SUPPORTING INFORMATION

for

Scalable Mechanochemical Synthesis of Amides Using Bead Milling Technology

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1. General Information

All the solvents and chemicals were obtained from commercial vendors (Sigma-Aldrich, Fisher Scientific, VWR or BLDpharm) and were used as received, without any further purification.

^{1}H- and ^{13}C-NMR spectra were recorded on a Bruker Avance III 300 MHz instrument at room temperature (RT), in acetone-d_6 as solvent, at 300 MHz and 75 MHz, respectively. Chemical shifts (δ) are reported in ppm using TMS as internal standard. Coupling constants are given in Hz units. The residual solvent peak of acetone and the water signal appear in the ^{1}H NMR spectra at approximately 2.0 ppm and 2.8–3.3 ppm, respectively.

Analytical HPLC measurements were carried out on a C18 reversed-phase column (150 × 4.6 mm, particle size 5 µm) at 37 °C using mobile phases A [H_2O/MeCN 90:10 (v/v) + 0.1% TFA] and B (MeCN + 0.1% TFA) at a flow rate of 1.5 mL min^{-1}. The gradient applied was as follows: linear increase from 30% solution B to 100% B in 5 min, hold at 100% solution B for 5 min. All samples were prepared in HPLC-grade acetonitrile and analyzed via UV-Vis.

High resolution mass spectra (HRMS) were recorded on an Agilent 6230 TOF LC/MS (G6230B) by flow injections on an Agilent 1260 Infinity Series HPLC (HiP Degasser G4225A, Binary Pump G1312B, ALS Autosampler G1329B, TCC Column thermostat G1316A, DAD Detector G4212B).

2. Experimental Descriptions and Protocols

2.1. Description of the Mechanochemical Reactor Setup

A commercially available agitator bead mill, the Dyno®-Mill Research Lab (WAB-GROUP®, Switzerland), was employed (Figure S1, and Figure 1 in the manuscript).[S1] The instrument is equipped with a cylindrical silicon carbide grinding chamber (80 mL volume) and a specialized rotational grinding system known as the Dyno®-Accelerator (fabricated from hardened stainless steel). The reaction mixture is continuously fed into the chamber via a feed screw, where it is mixed with a large number of miniature grinding beads (ø 0.05–1.0 mm). The accelerator is mounted on a shaft driven by a motor at 1500–6000 rpm, adjustable via an external control panel. Its intensive circulatory motion accelerates the grinding beads, delivering high energy input and propelling the reaction mixture through the outlet, while the beads are retained in the chamber by a sieve plate. The grinding chamber was temperature-controlled using an external thermostat (Ministat 240, Huber, Germany) with a 9:1 water/ethylene glycol mixture as the cooling medium. The system can be operated either in continuous single-pass mode or in recirculation mode, the latter achieved by directing the outlet tube back to the inlet funnel.

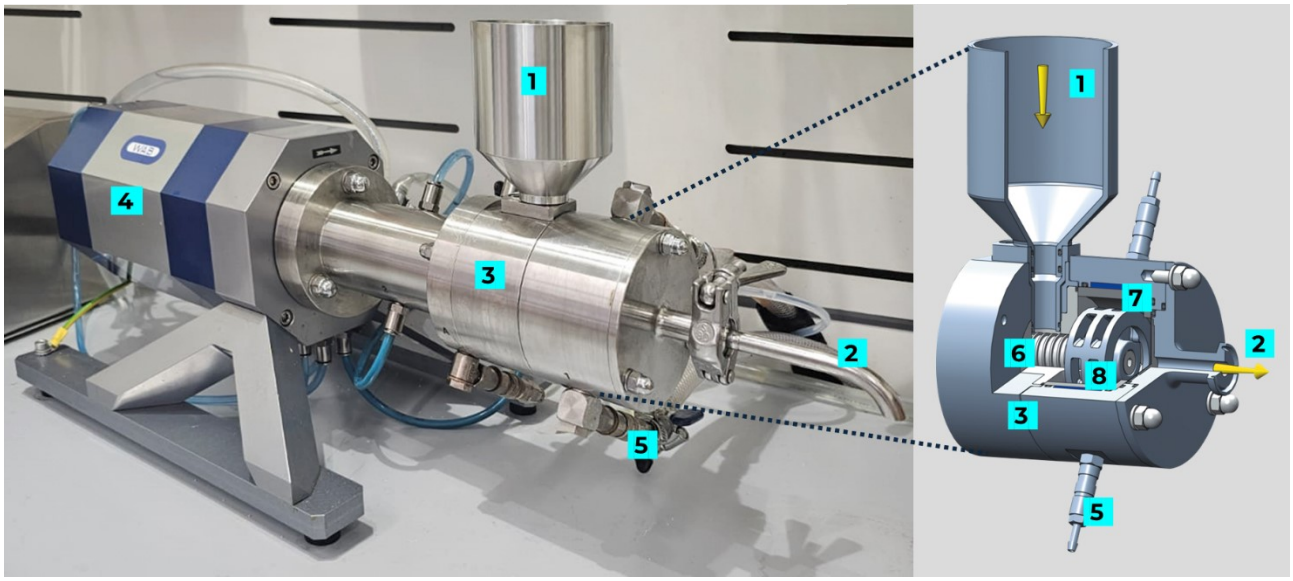


Figure S1. Photograph and detailed schematic representation of the Dyno®-Mill Research Lab reactor. (1) Inlet hopper, (2) outlet, (3) grinding chamber housing, (4) motor, (5) thermostat connections, (6) feed screw, (7) heated grinding chamber, (8) Dyno®-Accelerator.

2.2. Representative Procedures for Small Scale Amidations in the Dyno®-Mill Reactor

The grinding chamber of the Dyno®-Mill was charged with 187 g of ZrO_2/Y_2O_3 beads (ø 0.8 mm), corresponding to a filling degree of 60 v/v%. Carboxylic acid (50 mmol) and 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride (EDC·HCl; 50 mmol) were weighed in plastic cups and introduced into the inlet hopper together with EtOH, EtOAc, DMSO, or 2MeTHF (η= 0.5, 0.34, or 0.25 µL/mg) as liquid-assisted grinding (LAG) additive. In selected experiments, 1.0 equiv of pyridine, TEA, or KOH was also added as base. The reactor was then operated in continuous flow single-pass mode at rotation speeds of 1500, 3800, or 6000 rpm, allowing the reagents to enter the chamber through the feed screw. The corresponding amine derivative (50 mmol) was subsequently added via the inlet hopper. Reactions were performed at 25 °C or 50 °C. Under these conditions, the complete reaction mixture was discharged from the reactor within 5–6 min. The amide product was then precipitated by quenching the collected material with cold water, and the resulting solid was filtered under reduced pressure, washed with water, and dried under vacuum. For HPLC analysis, quenched samples were diluted with 1 mL of MeCN prior to injection. The isolated compound was characterized by ^{1}H and ^{13}C NMR spectroscopy as well as HRMS. The corresponding results are shown in Table 1 and Scheme 1 in the manuscript.

2.3. Representative Procedures for Control Experiments

<u>Control experiment without mixing</u>
4-Hydroxyphenylacetic acid (**2**; 1.0 mmol), 3,5-dimethylaniline (**1**; 1.0 mmol), and EDC·HCl (1.0 mmol) were combined in a flask with EtOAc (η= 0.34 µL/mg) and left standing for 5 min at 25 °C without mixing or agitation. The reaction was then quenched with cold water and analyzed by HPLC.

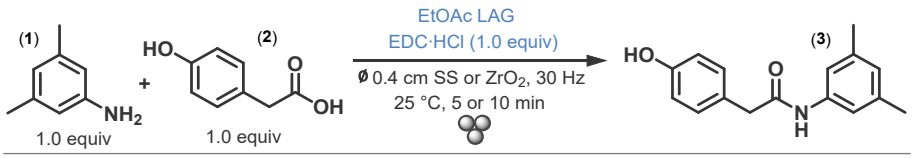
<u>Mixing with spatula in a beaker</u>
4-Hydroxyphenylacetic acid (**2**; 1.0 mmol), 3,5-dimethylaniline (**1**; 1.0 mmol), and EDC·HCl (1.0 mmol) were combined in a flask with EtOAc (η= 0.34 µL/mg) and stirred manually with a spatula for 5 min at 25 °C. The reaction was then quenched with cold water and analyzed by HPLC.

<u>Agitated using a horizontal shaker</u>
4-Hydroxyphenylacetic acid (**2**; 1.0 mmol), 3,5-dimethylaniline (**1**; 1.0 mmol), and EDC·HCl (1.0 mmol) were combined in a flask with EtOAc (η= 0.34 µL/mg) and agitated using a horizontal shaker (100 rpm) for 5 min at 25 °C. The reaction was then quenched with cold water and analyzed by HPLC.

2.4. Representative Procedures for Ball Milling Experiments

Ball-milling experiments were carried out using a Retsch MM400 mixer mill equipped with two milling stations operating at frequencies of up to 30 Hz. Reactions were performed in 10 mL stainless steel (SS) jars, each loaded with twenty ø 0.4 cm balls (SS or ZrO_2). A mixture of 4-hydroxyphenylacetic acid (**2**, 1.0 mmol), 3,5-dimethylaniline (**1**, 1.0 mmol), EDC·HCl (1.0 mmol), and the selected LAG additive was placed in a 10 mL jar together with the milling media. The reaction mixture was ground at 30 Hz for 5 min or 10 min at 25 °C. Upon completion, the reaction was quenched with cold water and samples of the crude product was analyzed by HPLC after diluting with 1 mL of MeCN prior to injection.

Table S1 Results of ball milling control experiments.



#	η [µL/mg]	Ball material	Time [min]	Yield[a] [%]
1	0.5	SS	5	55
2	0.34	SS	5	75

3	0.34	SS	10	78
4	0.34	ZrO_2	5	65
5	0.25	SS	5	71

[a] Determined by HPLC-UV at 215 nm.

2.5 Representative Procedure for Large Scale Amidations in the Dyno®-Mill Reactor

The grinding chamber of the Dyno®-Mill was charged with 187 g of ZrO_2/Y_2O_3 beads (Ø 0.8 mm), corresponding to a filling degree of 60 v/v%. 4-Hydroxyphenylacetic acid (**2**; 1.0 mol) and EDC·HCl (1.0 mol) were weighed into plastic cups and transferred to a beaker together with EtOAc (η = 0.34 µL/mg) as LAG additive. The components were mixed until a paste was formed, then fed into the inlet hopper of the Dyno®-Mill. Owing to the large reagent quantities, the mill was operated in recirculation mode at a rotation speed of 3800 rpm. Once the reagents had entered the grinding chamber through the feed screw and recirculation was established, the corresponding aniline derivative (1.0 mol) was added via the inlet hopper. Recirculation was continued for 5 min while maintaining the reactor temperature at 25 °C. The amide product was then precipitated by quenching the collected material with cold water, and the resulting solid was filtered under reduced pressure, washed with water, and dried under vacuum. For HPLC analysis, quenched samples were diluted with 1 mL of MeCN prior to injection. The isolated compound was characterized by ^{1}H and ^{13}C NMR spectroscopy as well as HRMS. The corresponding results are shown in Scheme 2 in the manuscript. See also Figures S2 and S3 for photographs taken during the scale-up experiments.

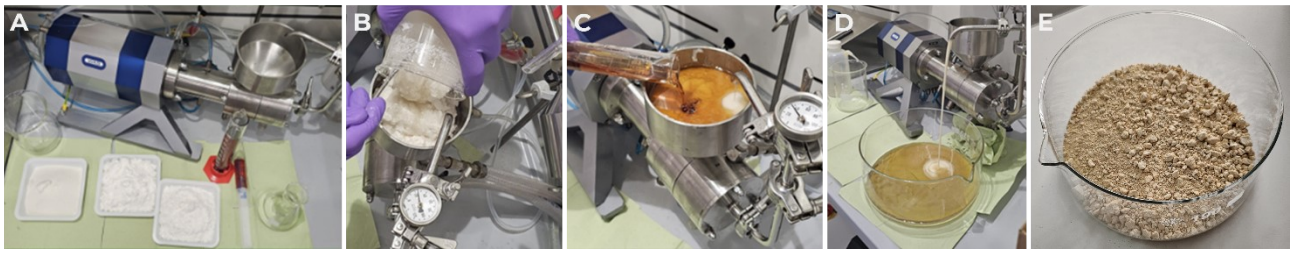


Figure S2. Photographs taken during 1.0 mol scale amidation of 4-hydroxyphenylacetic acid with 3,5-dimethylaniline. (A) Reagents weighed and prepared. (B) Feeding the carboxylic acid/EDC·HCl/EtOAc mixture. (C) Adding the aniline component. (D) Collection of crude material. (E) Isolated amide product **3**.

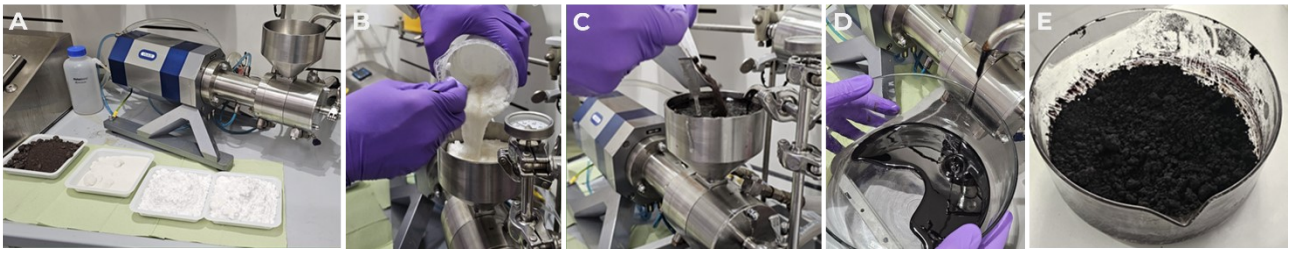


Figure S3. Photographs taken during 1.0 mol scale amidation of 4-hydroxyphenylacetic acid with 3,4-dimethoxyaniline. (A) Reagents weighed and prepared. (B) Feeding the carboxylic acid/EDC·HCl/EtOAc mixture. (C) Adding the aniline component. (D) Collection of crude material. (E) Isolated amide product **6**.

3. Calculation of Green Chemistry Metrics

E-factor, process mass intensity (PMI), reaction mass efficiency (RME), atom economy (AE) and optimum efficiency (OE) were calculated using the following equations.[S2] For the calculation of the E factor and PMI, the mass of washing H_2O was excluded. The corresponding data are shown in Table 3 in the manuscript.

$$E-factor = \frac{total\ mass\ of\ waste}{mass\ of\ product}$$

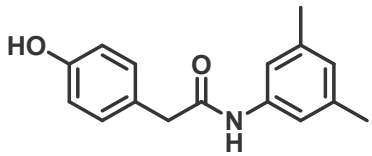
$$PMI = \frac{total\ mass\ of\ raw\ materials\ used}{mass\ of\ product}$$

$$RME = \frac{mass\ of\ isolated\ product}{total\ mass\ of\ reactants} \times 100$$

$$AE = \frac{molecular\ weight\ of\ product}{total\ molecular\ weight\ of\ reactants} \times 100$$

$$OE = \frac{RME}{AE} \times 100$$

4. Characterization of Prepared Compounds



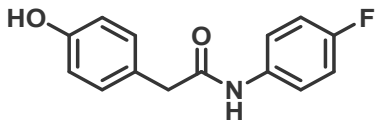
N-(3,5-Dimethylphenyl)-2-(4-hydroxyphenyl)acetamide (**3**)

^{1}H NMR (300 MHz, acetone-d_6) δ 9.07 (s, 1H), 8.31 (s, 1H), 7.27 (s, 2H), 7.23 – 7.16 (m, 2H), 6.81 – 6.74 (m, 2H), 6.67 (m, 1H), 3.55 (s, 2H), 2.21 (s, 6H).

^{13}C NMR (75 MHz, acetone-d_6) δ 170.2, 157.2, 140.3, 138.9, 131.0, 127.6, 125.6, 117.8, 116.0, 44.0, 21.4.

HRMS: calculated for $C_{16}H_{18}NO_2^+$: 256.1332 [M+H$^+$]; found: 256.1333

NMR data of the compound matches the reported literature.[S3]



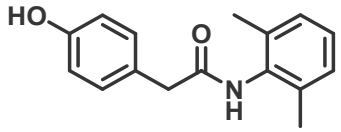
N-(4-Fluorophenyl)-2-(4-hydroxyphenyl)-acetamide (**4**)

^{1}H NMR (300 MHz, acetone-d_6) δ 9.37 (s, 1H), 8.33 (s, 1H), 7.75 – 7.61 (m, 2H), 7.19 (d, *J* = 8.6 Hz, 2H), 7.03 (m, 2H), 6.78 (d, *J* = 8.6 Hz, 2H), 3.57 (s, 2H).

^{13}C NMR (75 MHz, acetone-d_6) δ 170.4, 161.1, 157.9, 157.2, 136.7, 131.0, 127.4, 121.9, 121.8, 116.0, 115.9, 115.7, 43.8.

HRMS: calculated for $C_{14}H_{13}FNO_2^+$: 246.0925 [M+H$^+$]; found: 246.0934

NMR data of the compound matches the reported literature.[S4]

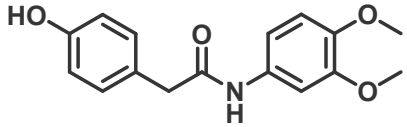


N-(2,6-Dimethylphenyl)-2-(4-hydroxyphenyl)-acetamide (**5**)

^{1}H NMR (300 MHz, acetone-d_6) δ 8.41 (s, 1H), 7.25 (d, *J* = 8.6 Hz, 2H), 7.01 (s, 3H), 6.80 (d, *J* = 8.6 Hz, 2H), 3.61 (s, 2H), 2.12 (s, 6H)

^{13}C NMR (75 MHz, acetone-d_6) δ 170.1, 157.1, 136.5, 134.7, 131.1, 128.5, 127.9, 127.3, 116.1, 43.2, 18.5.

