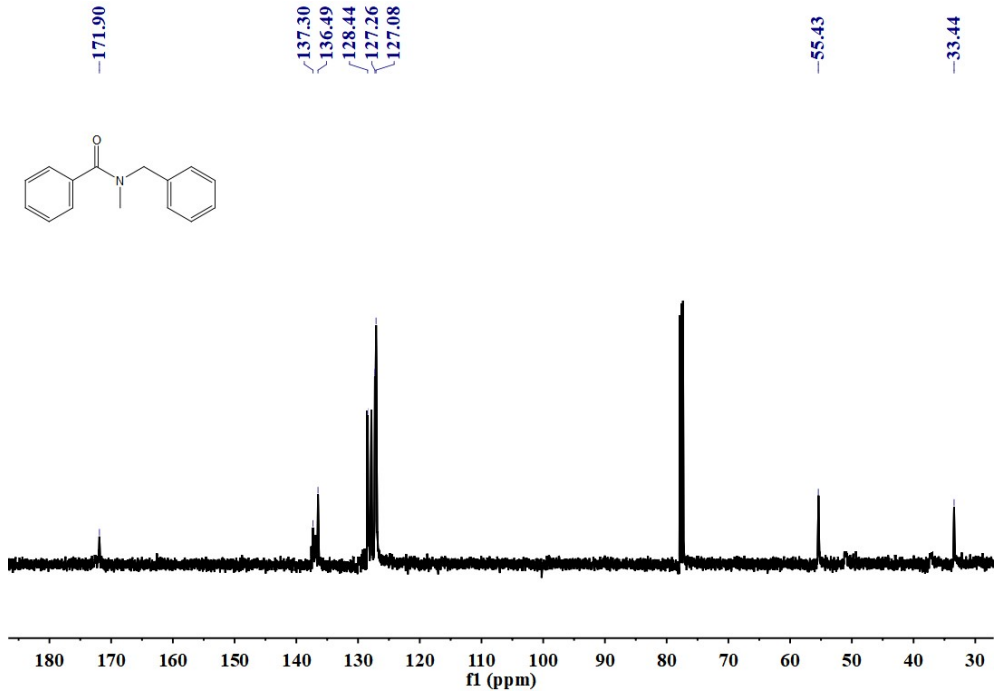
^{13}C NMR spectra of **32** (126 MHz, DMSO)



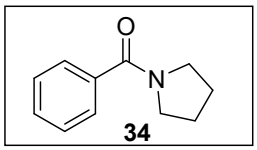
***N*-benzyl-N-methylbenzamide**[3-6]**:** ^{1}H NMR (501 MHz, $CDCl_3$) δ 7.99 (s, 2H), 7.53 (s, 3H), 7.27 – 7.26 (m, 5H), 4.76 (s, 2H), 3.01 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 171.90 (s), 137.30 (s), 136.49 (s), 128.44 (s), 127.17 (d, J = 21.6 Hz), 126.97 (s), 55.43 (s), 33.44 (s).



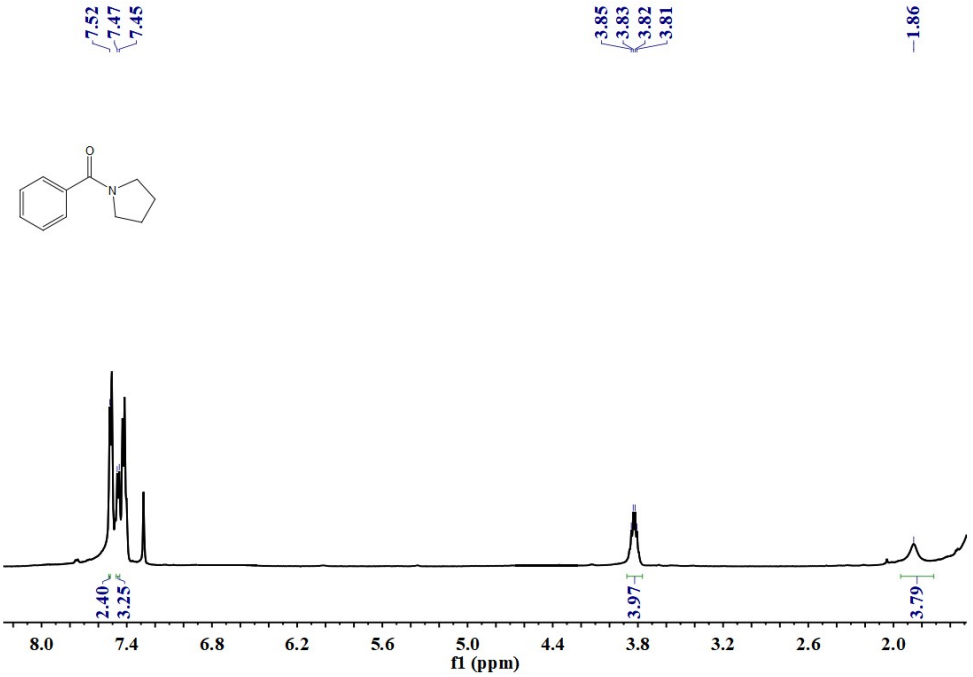
^{1}H NMR spectra of **33** (501 MHz, $CDCl_3$)



