Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supplementary Information

Rhodium-catalyzed sequential intermolecular hydroacylation and deconjugative isomerization toward diversified diketones

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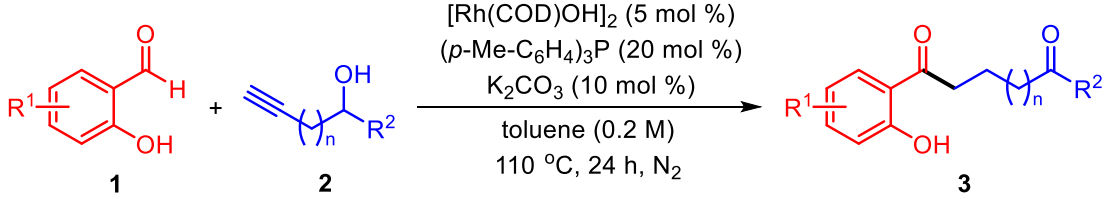
1. General Experiment Information

The ^{1}H NMR (400 MHz) and ^{13}C NMR spectra (100 MHz) were recorded on the Bruker Ascend™ 400 Spectrometer using $CDCl_3$ as the solvent. Chemical shifts are given in ppm and coupling constants in Hertz (Hz). ^{1}H spectra were calibrated in relation to the reference measurement of TMS (0.000 ppm) or the residual solvent signal of $CDCl_3$ (7.260 ppm). ^{13}C spectra were calibrated in relation to $CDCl_3$ (77.10 ppm). The following abbreviations were used for ^{1}H NMR spectra to indicate the signal multiplicities: s (singlet), d (doublet), t (triplet), q (quartet) and m (multiplets) as well as combinations of them. Analytical thin layer chromatography (TLC) was performed on 0.25 mm silica gel plates (purchased from Qingdao Haiyang Chemical, China), and the products were visualized with the UV light at 254 nm and 365 nm. Column chromatography was performed on silica gel 200−300 mesh (purchased from Qingdao Haiyang Chemical, China). High-resolution mass spectra (HRMS) using electrospray ionization (ESI) as the ion source was carried out by LC−MSD TOF using a column of C18 (rapid resolution, 3.5 μm, 2.1 mm × 30 mm) at a flow of 0.40 mL/min.

Unless specified otherwise, all chemicals of commercial grade were used without further purification. Organic solvent was concentrated under reduced pressure on a EYELA rotary evaporator (Japan). All of the salicylaldehydes and benzaldehyde were commercially available. Toluene as the solvent was purified and dried according to the standard method prior to use. Other anhydrous solvents such as DCM and THF were commercially available.

2. General Procedures

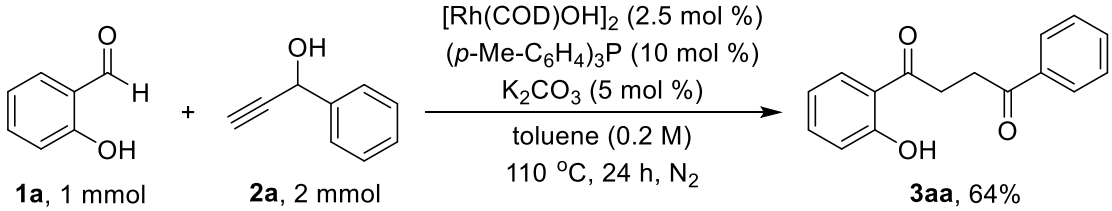
(1) General Procedure for the Synthesis of Diketone 3



To an oven-dried sealed tube (10 mL) equipped with a stirrer bar in the glove box

(filled with N_2) was added $[Rh(COD)OH]_2$ (4.6 mg, 0.01 mmol, 5 mol %), tri(*p*-tolyl)phosphine (12.2 mg, 0.04 mmol, 20 mol %), K_2CO_3 (2.8 mg, 0.02 mmol, 10 mol %), chelating aldehyde **1** (0.2 mmol, 1.0 equiv), and alkynol **2** (0.4 mmol, 2.0 equiv). Then anhydrous toluene (1.0 mL, 0.2 M) was added. The tube was sealed and removed from the glove box. The resulting mixture was stirred at room temperature for 15 min, and heated at 110 °C for 24 h using a Heidolph MR Hei-Tec heating magnetic stirrer (Heidolph Instruments, Germany). Upon completion, the reaction mixture was cooled to room temperature and concentrated. The residue was purified by silica gel chromatography (*n*-hexane/EtOAc) to afford the desired diketone **3**.