HRMS: calculated for $C_{16}H_{18}NO_2^+$: 256.1332 [M+H$^+$]; found: 256.1343

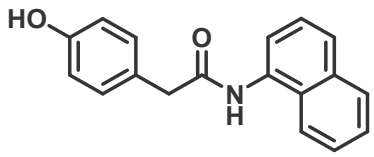


N-(3,4-Dimethoxyphenyl)-2-(4-hydroxyphenyl)-acetamide (**6**)

^{1}H NMR (300 MHz, acetone-d_6) δ 9.15 (s, 1H), 8.33 (s, 1H), 7.43 (s, 1H), 7.19 (d, *J* = 8.0 Hz, 2H), 7.10 (d, *J* = 8.0 Hz, 1H), 6.80 (m, 3H), 3.74 (s, 6H), 3.55 (s, 2H).

^{13}C NMR (75 MHz, acetone-d_6) δ 170.0, 150.2, 146.4, 134.3, 131.1, 127.6, 116.0, 113.1, 111.9, 105.5, 56.4, 55.9, 43.9.

HRMS: calculated for $C_{16}H_{18}NO_4^+$: 288.1230 [M+H$^+$]; found: 288.1246

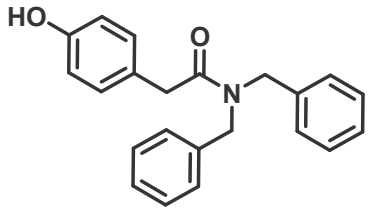


2-(4-Hydroxyphenyl)-*N*-(naphthalen-1-yl)-acetamide (**7**)

^{1}H NMR (300 MHz, acetone-d_6) δ 9.15 (s, 1H), 8.30 (s, 1H), 7.98 (m, 1H), 7.89 (m, 2H), 7.70 (s, 1H), 7.47 (m, *J* = 8.0, 3H), 7.31 (d, *J* = 8.0 Hz, 2H), 6.84 (d, *J* = 8.0 Hz, 2H), 3.78 (s, 2H).

^{13}C NMR (75 MHz, acetone-d_6) δ 170.9, 157.2, 135.1, 134.6, 131.2, 129.2, 127.7, 126.7, 126.7, 126.4, 125.9, 122.7, 121.7, 116.2, 43.7.

HRMS: calculated for $C_{18}H_{16}NO_2^+$: 278.1176 [M+H$^+$]; found: 278.1191

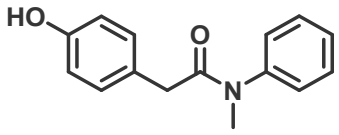


N,*N*-Dibenzyl-2-(4-hydroxyphenyl)-acetamide (**8**)

^{1}H NMR (300 MHz, acetone-d_6) δ 8.28 (s, 1H), 7.53 – 7.05 (m, 12H), 6.78 (d, *J* = 8.4 Hz, 2H), 4.55 (d, *J* = 8.4 Hz, 4H), 3.72 (s, 2H).

^{13}C NMR (75 MHz, acetone-d_6) δ 172.3, 157.0, 138.9, 138.2, 130.9, 129.6, 129.2, 128.8, 128.2, 127.9, 127.6, 116.1, 51.0, 48.6, 40.3.

HRMS: calculated for $C_{22}H_{22}NO_2^+$: 332.1645 [M+H$^+$]; found: 332.1664

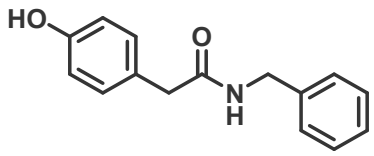


2-(4-Hydroxyphenyl)-*N*-methyl-*N*-phenylacetamide (**9**)

^{1}H NMR (300 MHz, acetone-d_6) δ 8.25 (s, 1H), 7.44 (d, *J* = 7.8 Hz, 2H), 7.38 (d, *J* = 7.2 Hz, 1H), 7.26 (d, *J* = 7.2 Hz, 2H), 6.87 (d, *J* = 7.8 Hz, 2H), 6.69 (m, 2H), 3.33 (s, 2H), 3.21 (s, 3H).

^{13}C NMR (75 MHz, acetone-d_6) δ 171.2, 156.8, 145.4, 130.9, 130.4, 128.6, 127.5, 122.1, 115.7, 40.3, 37.5.

HRMS: calculated for $C_{15}H_{16}NO_2^+$: 242.1170 [M+H$^+$]; found: 242.1188



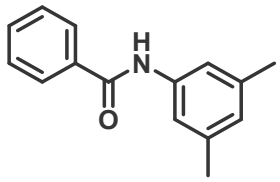
N-Benzyl-2-(4-hydroxyphenyl)-acetamide (**10**)

^{1}H NMR (300 MHz, acetone-d_6) δ 7.25 (s, 5H), 7.14 (d, *J* = 8.0 Hz, 2H), 6.79 (d, *J* = 8.0 Hz, 2H), 4.36 (s, 2H), 3.47 (s, 1H).

^{13}C NMR (75 MHz, acetone-d_6) δ 171.5, 156.4, 139.5, 130.2, 128.3, 127.4, 126.8, 126.6, 115.2, 42.7, 42.1.

HRMS: calculated for $C_{15}H_{16}NO_2^+$: 242.1176 [M+H$^+$]; found: 242.1185

NMR data of the compound matches the reported literature.[S5]



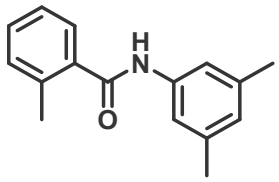
N-(3,5-Dimethylphenyl)-benzamide (**11**)

^{1}H NMR (300 MHz, acetone-d_6) δ 9.39 (s, 1H), 8.04 – 7.96 (m, 2H), 7.60 – 7.45 (m, 5H), 6.76 (s, 1H), 2.28 (s, 6H).

^{13}C NMR (75 MHz, acetone-d_6) δ 166.1, 140.2, 138.8, 136.5, 132.2, 129.2, 128.3, 126.1, 118.7, 21.5.

HRMS: calculated for $C_{15}H_{16}NO_2^+$: 226.1226 [M+H$^+$]; found: 226.1238

NMR data of the compound matches the reported literature.[S6]



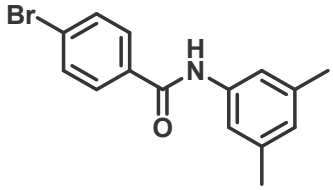
N-(3,5-Dimethylphenyl)-2-methylbenzamide (**12**)

^{1}H NMR (300 MHz, acetone-d_6) δ 9.20 (s, 1H), 7.46 (m, 3H), 7.39 – 7.18 (m, 3H), 6.76 (s, 1H), 2.87 (s, 3H), 2.28 (s, 6H).

^{13}C NMR (75 MHz, acetone-d_6) δ 140.4, 138.9, 138.4, 136.7, 131.5, 130.4, 127.9, 126.4, 126.0, 118.2, 21.5, 19.8.

HRMS: calculated for $C_{16}H_{18}NO^+$: 240.1383 [M+H$^+$]; found: 240.1395

NMR data of the compound matches the reported literature.[S7]



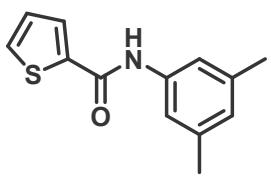
4-Bromo-*N*-(3,5-dimethylphenyl)-benzamide (**13**)

^{1}H NMR (300 MHz, acetone-d_6) δ 9.44 (s, 1H), 7.93 (d, *J* = 8.7 Hz, 2H), 7.69 (d, *J* = 8.6 Hz, 2H), 7.46 (s, 2H), 6.77 (s, 1H), 2.27 (s, 6H).

^{13}C NMR (75 MHz, acetone-d_6) δ 164.3, 139.1, 138.0, 134.6, 131.5, 129.4, 125.4, 117.9, 117.9, 20.6.

HRMS: calculated for $C_{15}H_{15}BrNO^+$: 304.0332 [M+H$^+$]; found: 304.0346

NMR data of the compound matches the reported literature.[S7]



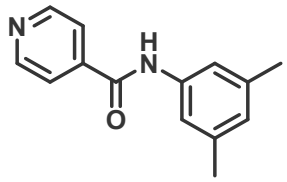
N-(3,5-Dimethylphenyl)thiophene-2-carboxamide (**14**)

^{1}H NMR (300 MHz, acetone-d_6) δ 9.38 (s, 1H), 7.92 (d, *J* = 5.0 Hz, 1H), 7.74 (d, *J* = 5.0 Hz, 1H), 7.42 (s, 2H), 7.17 (m, 1H), 6.75 (s, 1H), 2.27 (s, 6H).

^{13}C NMR (75 MHz, acetone-d_6) δ 160.7, 141.7, 139.8, 138.9, 131.9, 128.9, 128.6, 126.2, 118.7, 21.5.

HRMS: calculated for $C_{13}H_{13}NOS$: 232.0731 [M+H$^+$]; found: 232.0803

NMR data of the compound matches the reported literature.[S8]



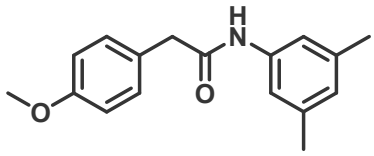
N-(3,5-Dimethylphenyl)isonicotinamide (**15**)

^{1}H NMR (300 MHz, acetone-d_6) δ 9.60 (s, 1H), 8.75 (d, *J* = 6.1 Hz, 2H), 7.86 (d, *J* = 6.1 Hz, 2H), 7.47 (s, 2H), 6.80 (s, 1H), 2.29 (s, 6H).

^{13}C NMR (75 MHz, acetone-d_6) δ 164.56, 151.29, 143.25, 139.61, 139.02, 126.65, 122.12, 118.90, 21.46.

HRMS: calculated for $C_{14}H_{15}N_2O^+$: 227.1179 [M+H$^+$]; found: 227.1191

NMR data of the compound matches the reported literature.[S7]

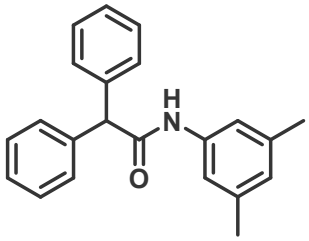


N-(3,5-Dimethylphenyl)-2-(4-methoxyphenyl)acetamide (**16**)

^{1}H NMR (300 MHz, acetone-d_6) δ 9.11 (s, 1H), 7.28 (d, *J* = 8.6 Hz, 4H), 6.87 (d, *J* = 8.6 Hz, 2H), 6.68 (s, 1H), 3.76 (s, 3H), 3.58 (s, 2H), 2.22 (s, 6H).

^{13}C NMR (75 MHz, acetone-d_6) δ 170.1, 159.5, 140.3, 138.8, 130.9, 128.9, 125.7, 117.8, 114.6, 55.4, 43.9, 21.4.

HRMS: calculated for $C_{17}H_{20}NO_2^+$: 270.1489 [M+H$^+$]; found: 270.1501



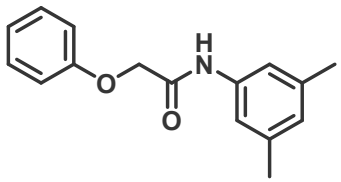
N-(3,5-dimethylphenyl)-2,2-diphenylacetamide (**17**)

^{1}H NMR (300 MHz, acetone-d_6) δ 9.71 (s, 1H), 7.43 (m, 4H), 7.39 – 7.18 (m, 8H), 6.68 (s, 1H), 5.20 (s, 1H), 2.21 (s, 6H).

^{13}C NMR (75 MHz, acetone-d_6) δ 170.8, 141.2, 140.1, 138.9, 129.6, 129.1, 127.6, 125.9, 117.9, 59.2, 21.4.

HRMS: calculated for $C_{22}H_{22}NO^+$: 316.1696 [M+H$^+$]; found: 316.1715

NMR data of the compound matches the reported literature.[S9]



N-(3,5-Dimethylphenyl)-2-phenoxyacetamide (**18**)

^{1}H NMR (300 MHz, acetone-d_6) δ 9.11 (s, 1H), 7.50 – 7.19 (m, 4H), 7.20 – 6.90 (m, 3H), 6.75 (s, 1H), 4.62 (s, 2H), 2.25 (s, 6H).

^{13}C NMR (75 MHz, acetone-d_6) δ 167.0, 158.7, 139.1, 138.9, 130.4, 126.4, 122.4, 118.5, 115.7, 68.3, 21.4.

HRMS: calculated for $C_{16}H_{18}NO_2^+$: 256.1332 [M+H$^+$]; found: 256.1342.

NMR data of the compound matches the reported literature.[S10]

5. Collection of NMR Spectra

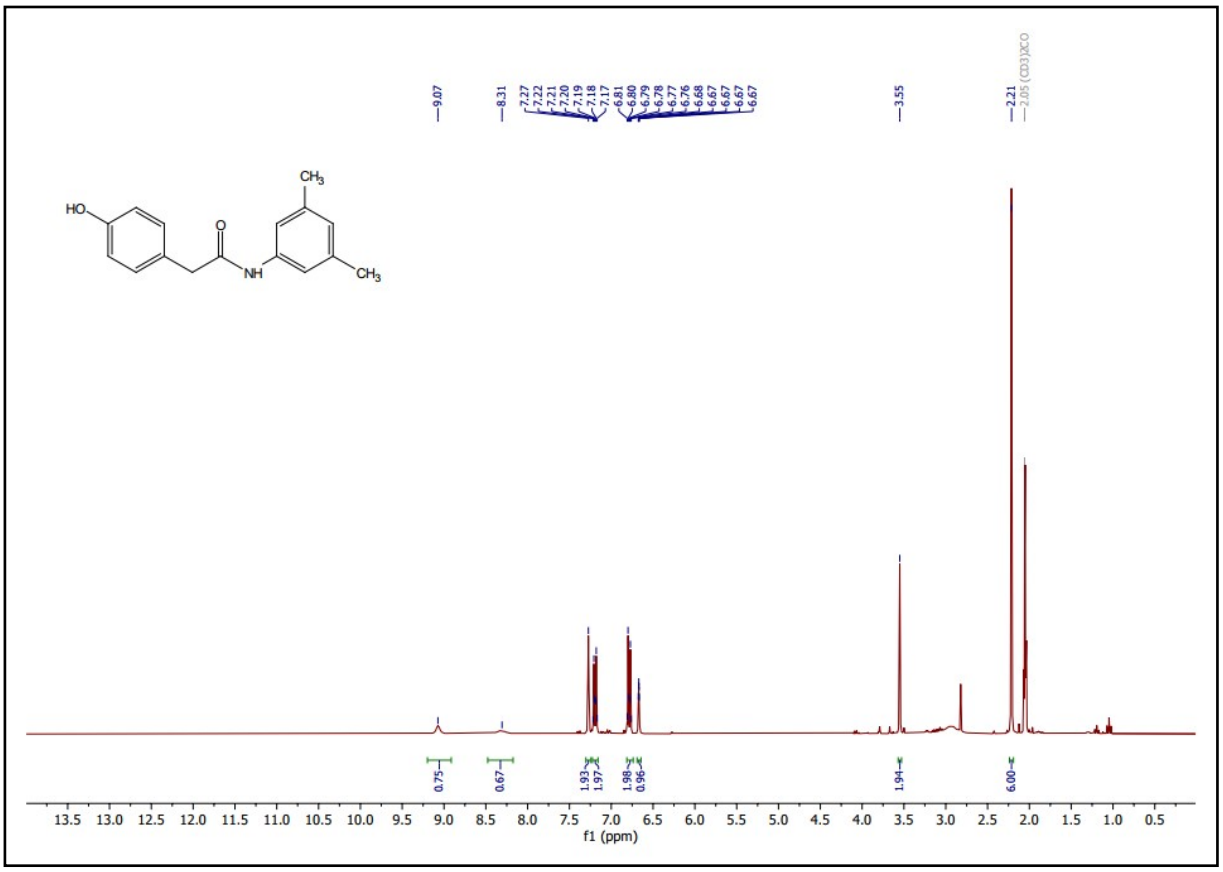


Figure S4. ^{1}H NMR spectrum of *N*-(3,5-dimethylphenyl)-2-(4-hydroxyphenyl)acetamide (**3**) in acetone-d_6 as solvent.

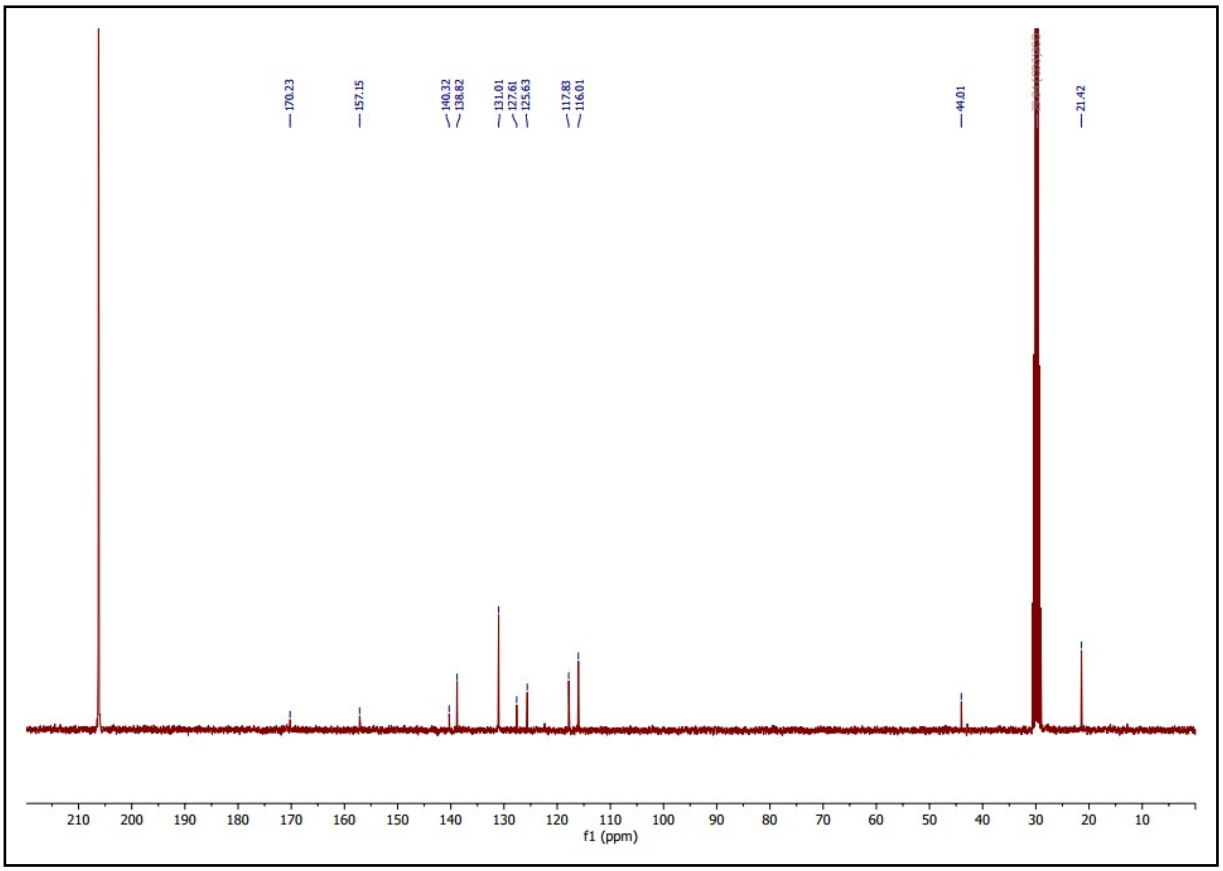


Figure S5. ^{13}C NMR spectrum of *N*-(3,5-dimethylphenyl)-2-(4-hydroxyphenyl)acetamide (**3**) in acetone-d_6 as solvent.