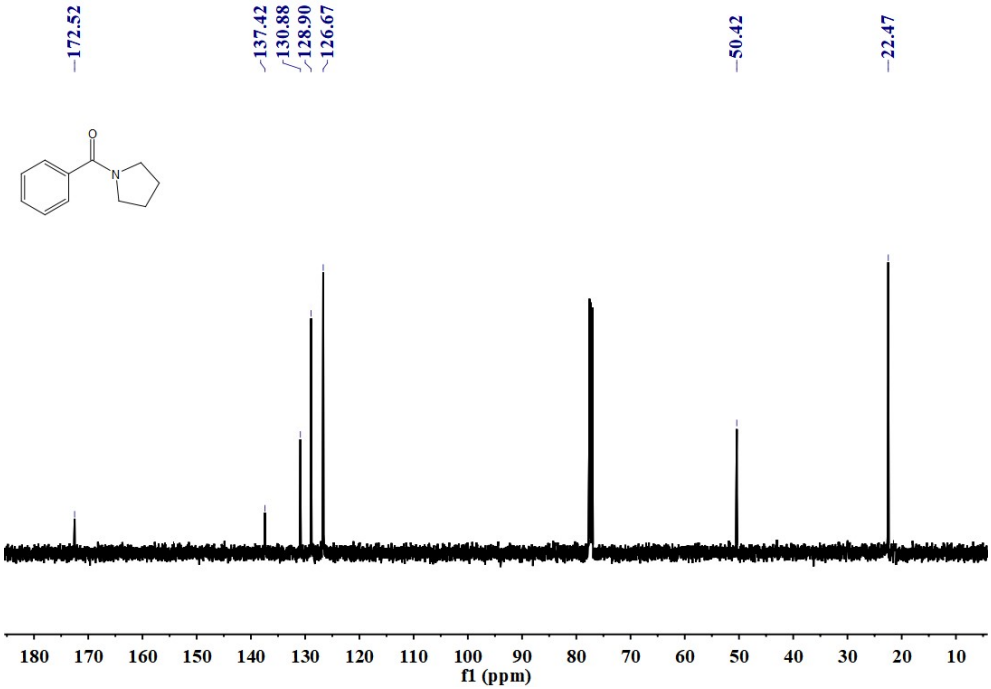
^{13}C NMR spectra of **33** (126 MHz, $CDCl_3$)



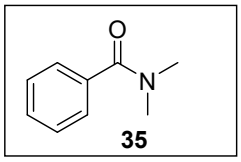
phenyl(pyrrolidin-1-yl)methanone[3-6]: ^{1}H NMR (501 MHz, $CDCl_3$) δ 7.52 (s, 2H), 7.46 (d, *J* = 7.2 Hz, 3H), 3.83 (dd, *J* = 13.3, 6.6 Hz, 4H), 1.86 (s, 4H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 172.52 (s), 137.42 (s), 130.88 (s), 128.90 (s), 126.67 (s), 50.42 (s), 22.47 (s).



^{1}H NMR spectra of **34** (501 MHz, $CDCl_3$)