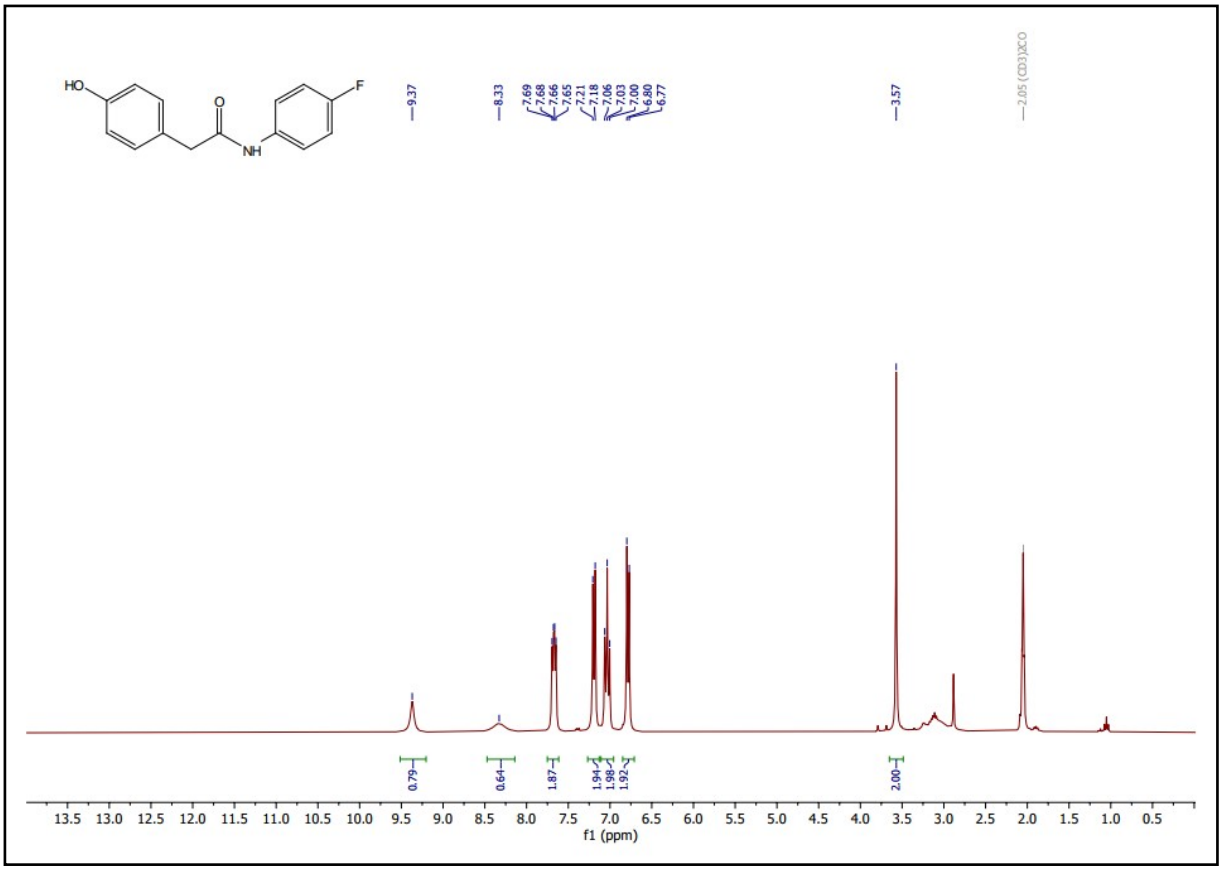


Figure S6. ^{1}H NMR spectrum of *N*-(4-fluorophenyl)-2-(4-hydroxyphenyl)-acetamide (**4**) in acetone-d_6 as solvent.

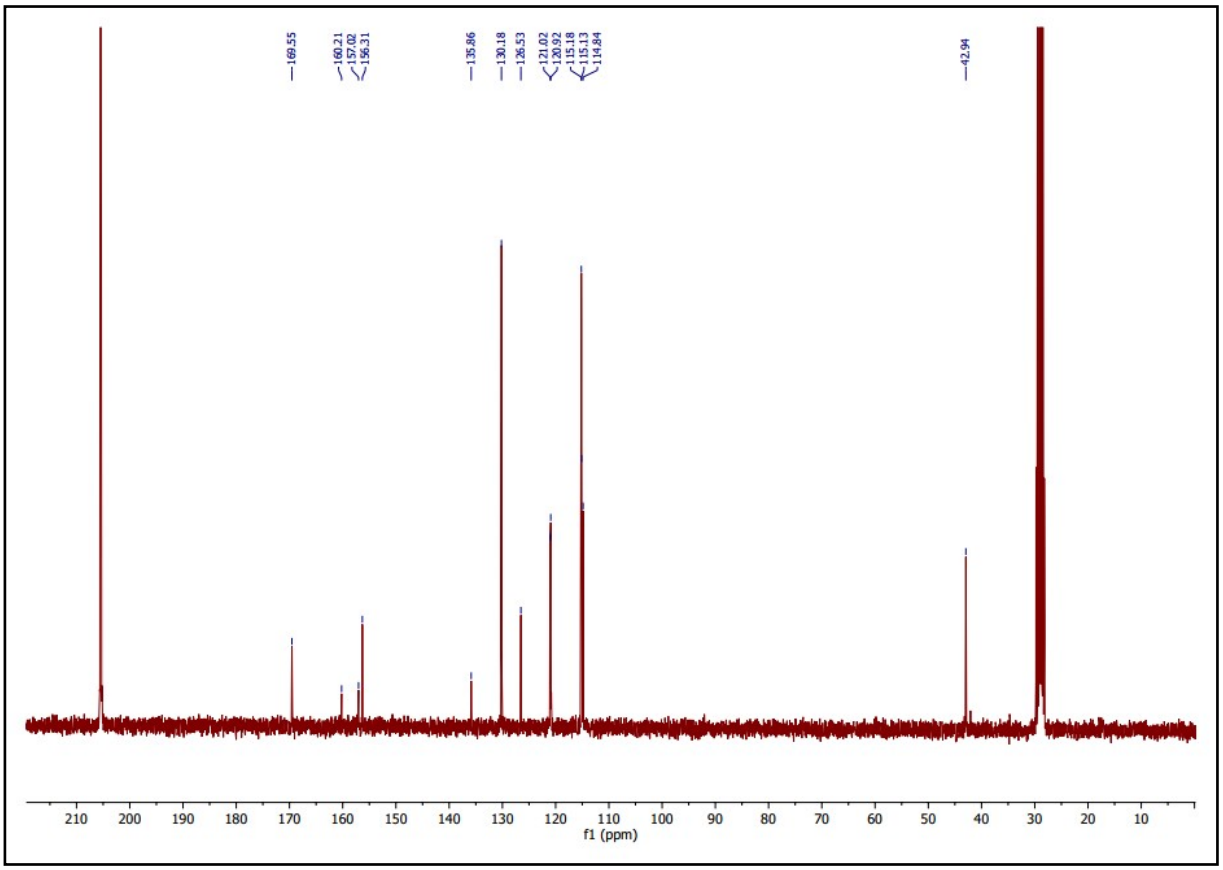


Figure S7. ^{13}C NMR spectrum of *N*-(4-fluorophenyl)-2-(4-hydroxyphenyl)-acetamide (**4**) in acetone-d_6 as solvent.

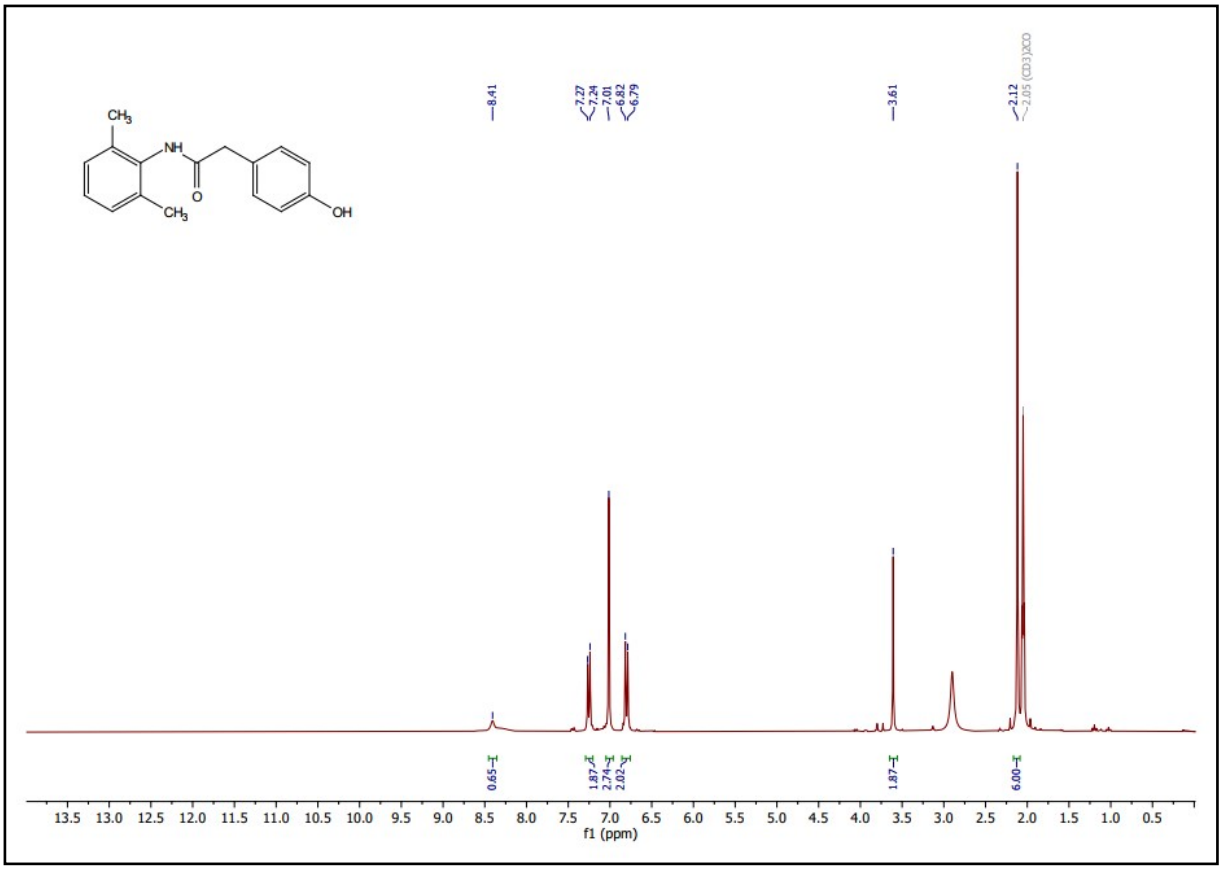


Figure S8. ^{1}H NMR spectrum of *N*-(2,6-dimethylphenyl)-2-(4-hydroxyphenyl)-acetamide (**5**) in acetone-d_6 as solvent.

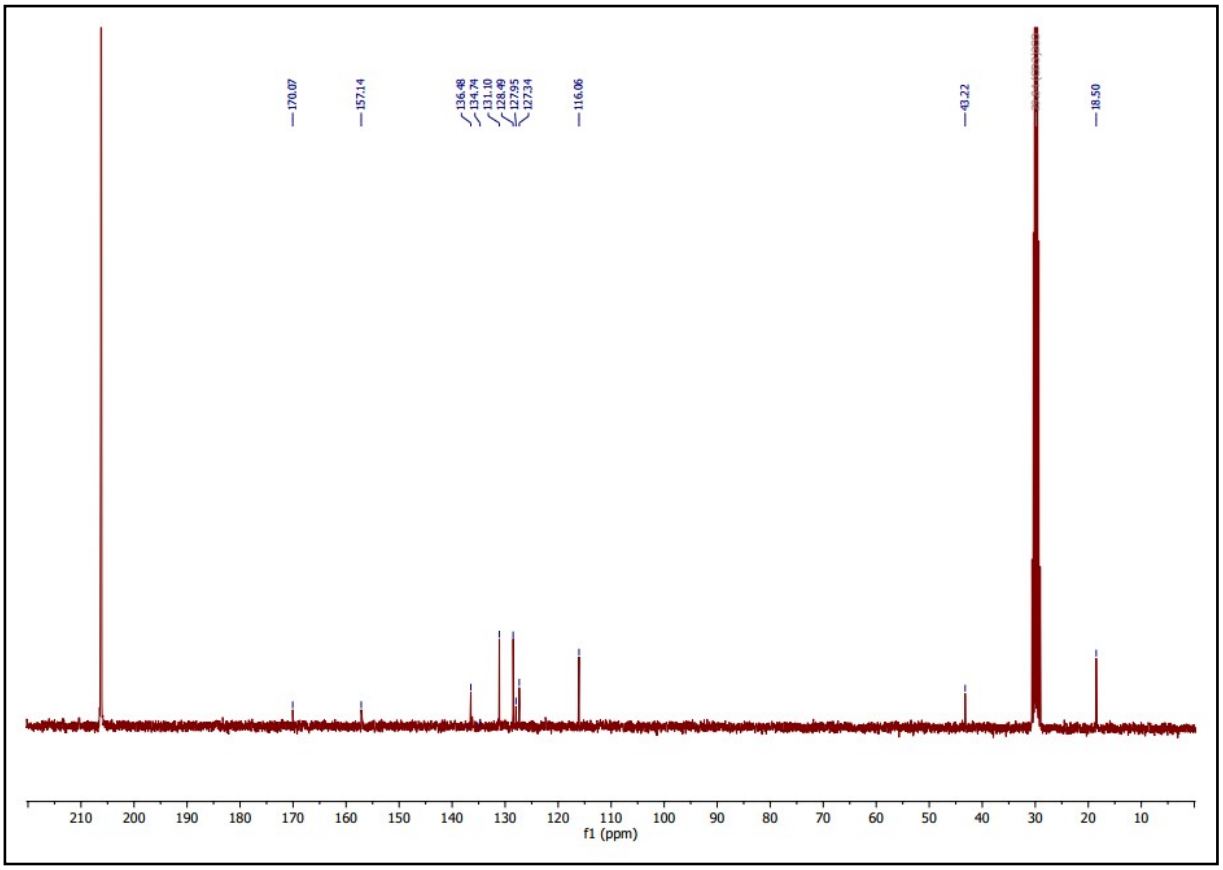


Figure S9. ^{13}C NMR spectrum of *N*-(2,6-dimethylphenyl)-2-(4-hydroxyphenyl)-acetamide (**5**) in acetone-d_6 as solvent.

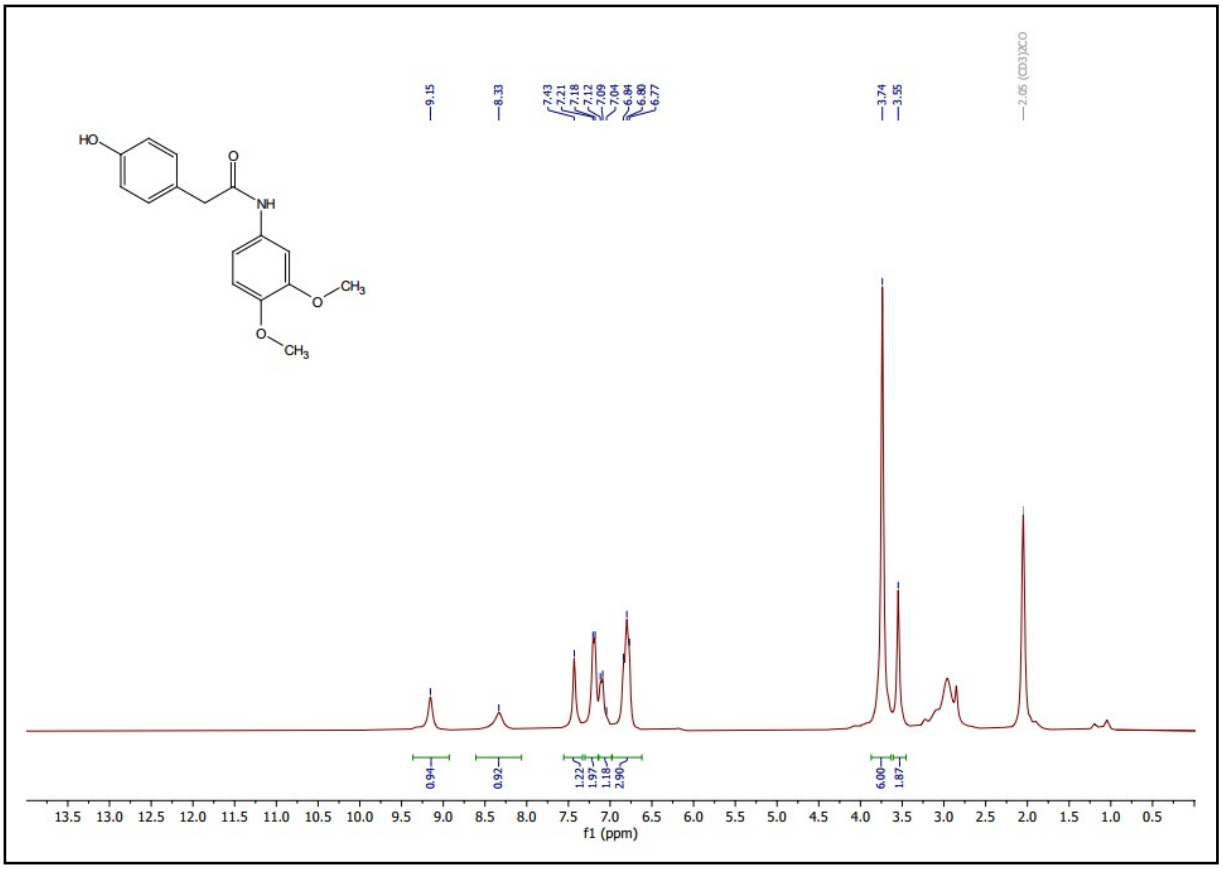


Figure S10. ^{1}H NMR spectrum of *N*-(3,4-dimethoxyphenyl)-2-(4-hydroxyphenyl)-acetamide (**6**) in acetone-d_6 as solvent.