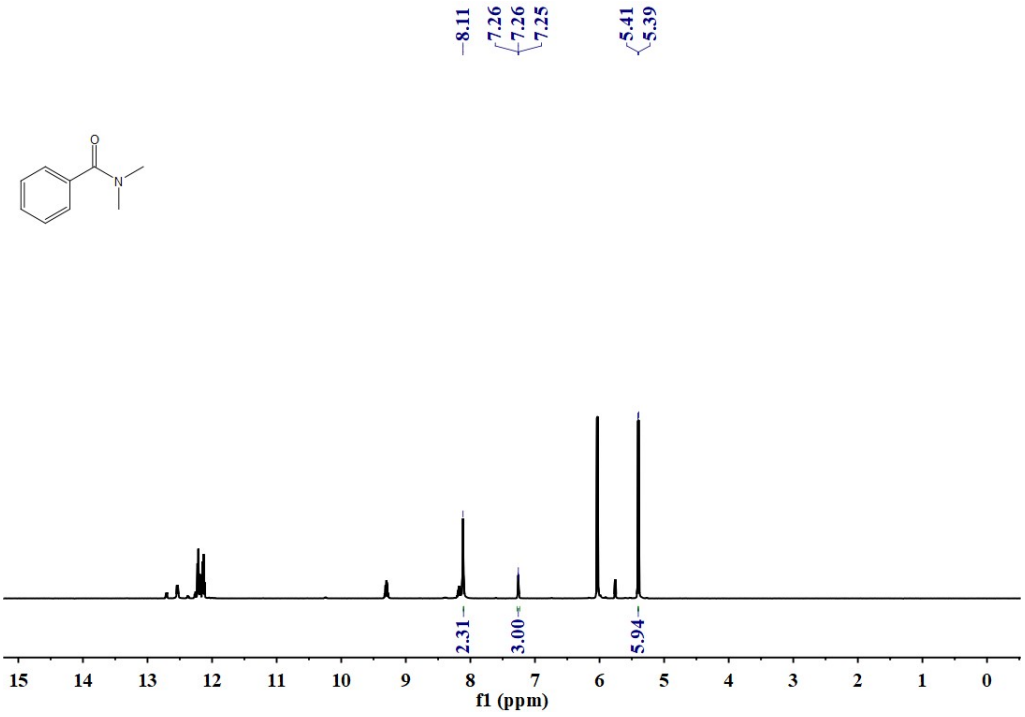


^{13}C NMR spectra of **34** (126 MHz, $CDCl_3$)

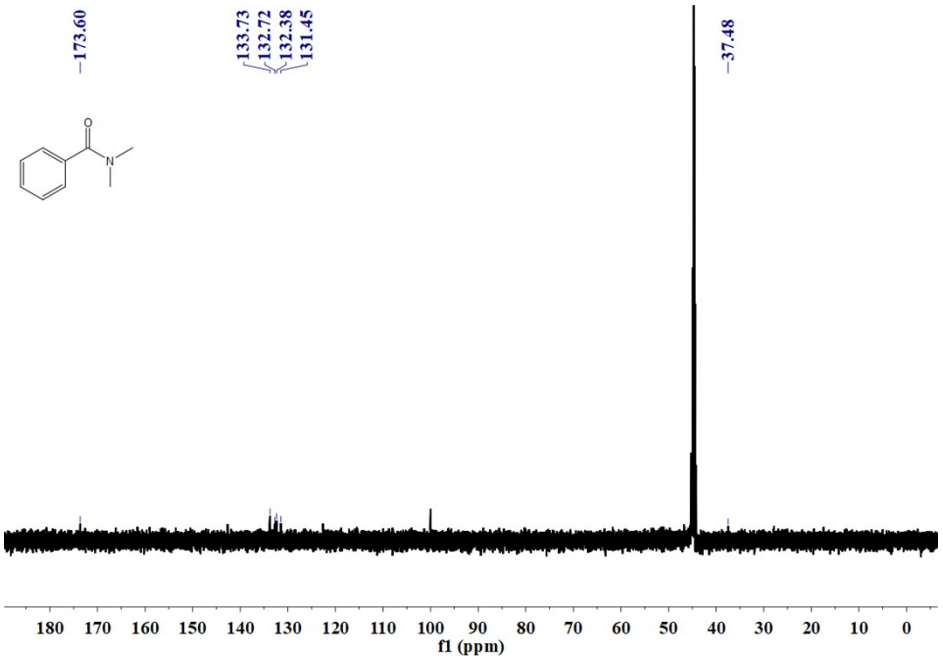


***N*, *N*-dimethylbenzamide[3-6]:** ^{1}H NMR (501 MHz, $CDCl_3$) δ 8.11 – 8.10 (m, 2H), 7.28 – 7.24 (m, 3H), 5.41 (s, 6H).

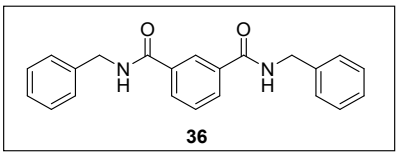
^{13}C NMR (126 MHz, $CDCl_3$) δ 173.60 (s), 133.73 (s), 132.72 (s), 132.38 (s), 131.45 (s).



^{1}H NMR spectra of **35** (501 MHz, $CDCl_3$)

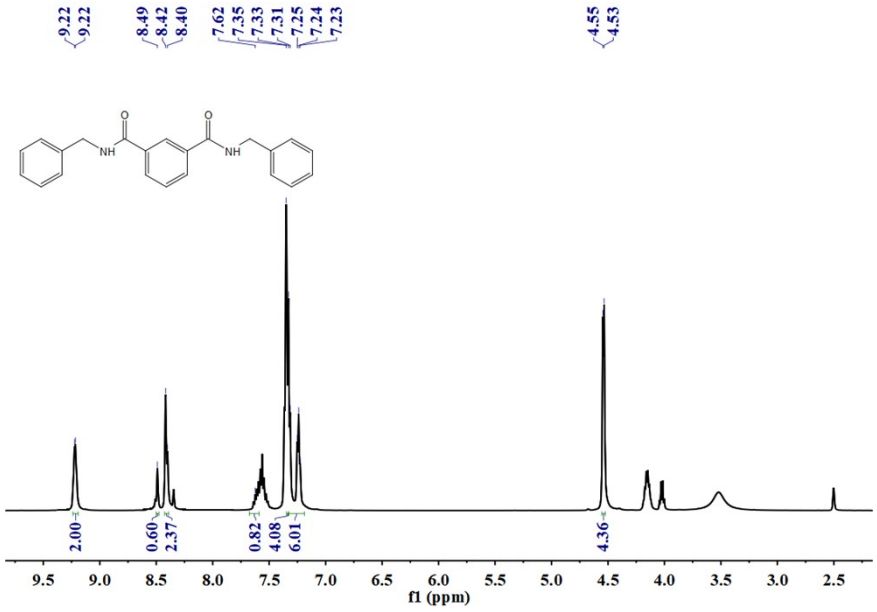


^{13}C NMR spectra of **35** (126 MHz, $CDCl_3$)

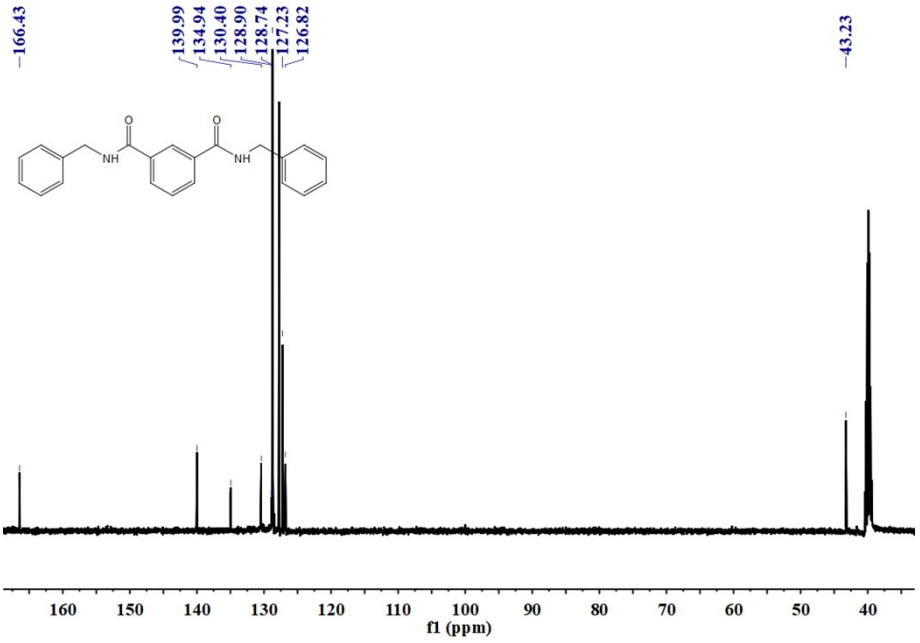


***N*^{1}, *N*^{3}-dibenzylisophthalamide:** ^{1}H NMR (501 MHz, DMSO) δ 9.22 (d, *J* = 3.5 Hz, 2H), 8.49 (s, 1H), 8.41 (d, *J* = 8.6 Hz, 2H), 7.62 (s, 1H), 7.33 (s, 4H), 7.33 – 7.19 (m, 6H), 4.54 (d, *J* = 5.6 Hz, 4H).

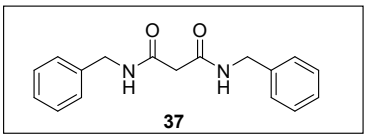
^{13}C NMR (126 MHz, DMSO) δ 166.43 (s), 139.99 (s), 134.94 (s), 130.40 (s), 128.90 (s), 128.74 (s), 127.23 (s), 126.82 (s), 43.23 (s).