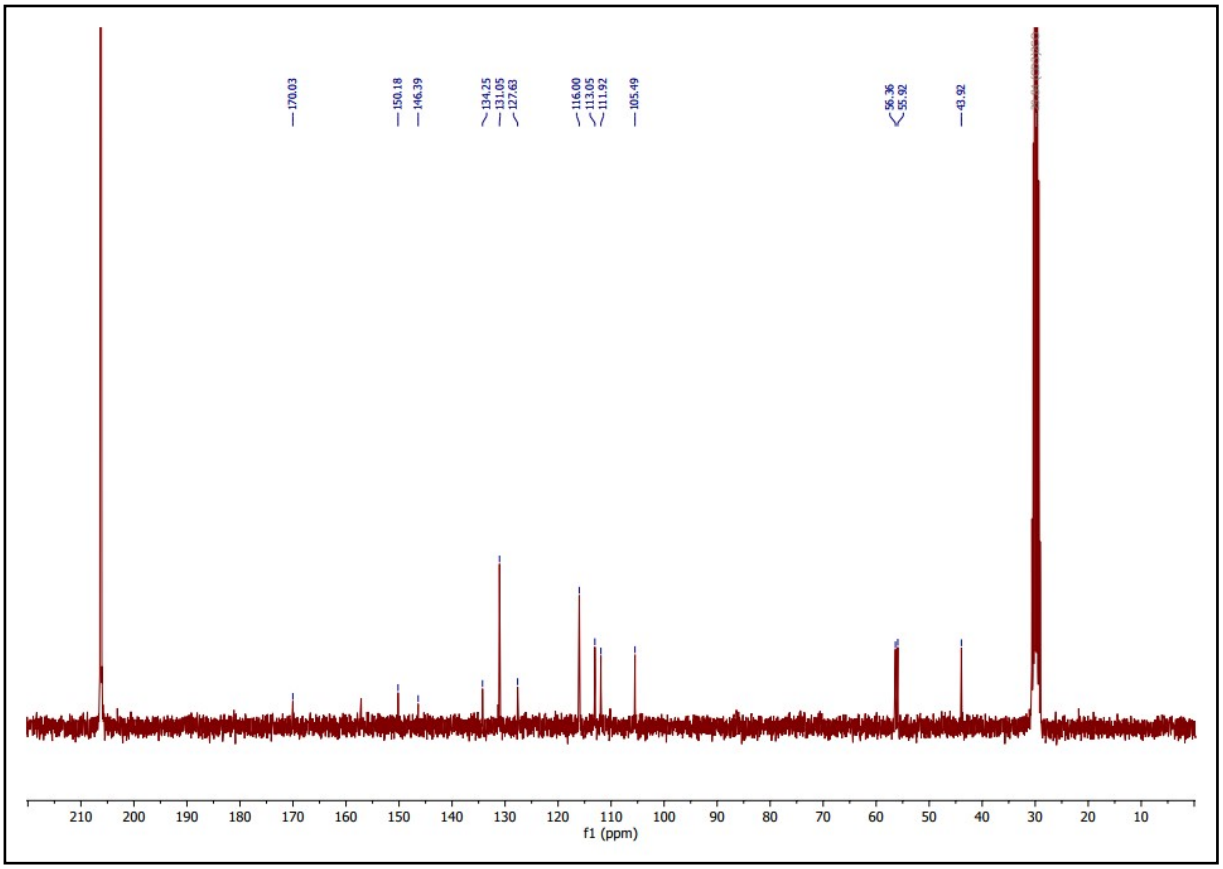


Figure S11. ^{13}C NMR spectrum of *N*-(3,4-dimethoxyphenyl)-2-(4-hydroxyphenyl)-acetamide (**6**) in acetone-d_6 as solvent.

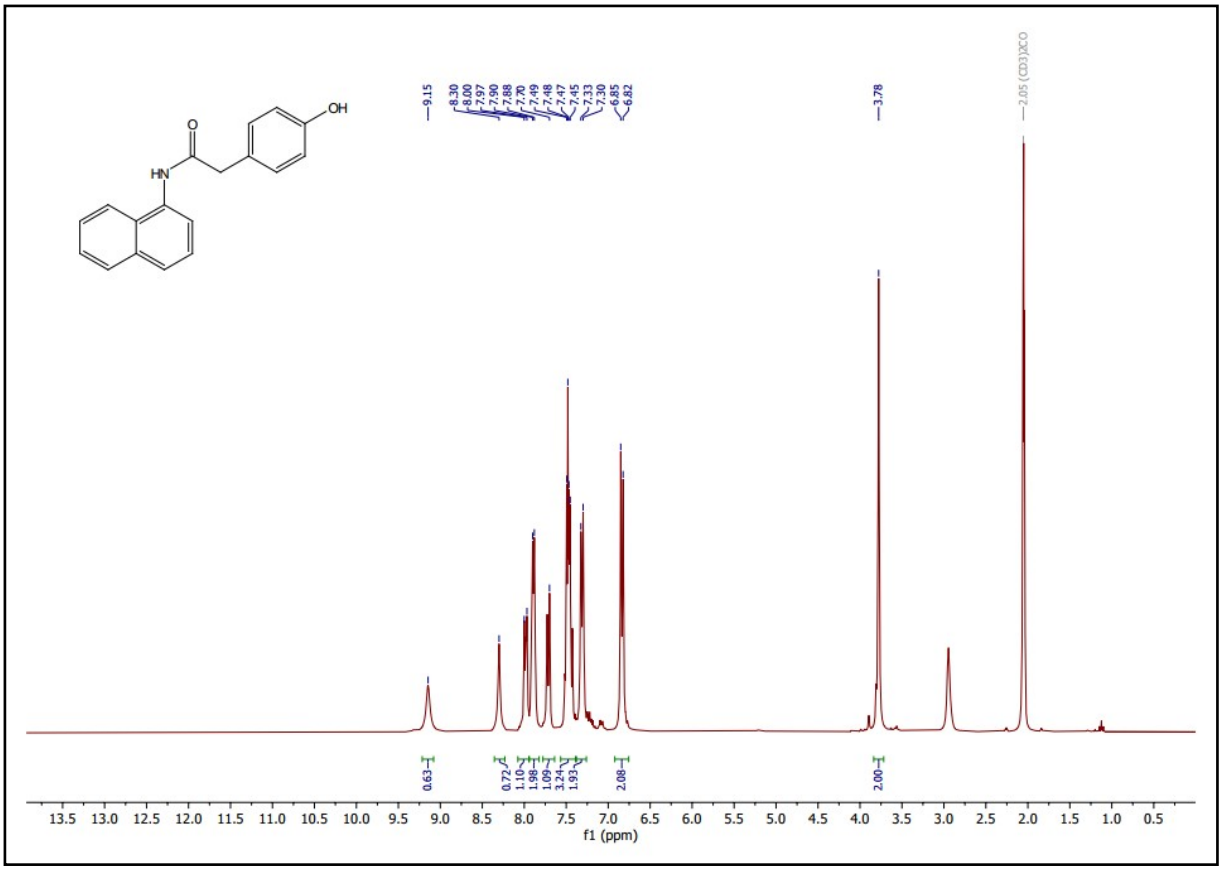


Figure S12. ^{1}H NMR spectrum of 2-(4-hydroxyphenyl)-*N*-(naphthalen-1-yl)-acetamide (**7**) in acetone-d_6 as solvent.

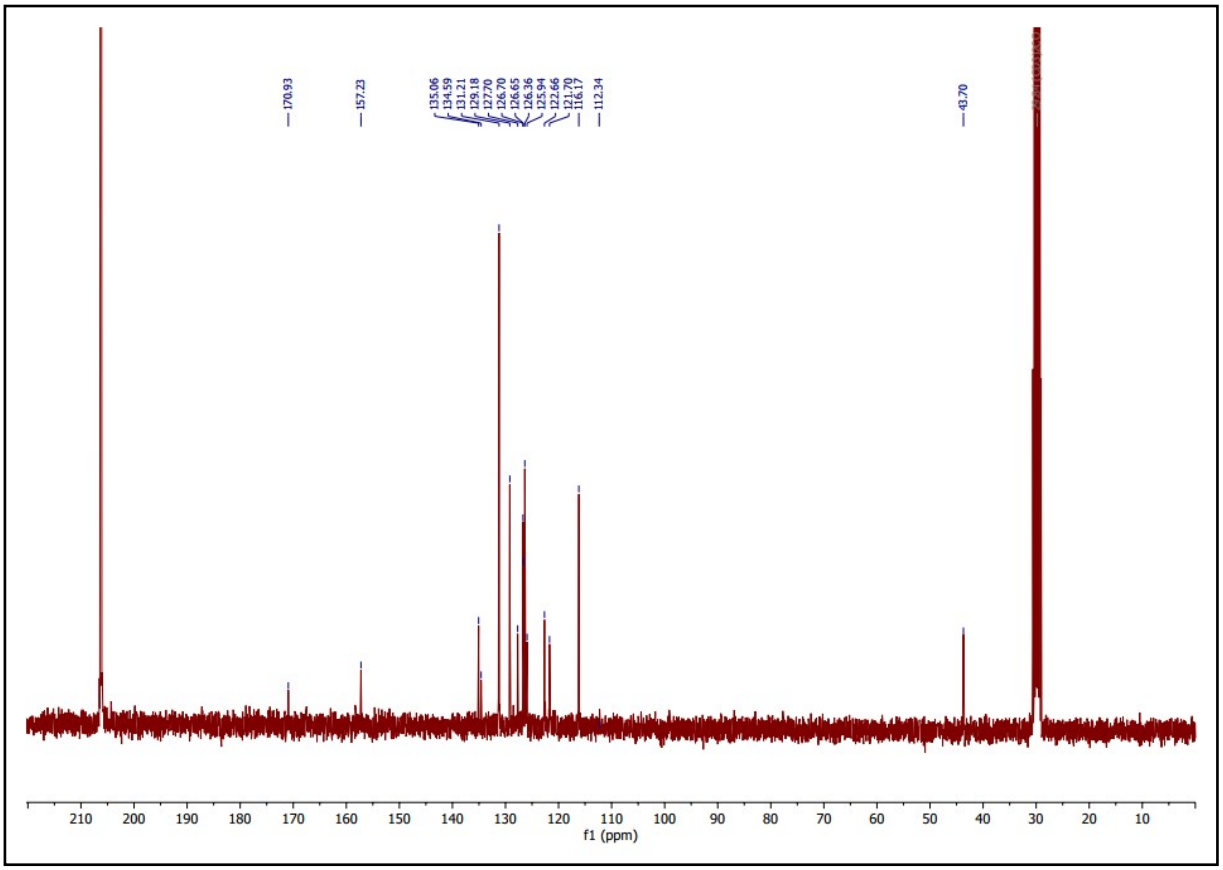


Figure S13. ^{13}C NMR spectrum of 2-(4-hydroxyphenyl)-N-(naphthalen-1-yl)-acetamide (**7**) in acetone-d_6 as solvent.

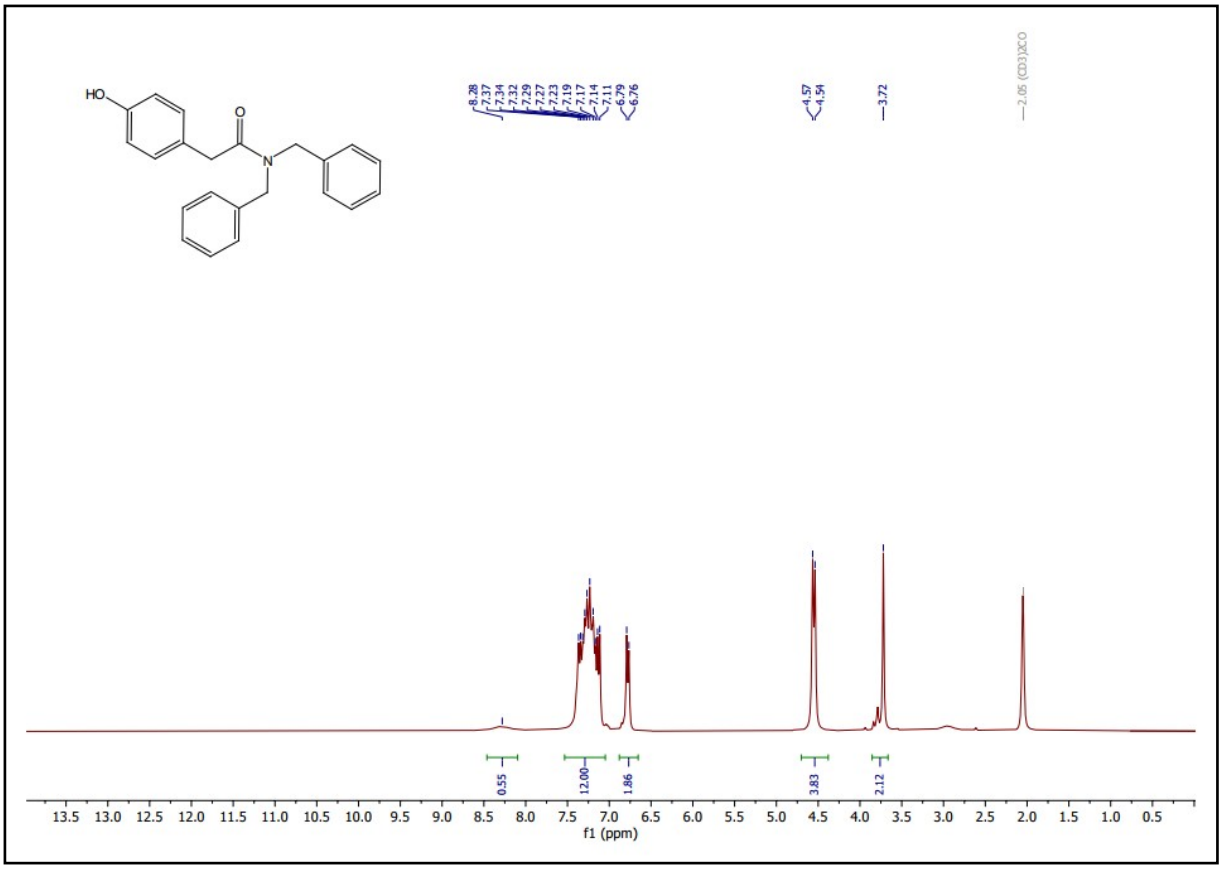


Figure S14. ^{1}H NMR spectrum of *N*,*N*-dibenzyl-2-(4-hydroxyphenyl)-acetamide (**8**) in acetone-d_6 as solvent.

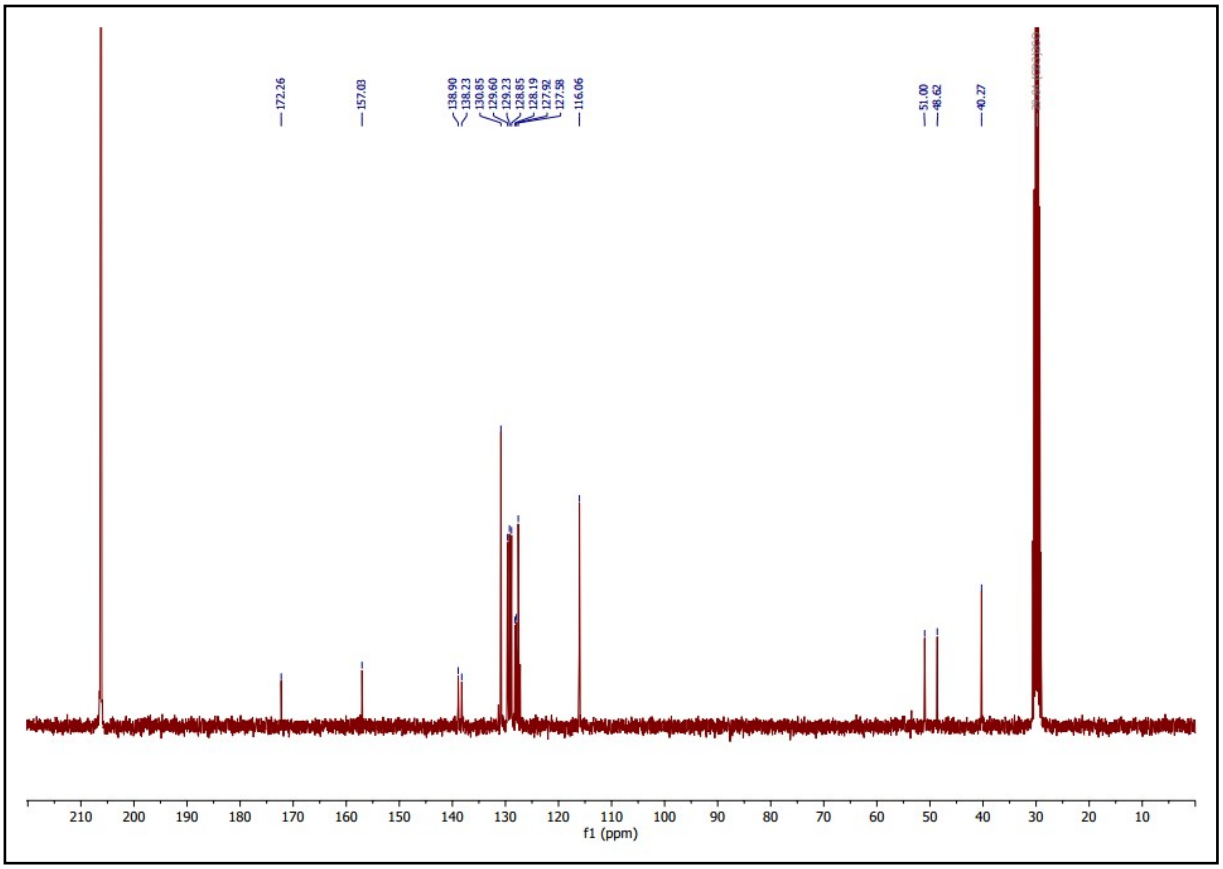


Figure S15. ^{13}C NMR spectrum of *N*,*N*-dibenzyl-2-(4-hydroxyphenyl)-acetamide (**8**) in acetone-d_6 as solvent.