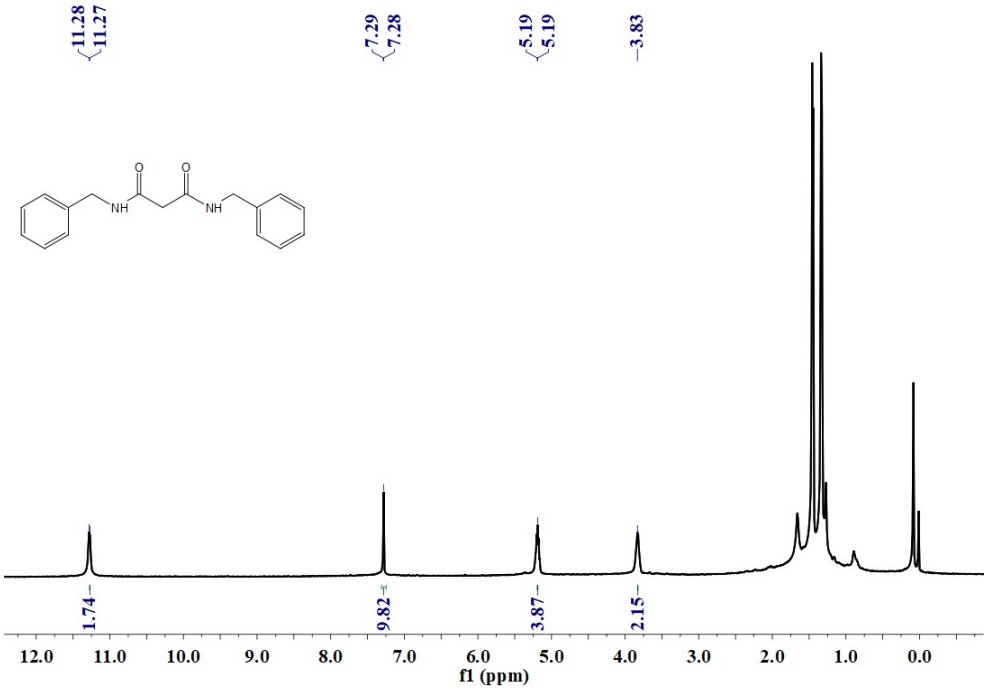
^{1}H NMR spectra of **36** (501 MHz, DMSO)



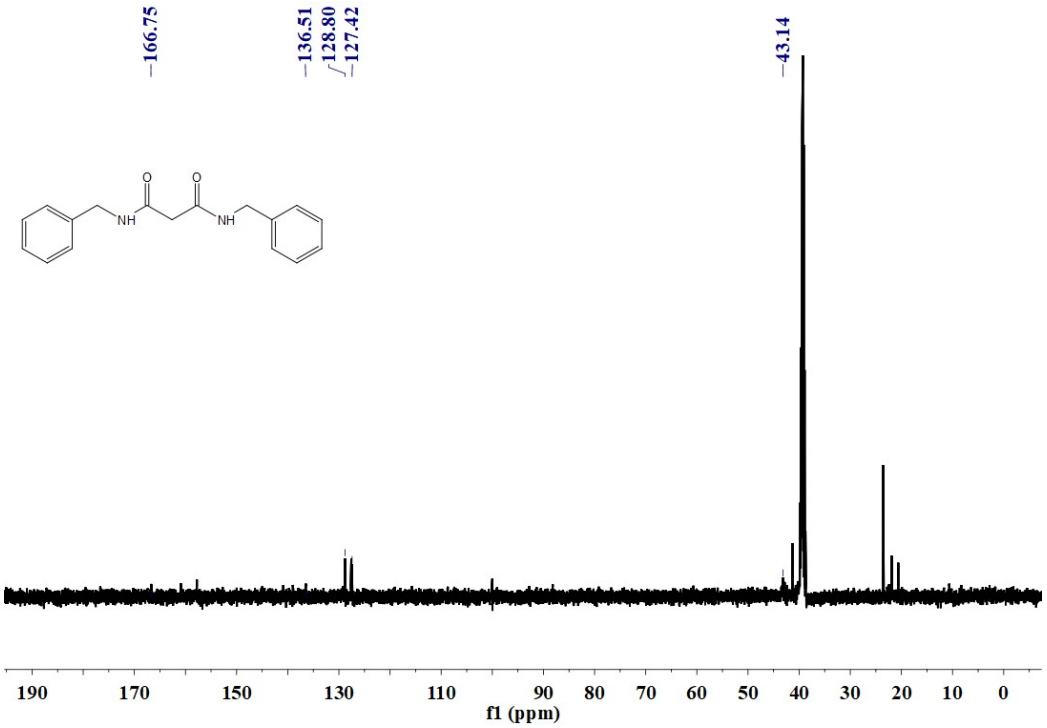
^{13}C NMR spectra of **36** (126 MHz, DMSO)



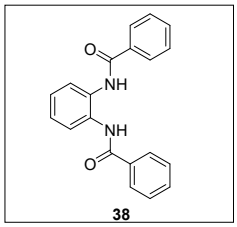
***N*^{1}, *N*^{3}-dibenzylmalonamide:** ^{1}H NMR (501 MHz, DMSO) δ 11.27 (s, 2H), 7.28 (d, *J* = 3.2 Hz, 10H), 5.19 (s, 4H), 3.83 – 3.82 (m, 2H).
^{13}C NMR (126 MHz, DMSO) δ 136.58 – 136.43 (m), 128.80 (s), 127.42 (s), 43.14 (s).