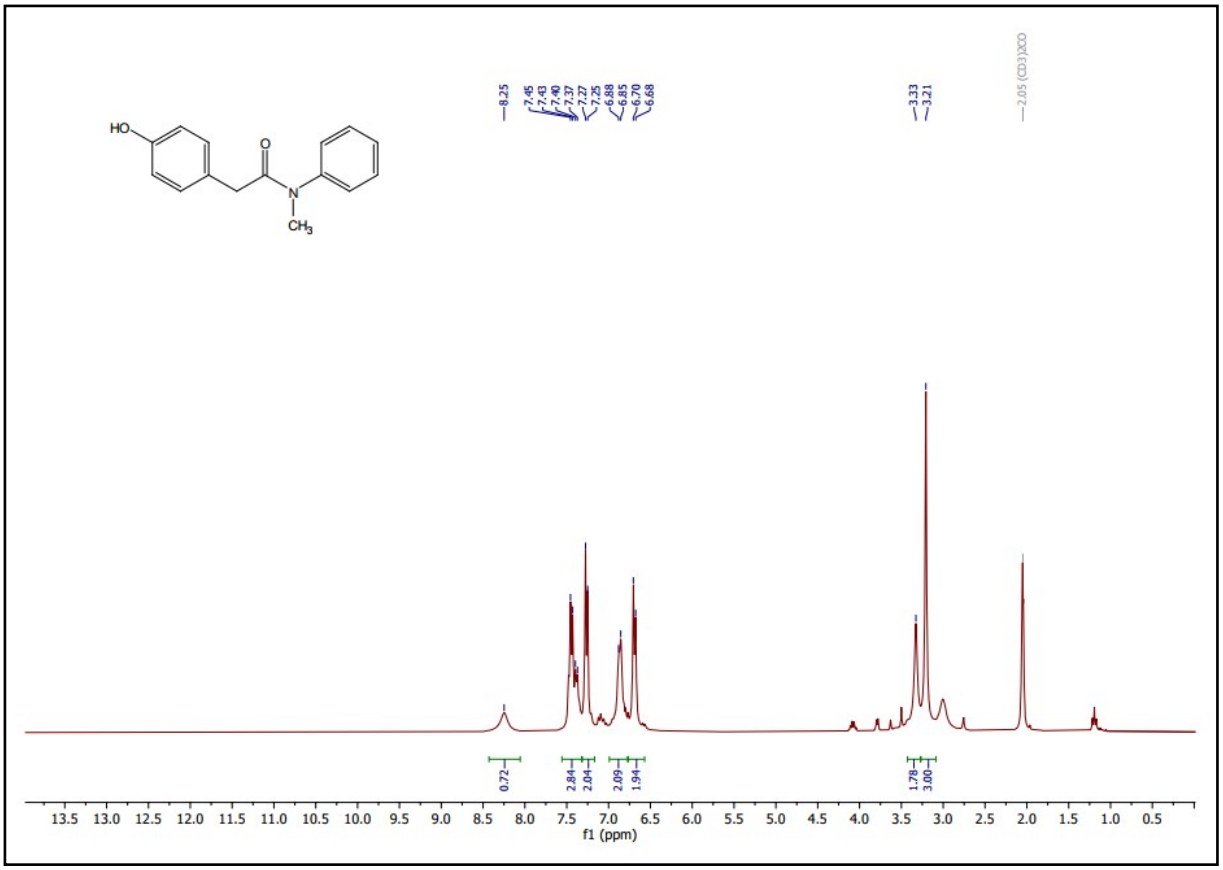


Figure S16. ^{1}H NMR spectrum of 2-(4-hydroxyphenyl)-*N*-methyl-*N*-phenylacetamide (**9**) in acetone-d_6 as solvent.

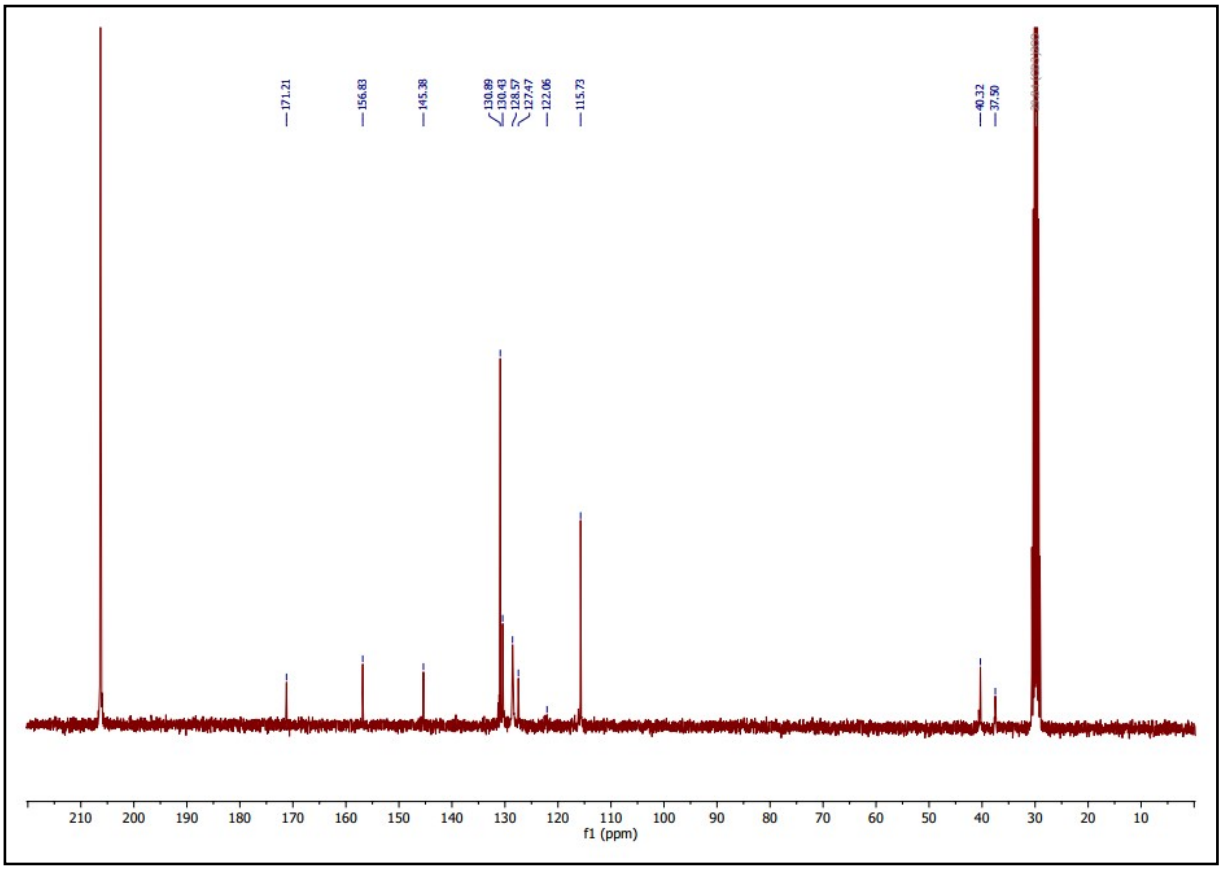


Figure S17. ^{13}C NMR spectrum of 2-(4-hydroxyphenyl)-*N*-methyl-*N*-phenylacetamide (**9**) in acetone-d_6 as solvent

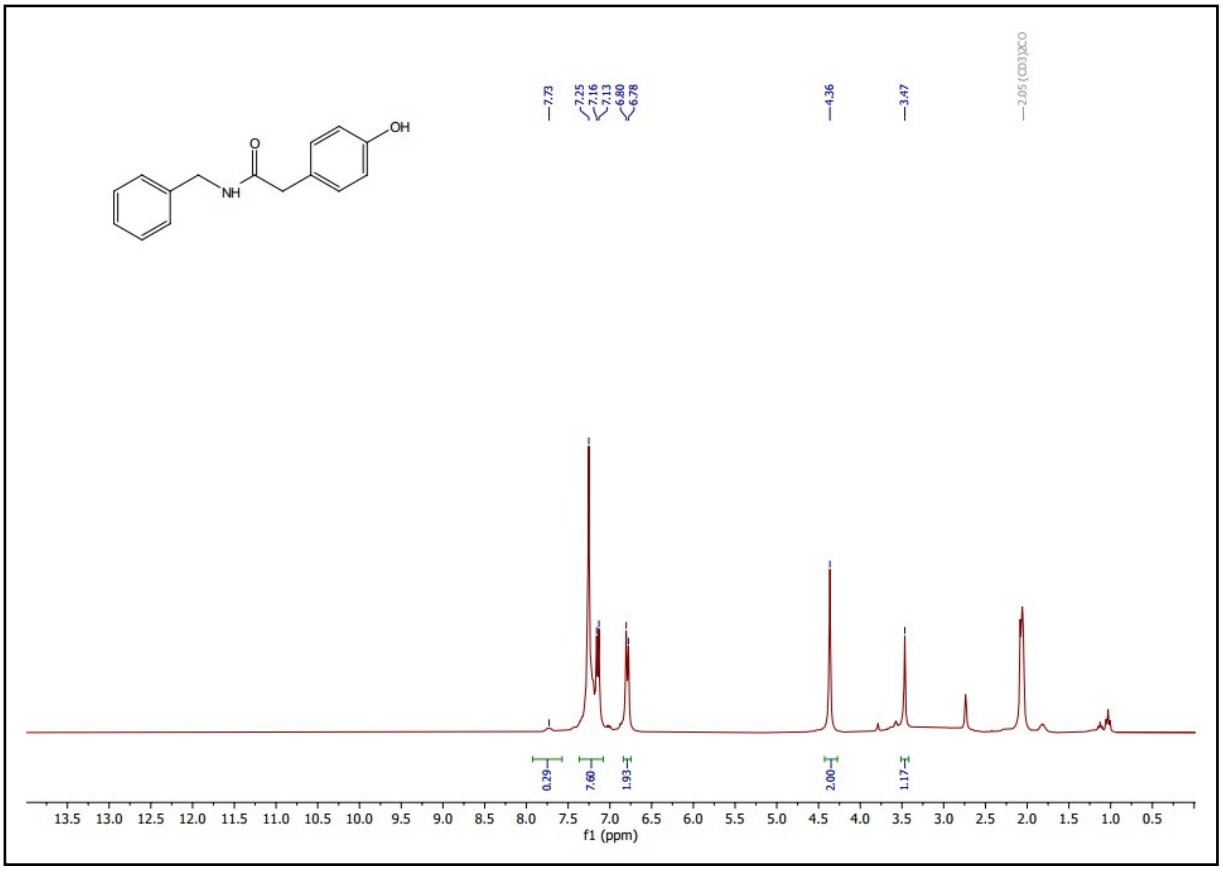


Figure S18. ^{1}H NMR spectrum of *N*-benzyl-2-(4-hydroxyphenyl)-acetamide (**10**) in acetone-d_6 as solvent.

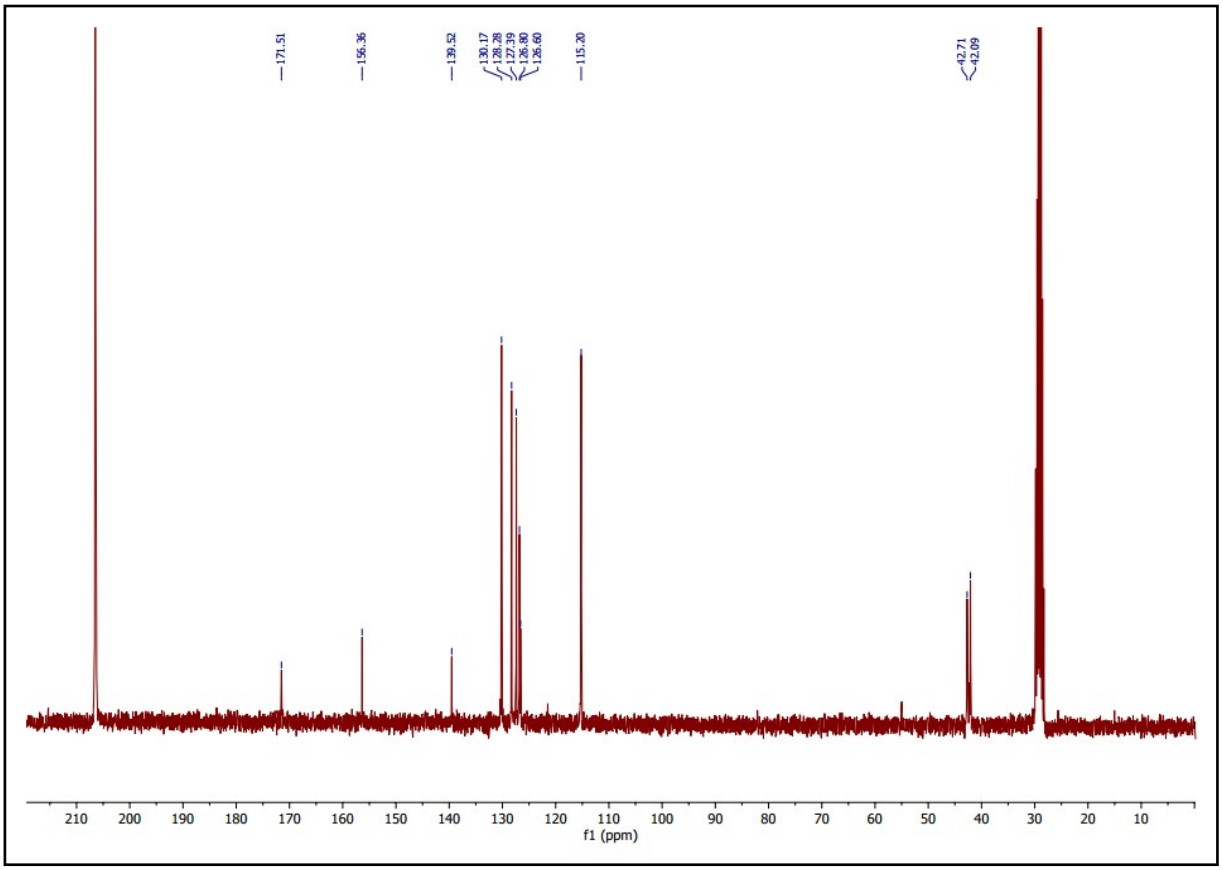


Figure S19. ^{13}C NMR spectrum of *N*-benzyl-2-(4-hydroxyphenyl)-acetamide (**10**) in acetone-d_6 as solvent.

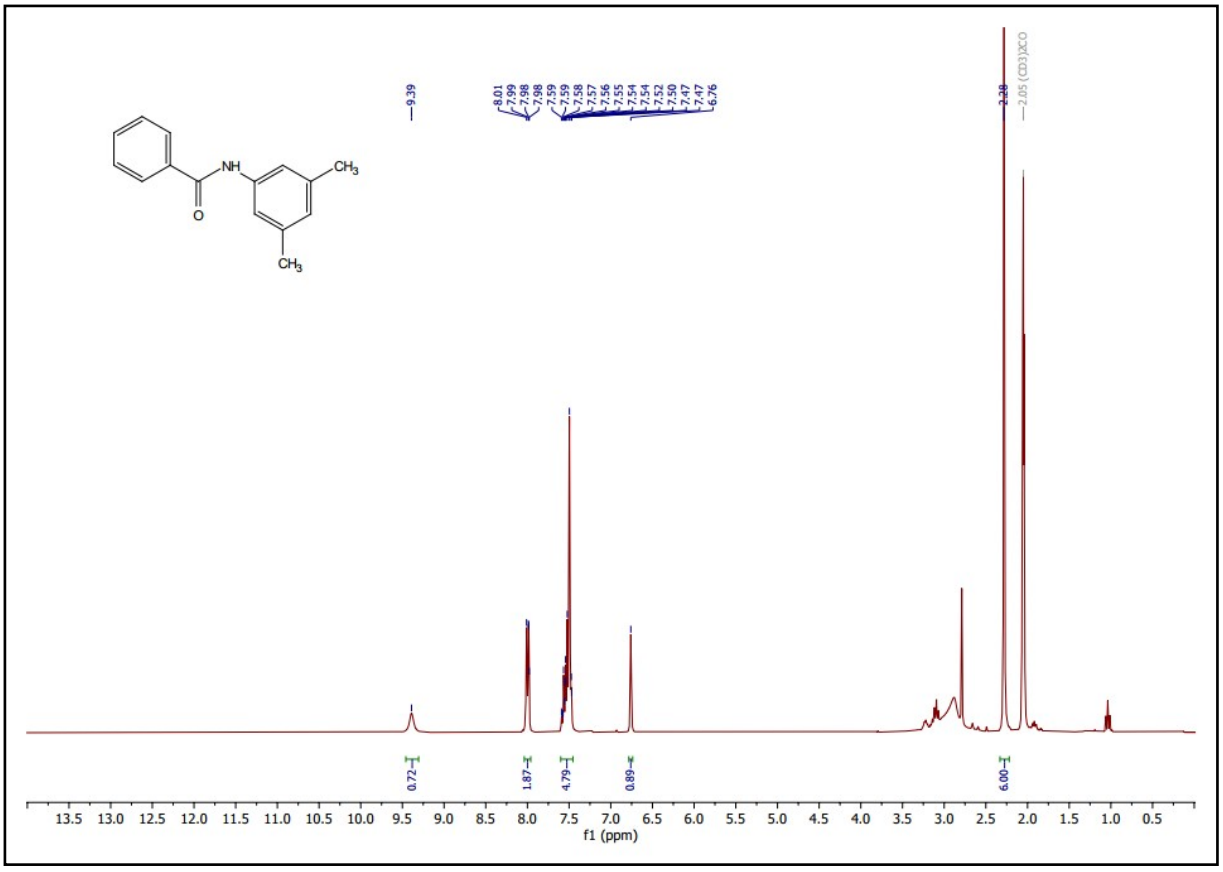


Figure S20. ^{1}H NMR spectrum of *N*-(3,5-dimethylphenyl)-benzamide (**11**) in acetone-d_6 as solvent.

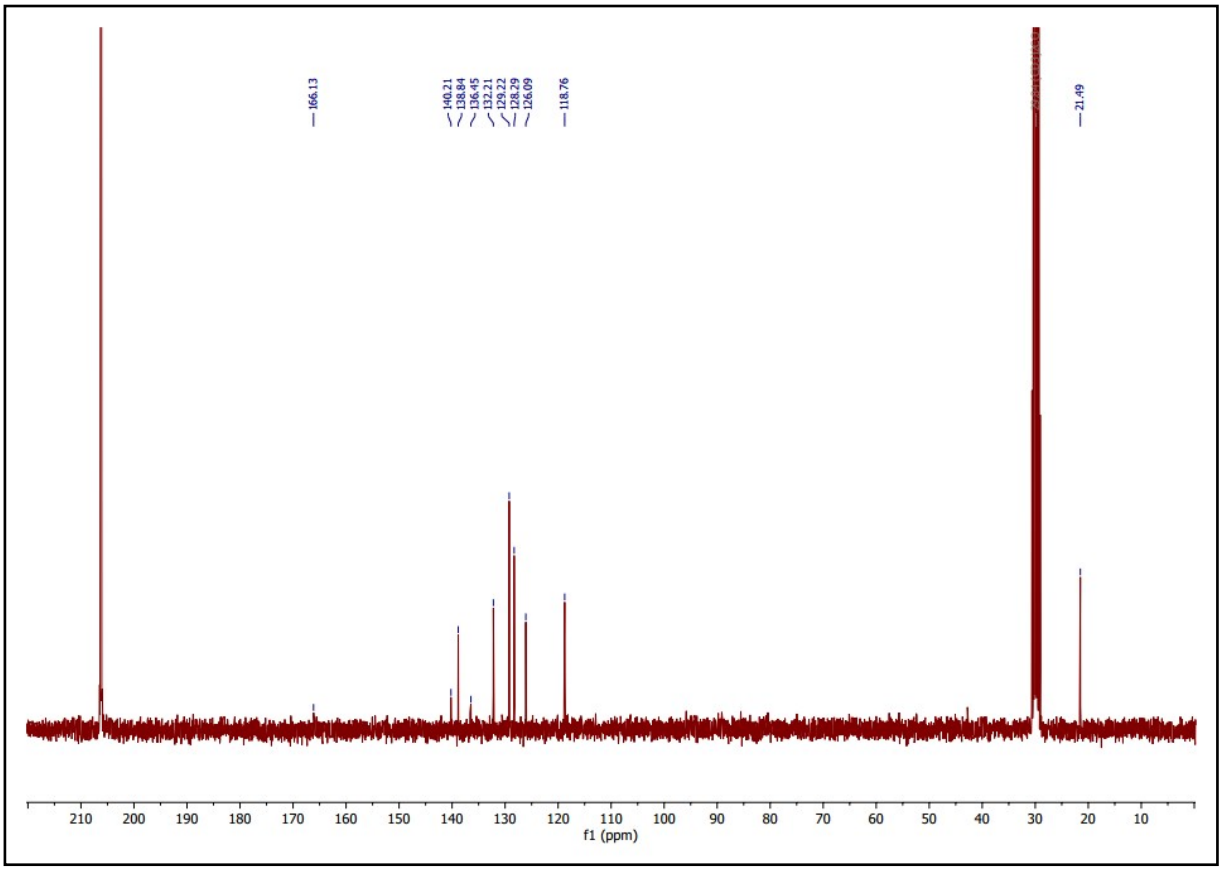


Figure S21. ^{13}C NMR spectrum of *N*-(3,5-dimethylphenyl)-benzamide (**11**) in acetone-d_6 as solvent.

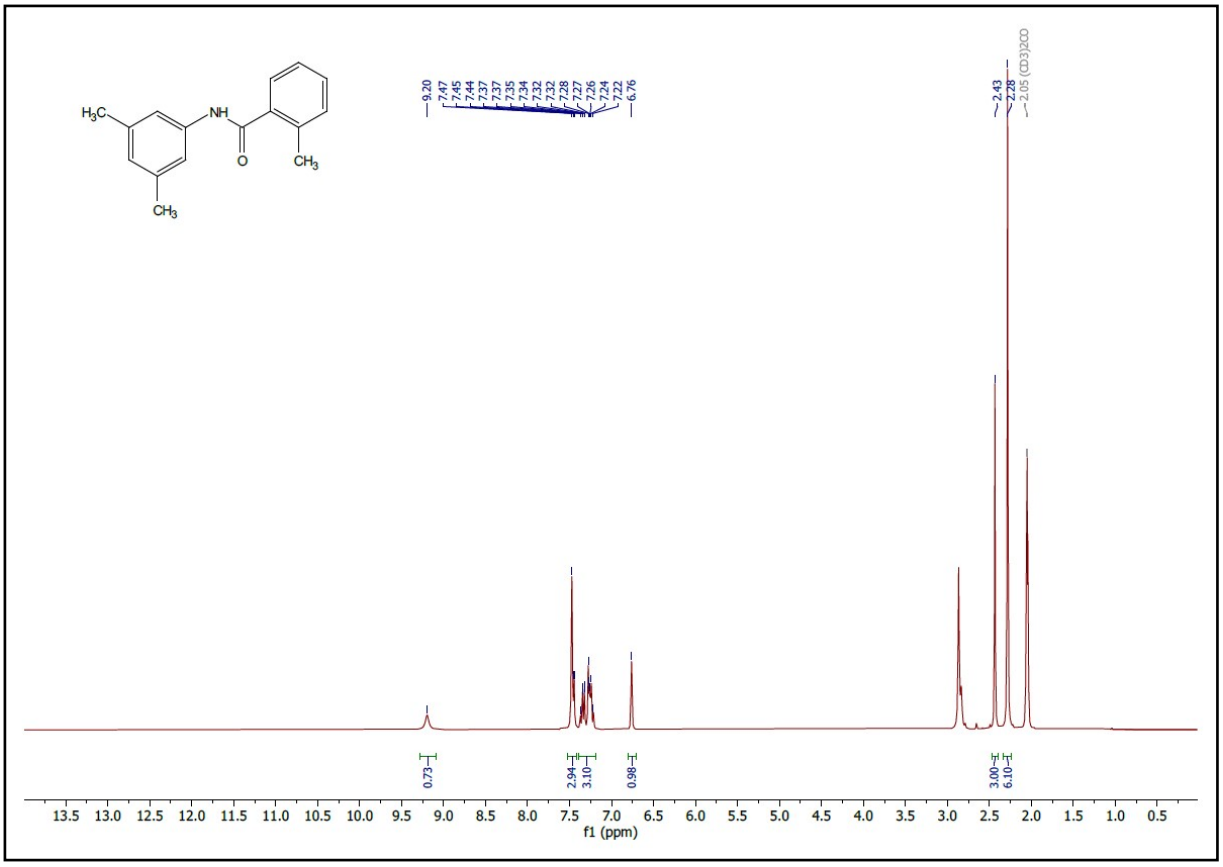


Figure S22. ^{1}H NMR spectrum of *N*-(3,5-dimethylphenyl)-2-methylbenzamide (**12**) in acetone-d_6 as solvent.

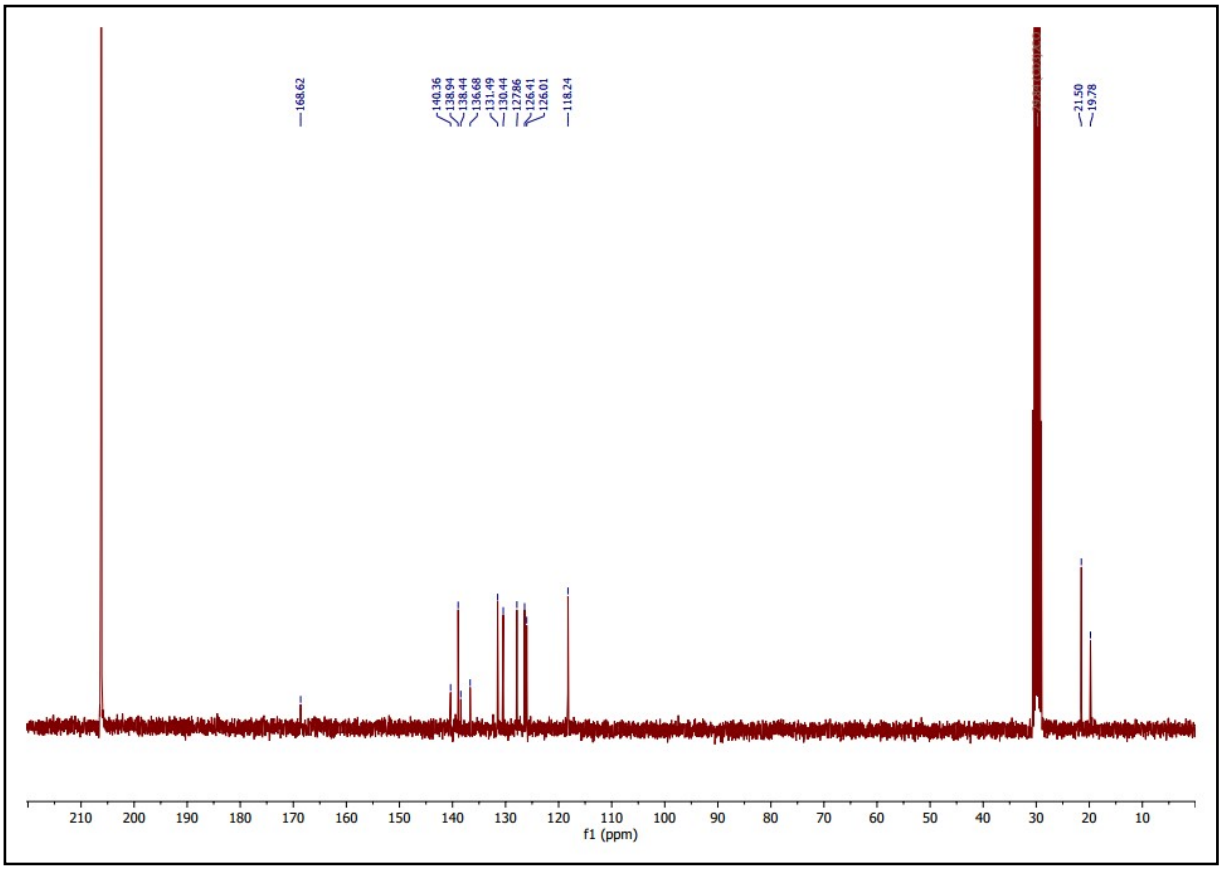


Figure S23. ^{13}C NMR spectrum of *N*-(3,5-dimethylphenyl)-2-methylbenzamide (**12**) in acetone-d_6 as solvent.

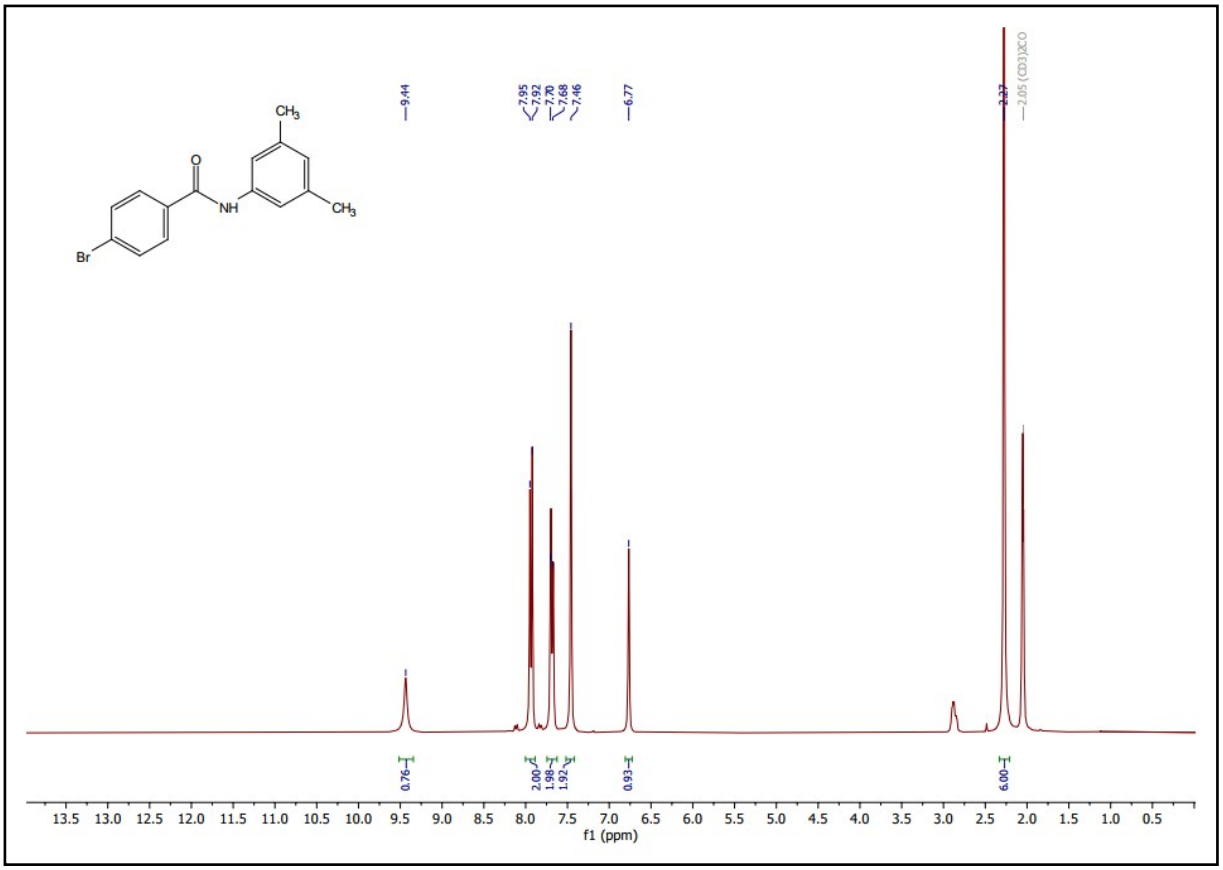


Figure S24. ^{1}H NMR spectrum of 4-bromo-*N*-(3,5-dimethylphenyl)-benzamide (**13**) in acetone-d_6 as solvent.

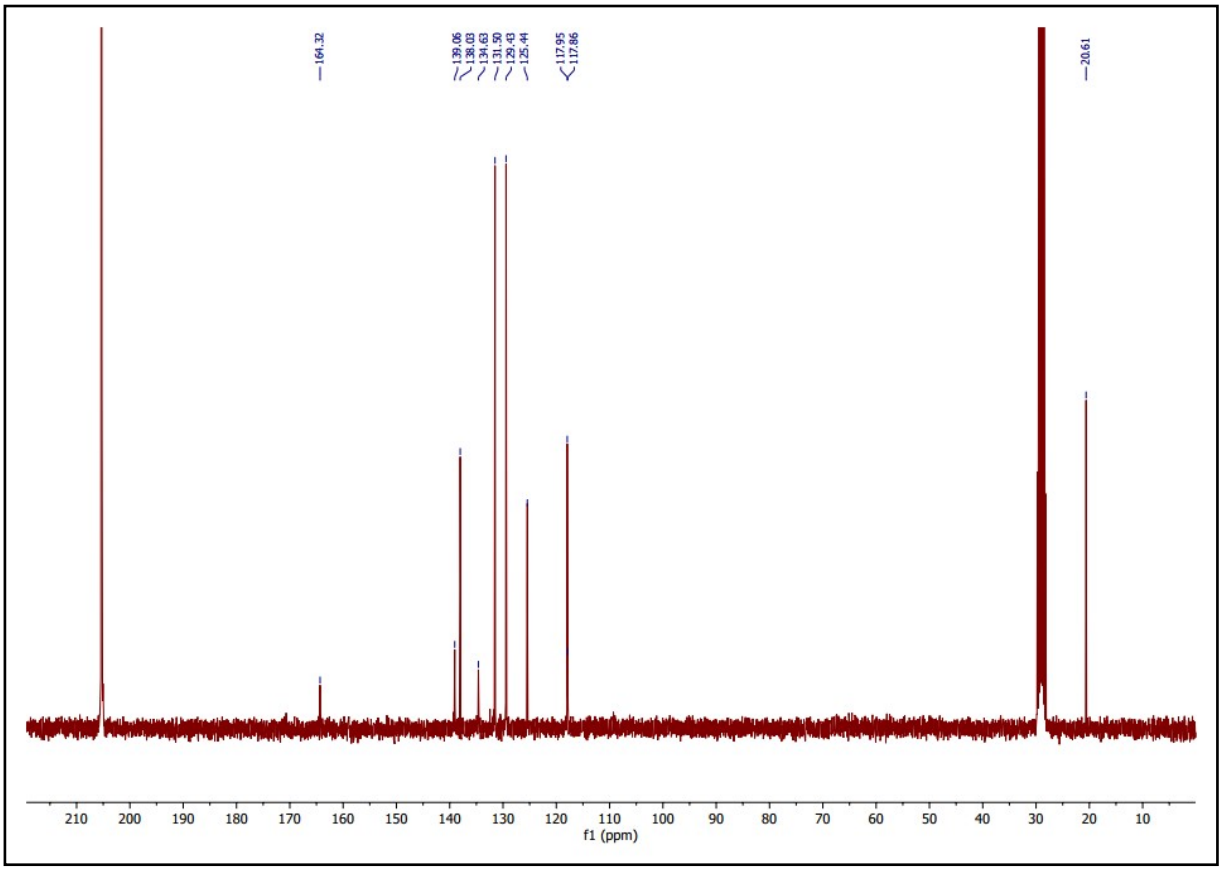


Figure S25. ^{13}C NMR spectrum of 4-bromo-*N*-(3,5-dimethylphenyl)-benzamide (**13**) in acetone-d_6 as solvent.