^{1}H NMR spectra of **37** (501 MHz, DMSO)

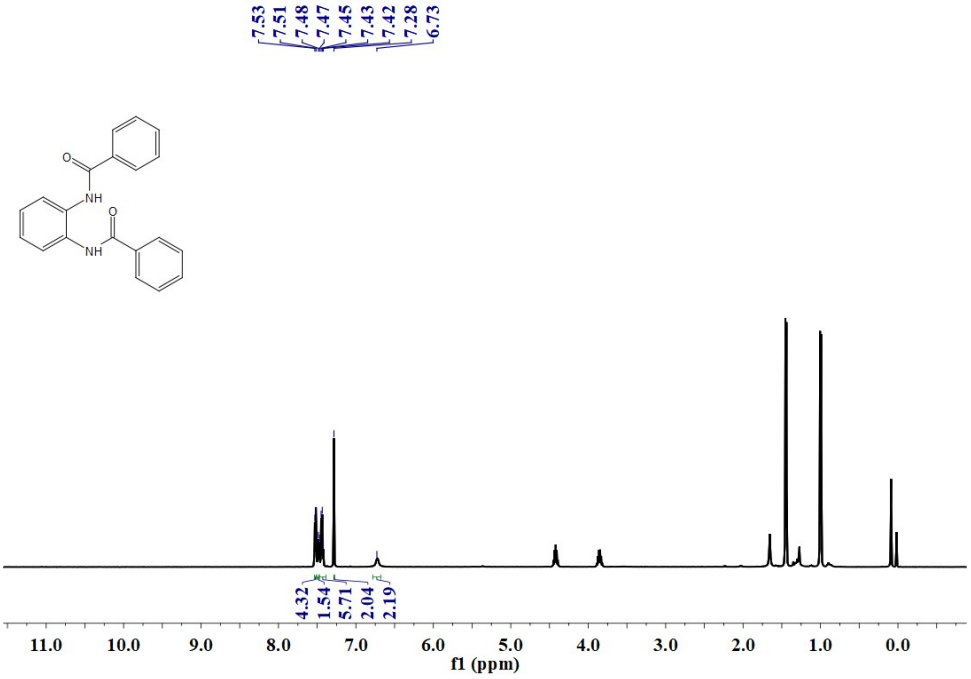


^{13}C NMR spectra of **37** (126 MHz, DMSO)

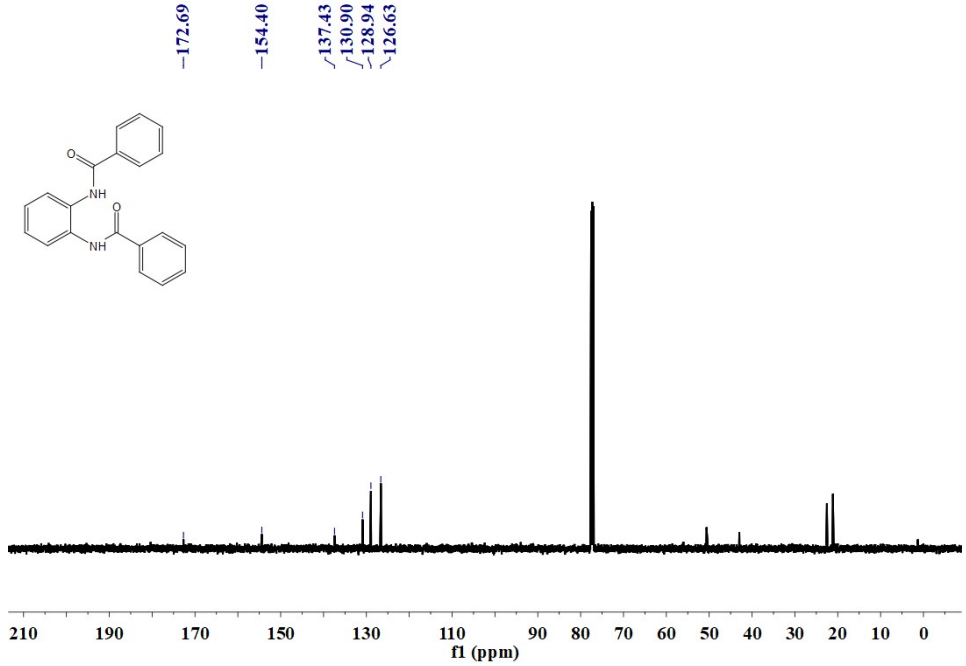


***N*, *N*'-(1,2-phenylene)dibenzamide:** ^{1}H NMR (501 MHz, $CDCl_3$) δ 7.53, 7.51, 7.48, 7.47, 7.45, 7.43, 7.42, 7.28, 6.73.

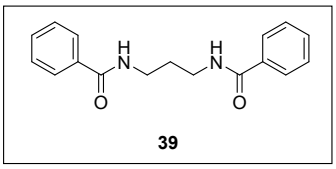
^{13}C NMR (126 MHz, $CDCl_3$) δ 172.69, 154.40, 137.43, 130.90, 128.94, 126.63.



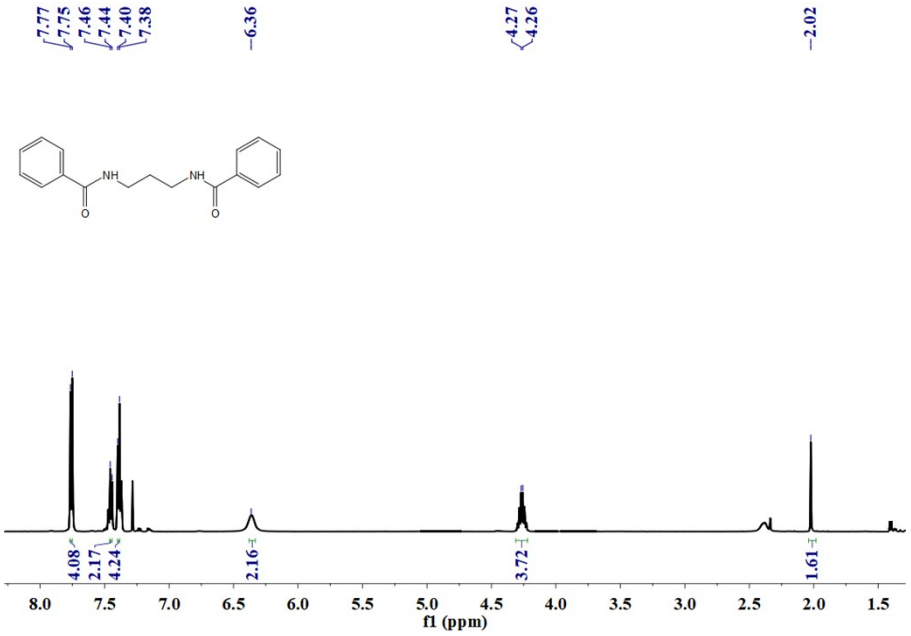
^{1}H NMR spectra of **38** ($CDCl_3$)



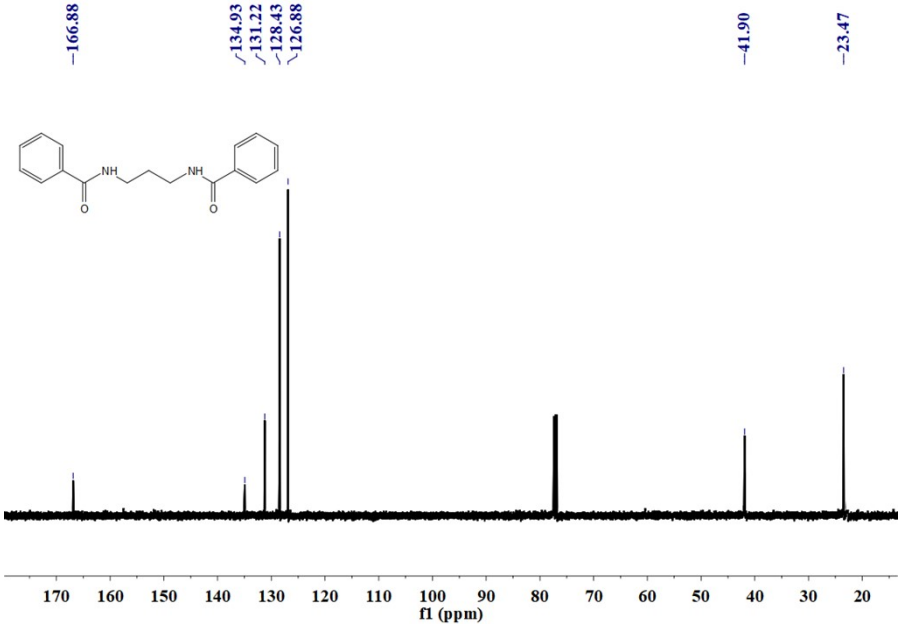
^{13}C NMR spectra of **38** ($CDCl_3$)



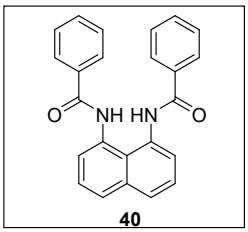
***N*, *N*'-(propane-1,3-diyl)dibenzamide:** ^{1}H NMR (501 MHz, $CDCl_3$) δ 7.77, 7.75, 7.46, 7.44, 7.40, 7.38, 6.36, 4.27, 4.26, 2.02.
^{13}C NMR (126 MHz, $CDCl_3$) δ 166.88, 134.93, 131.22, 128.43, 126.88, 41.90, 23.47.