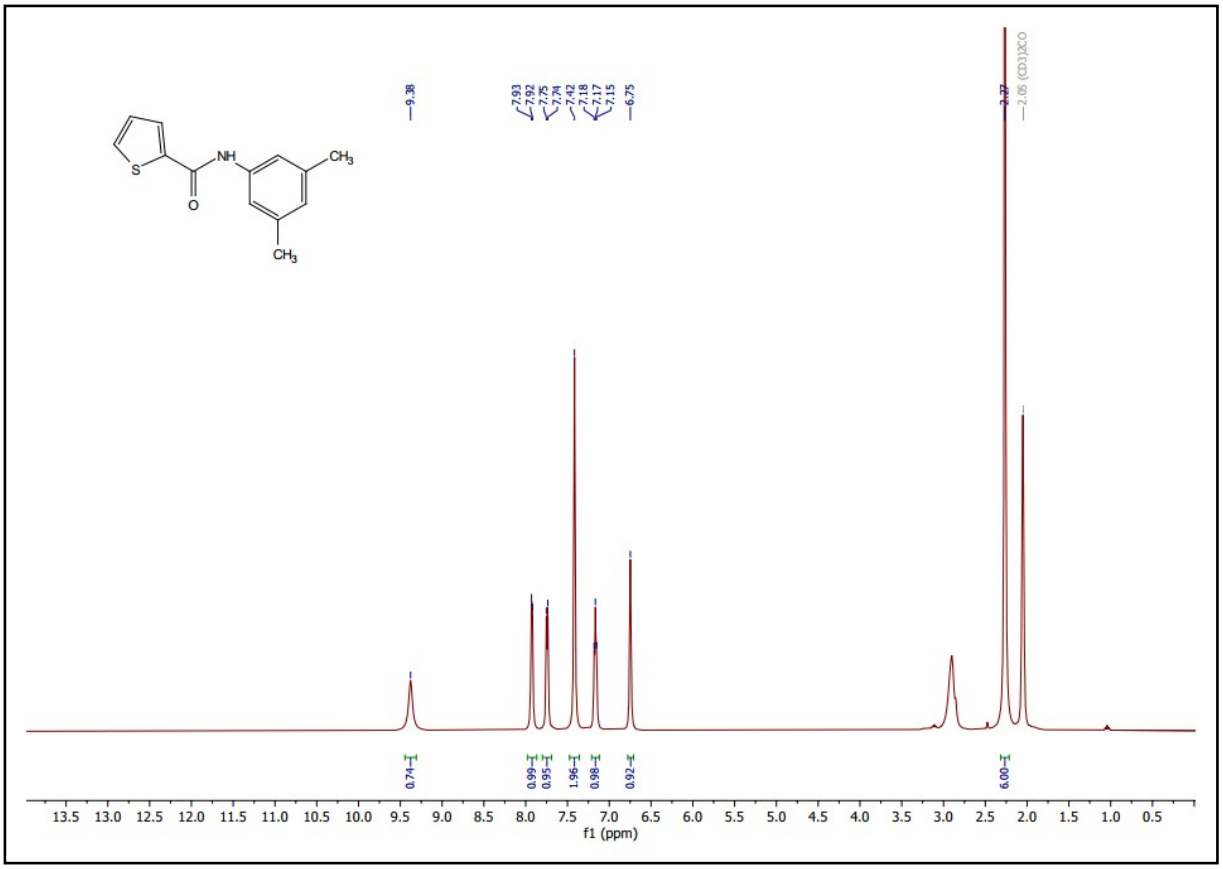


Figure S26. ^{1}H NMR spectrum of *N*-(3,5-dimethylphenyl)thiophene-2-carboxamide (**14**) in acetone-d_6 as solvent.

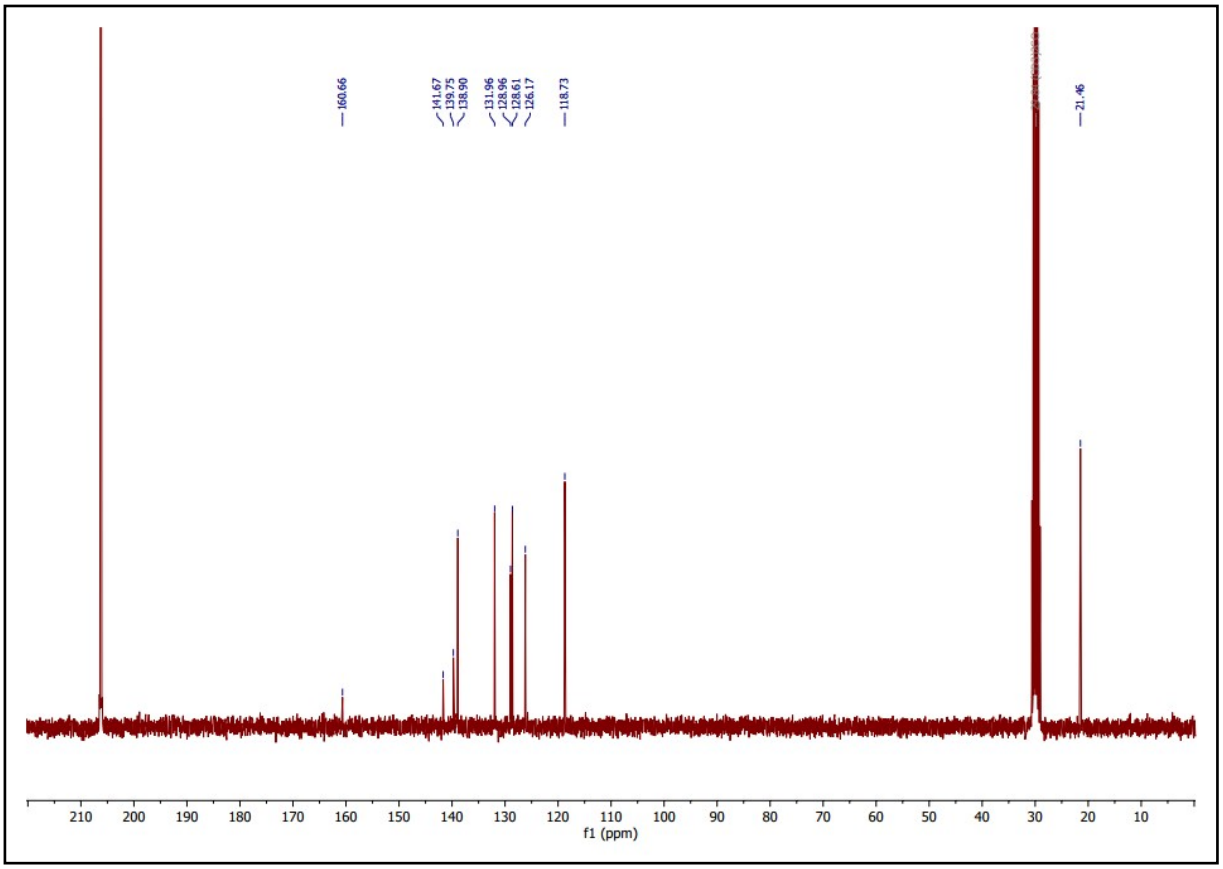


Figure S27. ^{13}C NMR spectrum of *N*-(3,5-dimethylphenyl)thiophene-2-carboxamide (**14**) in acetone-d_6 as solvent.

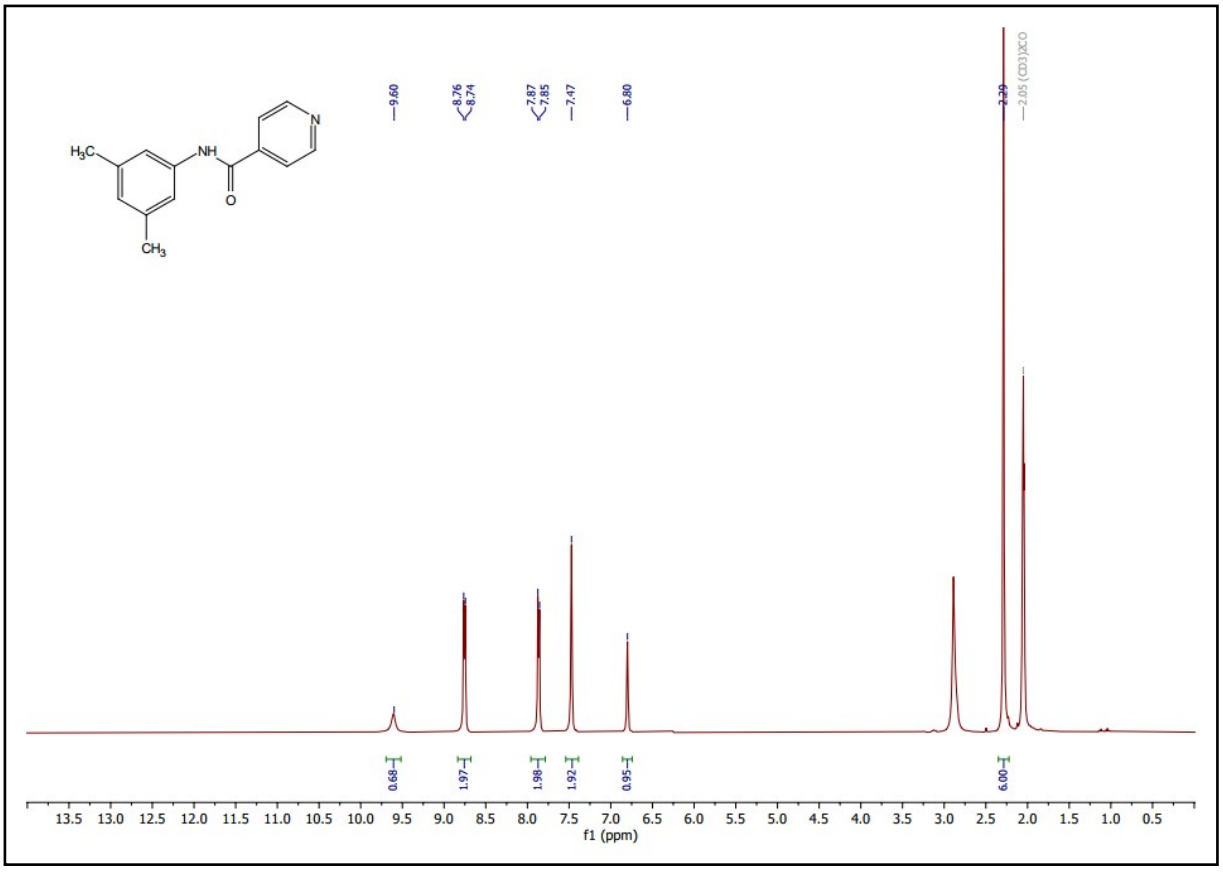


Figure S28. ^{1}H NMR spectrum of *N*-(3,5-dimethylphenyl)isonicotinamide (**15**) in acetone-d_6 as solvent.

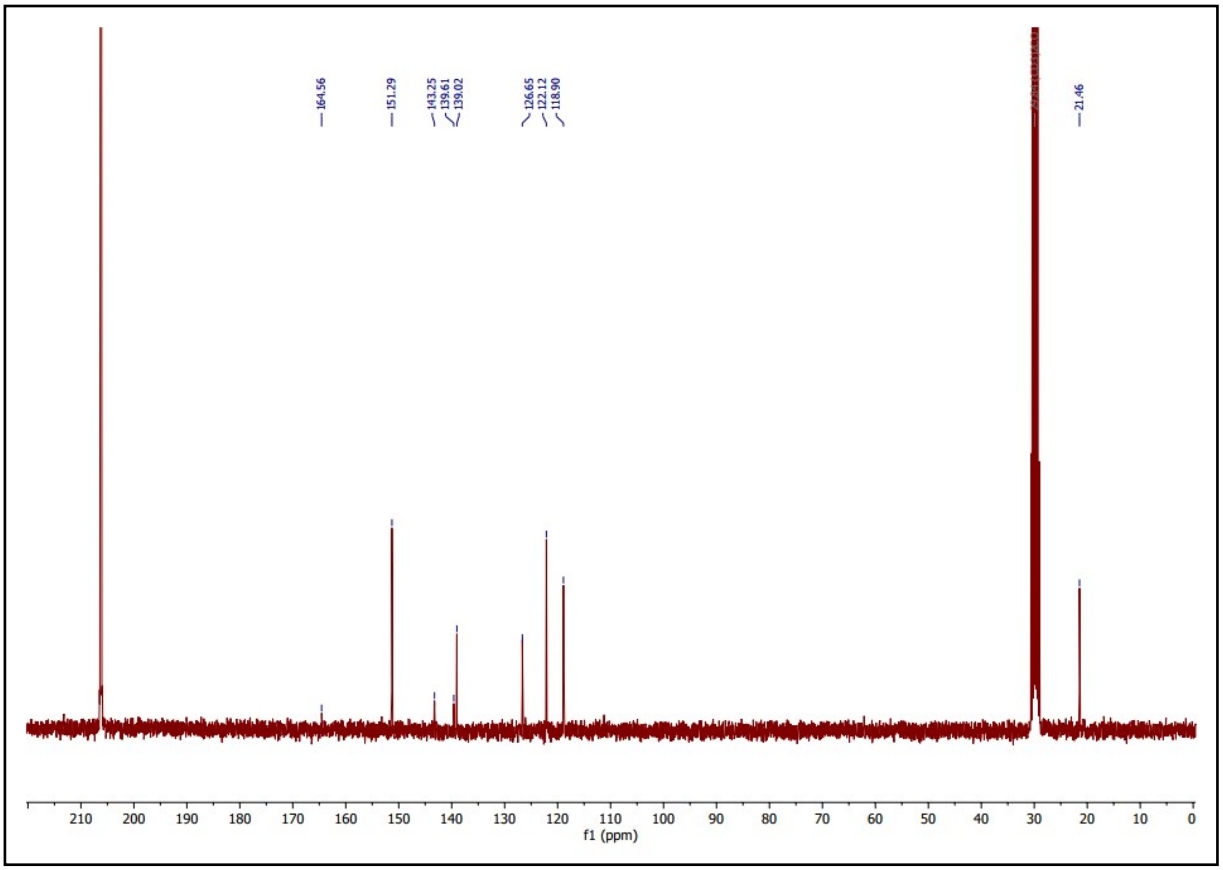


Figure S29. ^{13}C NMR spectrum of *N*-(3,5-dimethylphenyl)isonicotinamide (**15**) in acetone-d_6 as solvent.

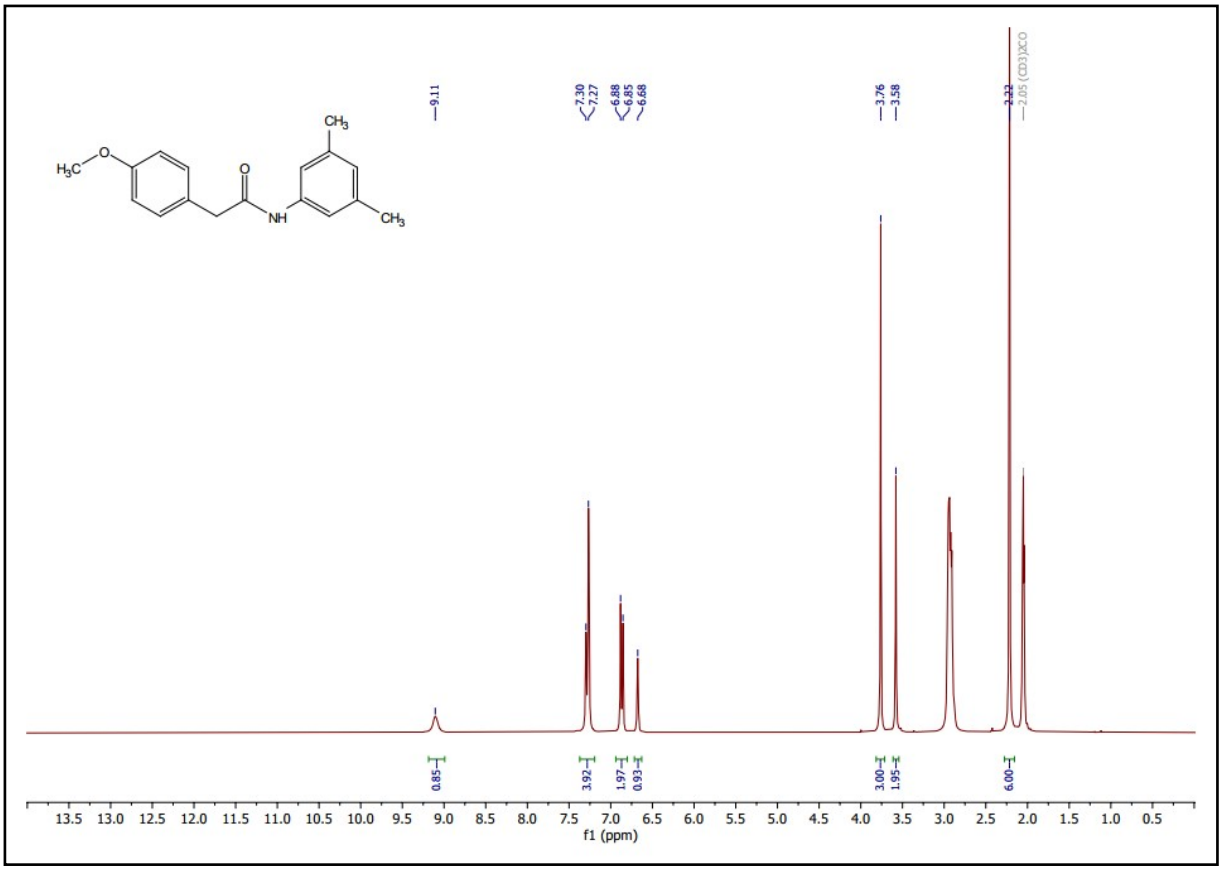


Figure S30. ^{1}H NMR spectrum of *N*-(3,5-dimethylphenyl)-2-(4-methoxyphenyl)acetamide (**16**) in acetone-d_6 as solvent.