^{1}H NMR spectra of **39** (501 MHz, $CDCl_3$)

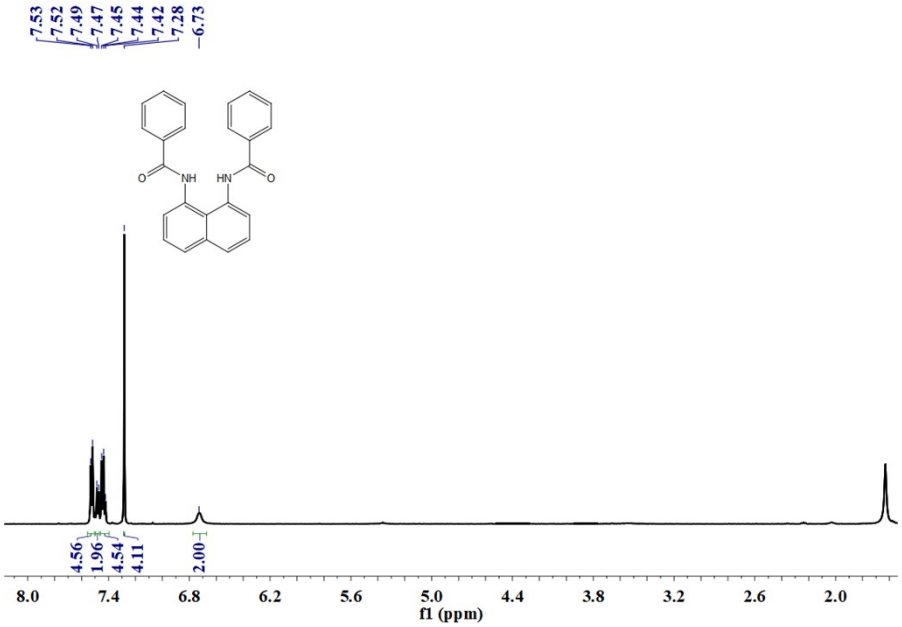


^{13}C NMR spectra of **39** (126 MHz, $CDCl_3$)

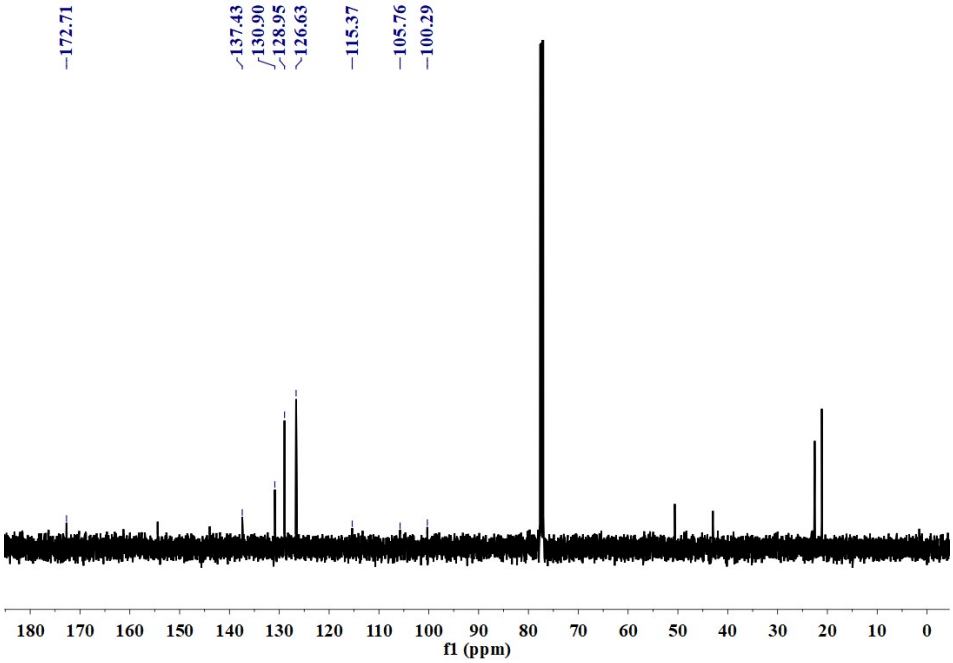


***N, N*'-(naphthalene-1,8-diyl)dibenzamide:** ^{1}H NMR (501 MHz, $CDCl_3$) δ 7.53, 7.52, 7.49, 7.47, 7.45, 7.44, 7.42, 7.28, 6.73.

^{13}C NMR (126 MHz, $CDCl_3$) δ 172.71, 137.43, 130.90, 128.95, 126.63, 115.37, 105.76, 100.29.



^{1}H NMR spectra of **40** (501 MHz, $CDCl_3$)



^{13}C NMR spectra of **40** (126 MHz, $CDCl_3$)