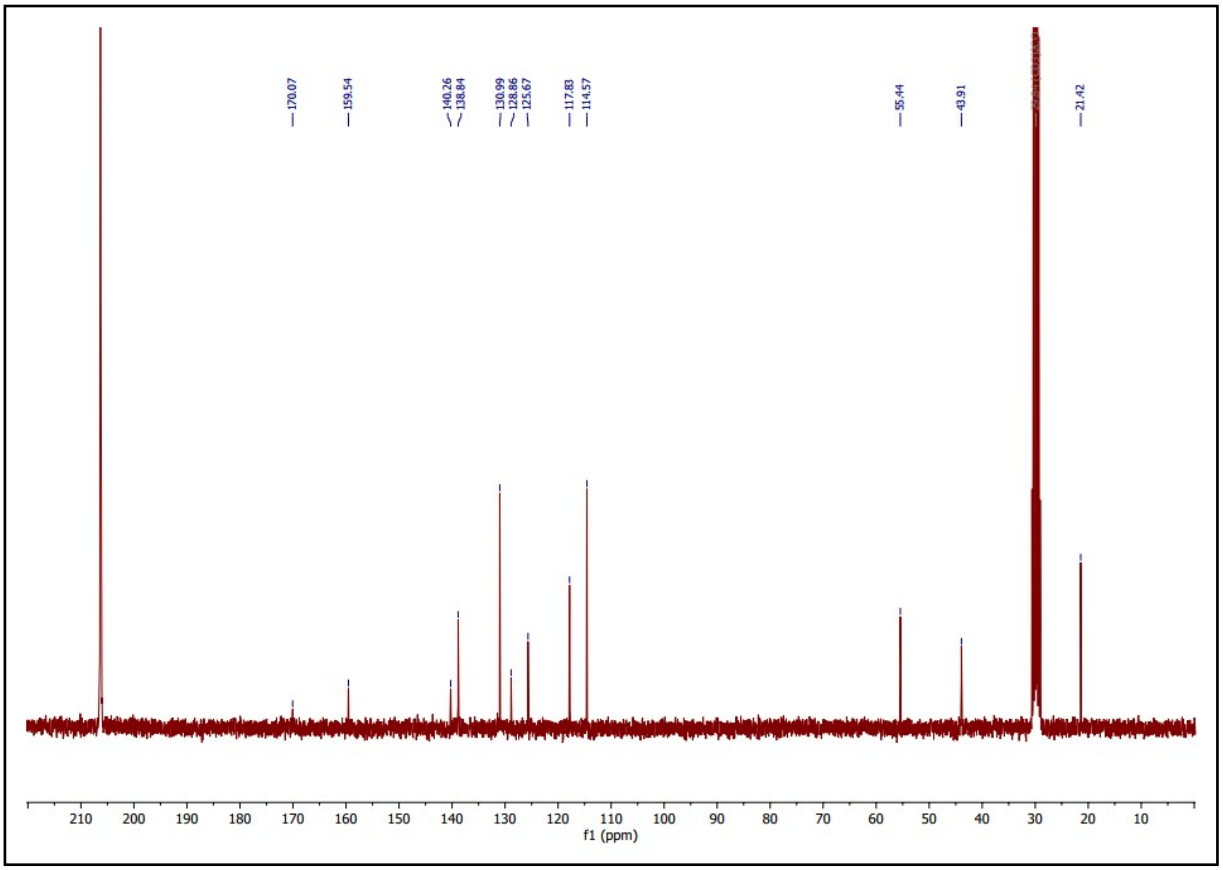


Figure S31. ^{13}C NMR spectrum of *N*-(3,5-dimethylphenyl)-2-(4-methoxyphenyl)acetamide (**16**) in acetone-d_6 as solvent.

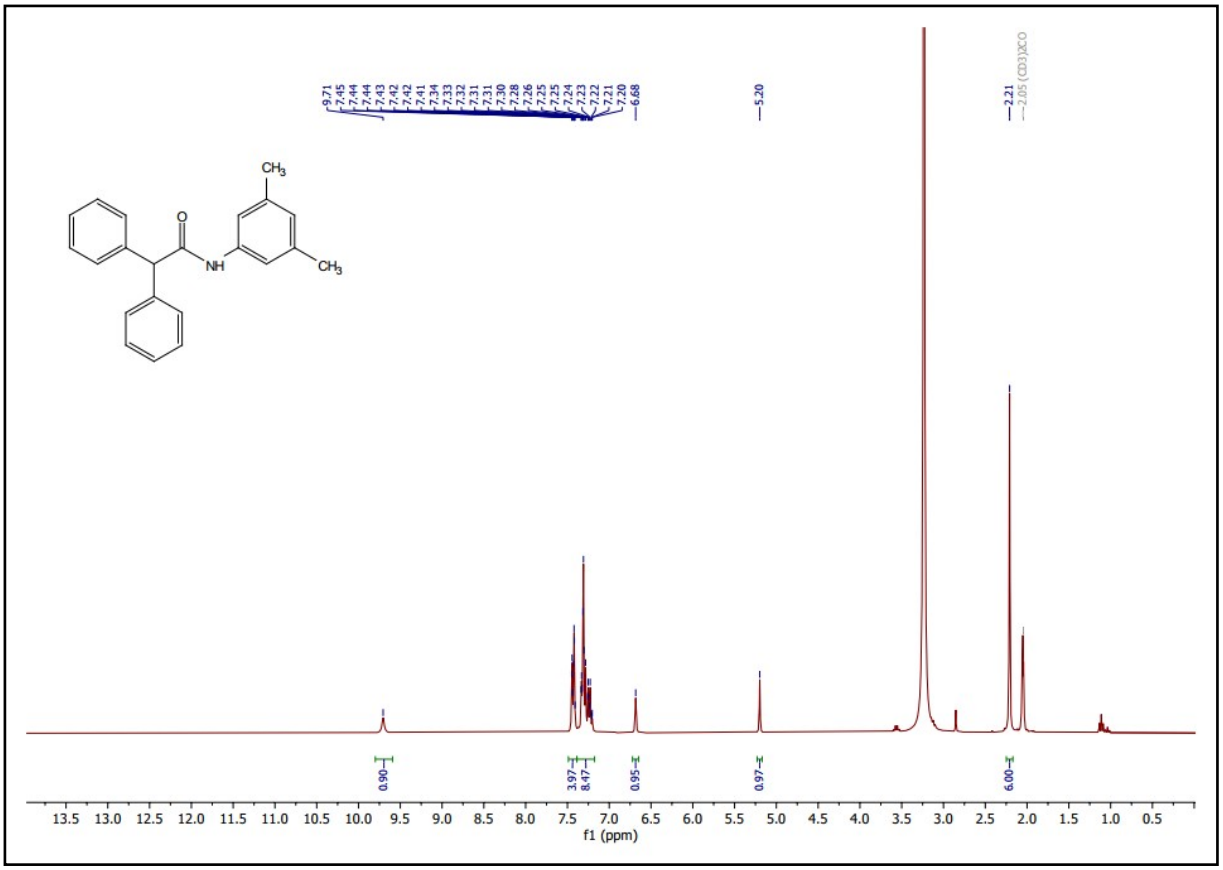


Figure S32. ^{1}H NMR spectrum of *N*-(3,5-dimethylphenyl)-2,2-diphenylacetamide (**17**) in acetone-d_6 as solvent.

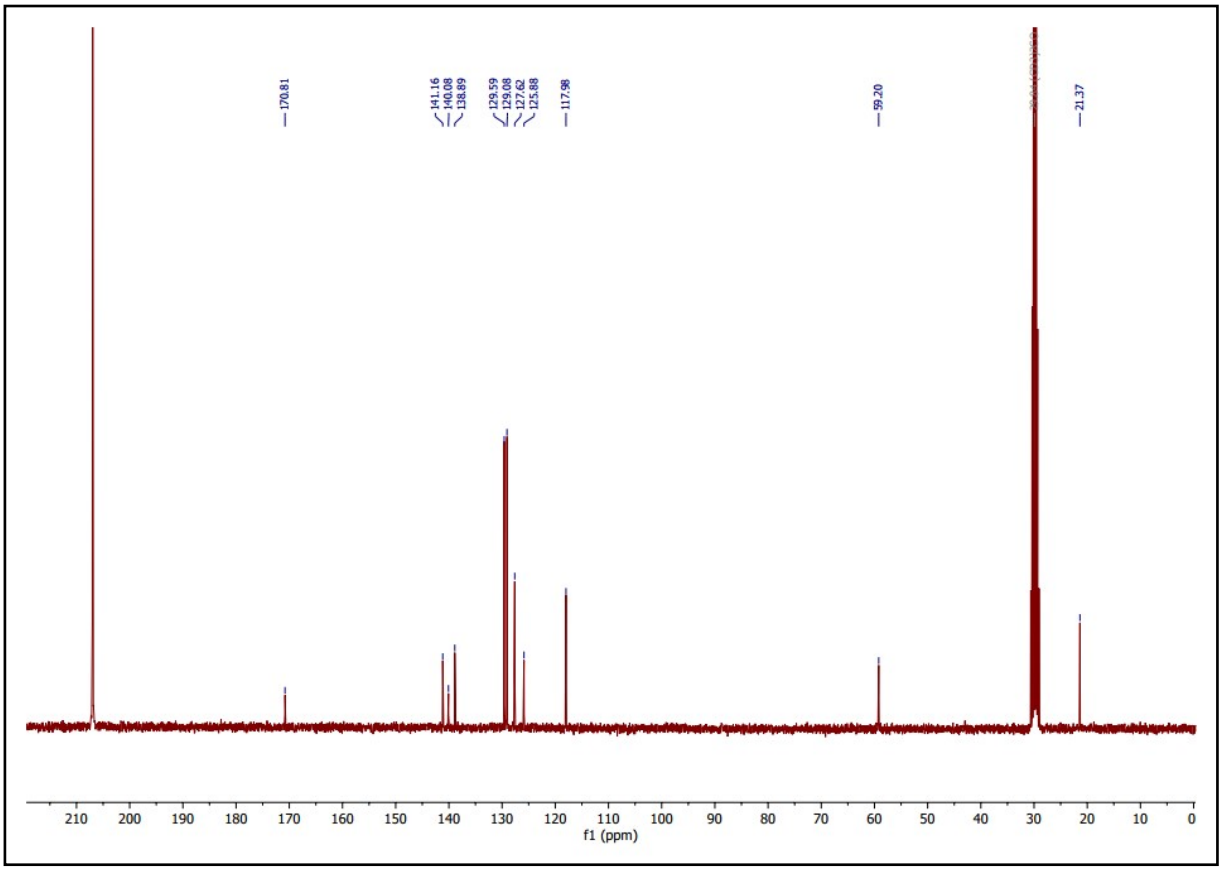


Figure S33. ^{13}C NMR spectrum of *N*-(3,5-dimethylphenyl)-2,2-diphenylacetamide (**17**) in acetone-d_6 as solvent.

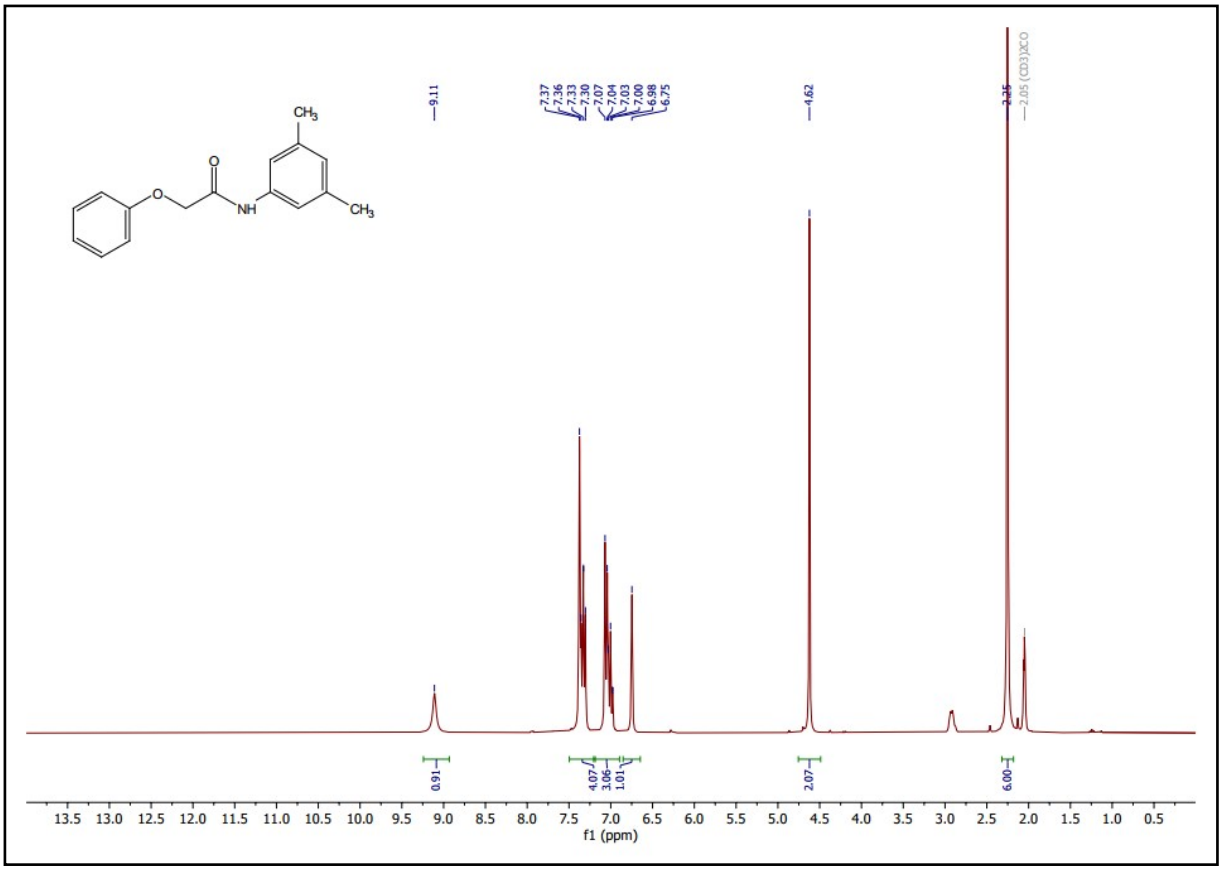


Figure S34. ^{1}H NMR spectrum of *N*-(3,5-Dimethylphenyl)-2-phenoxyacetamide (**18**) in acetone-d_6 as solvent.

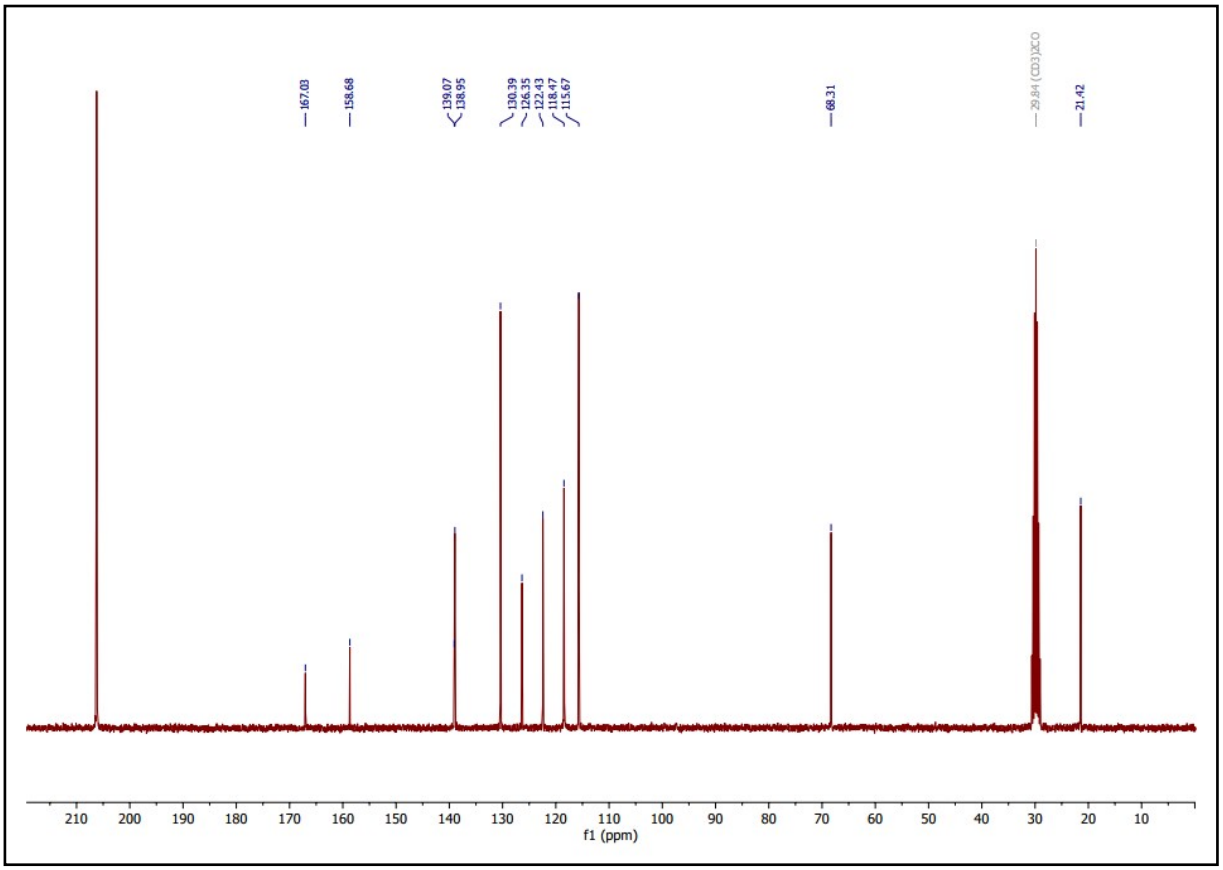


Figure S35. ^{13}C NMR spectrum of *N*-(3,5-Dimethylphenyl)-2-phenoxyacetamide (**18**) in acetone-d_6 as solvent.

6. References

(S1) https://www.wab-group.com/en/products/dyno-mill-research-lab/.

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