(2) Scale-Up Synthesis of 3aa

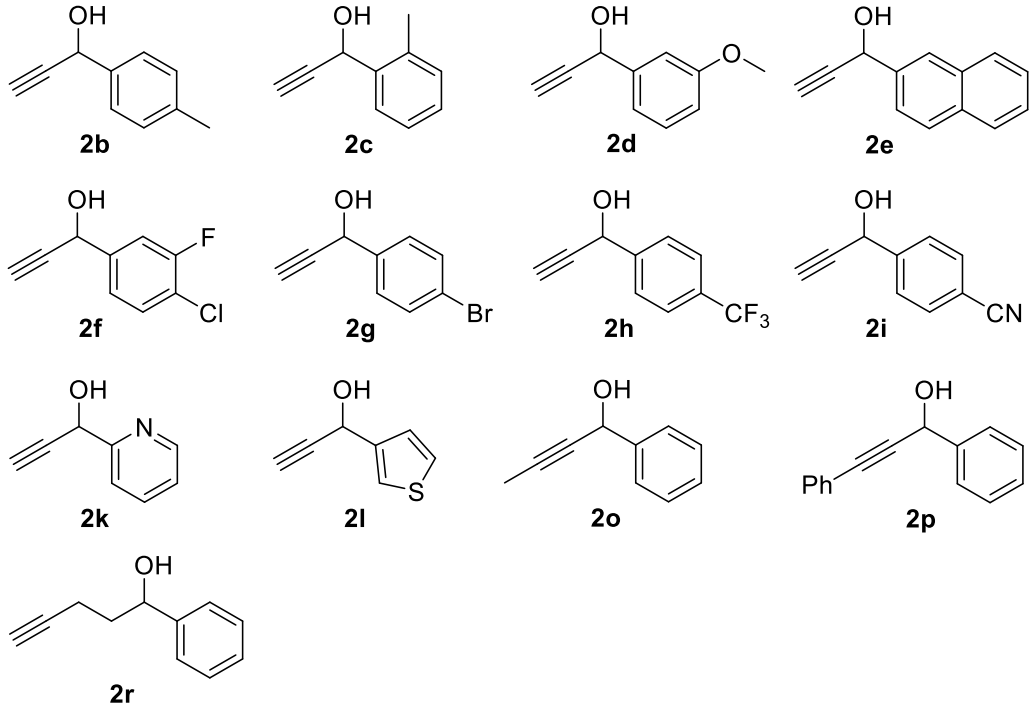


1a, 1 mmol **2a**, 2 mmol **3aa**, 64%

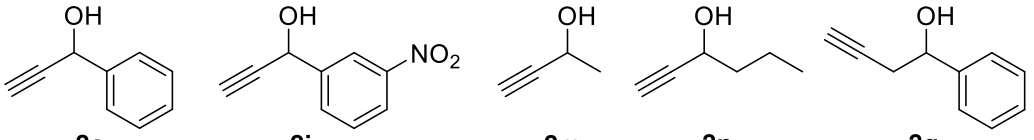
To an oven-dried sealed tube (35 mL) equipped with a stirrer bar in the glove box (filled with N_2) was added $[Rh(COD)OH]_2$ (11.4 mg, 0.025 mmol, 2.5 mol %), tri(*p*-tolyl)phosphine (30.4 mg, 0.1 mmol, 10 mol %), K_2CO_3 (6.9 mg, 0.05 mmol, 5 mol %), salicylaldehyde **1a** (122.1 mg, 1 mmol, 1.0 equiv), and 1-phenylprop-2-yn-1-ol **2a** (264.3 mg, 2 mmol, 2.0 equiv). Then anhydrous toluene (5.0 mL, 0.2 M) was added. The tube was sealed and removed from the glove box. The resulting mixture was stirred at room temperature for 15 min, and heated at 110 °C for 24 h using a Heidolph MR Hei-Tec heating magnetic stirrer (Heidolph Instruments, Germany). Upon completion, the reaction mixture was cooled to room temperature and concentrated. The residue was purified by silica gel chromatography (*n*-hexane/EtOAc = 120:1-50:1) to afford the desired 1,4-diketone **3aa** (162.8 mg, 64%) as a white solid.

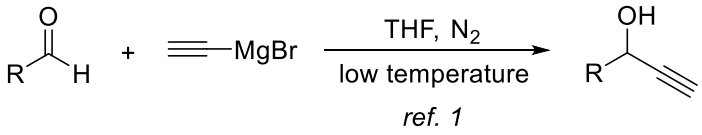
(3) General Procedure for the Synthesis of Alkynyl Alcohols 2

Readily prepared materials



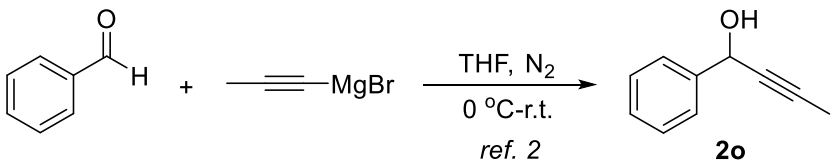
Commerially available materials





ref. 1

Alkynyl Alcohols (**2b**-**2i**, **2k**, and **2l**) were prepared through the reaction of ethynylmagnesium bromide with the corresponding aldehyde according to the literature procedure.[1] Other alkynyl alcohols (**2a**, **2j**, **2m**, **2n**, and **2q**) were commercially available.

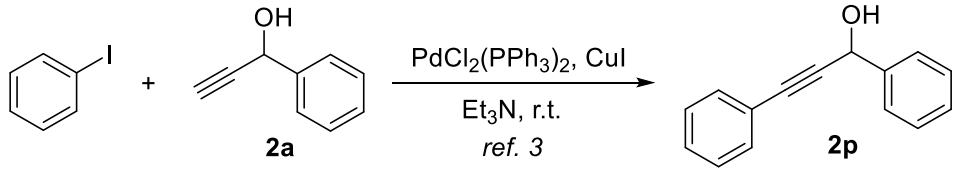


ref. 2

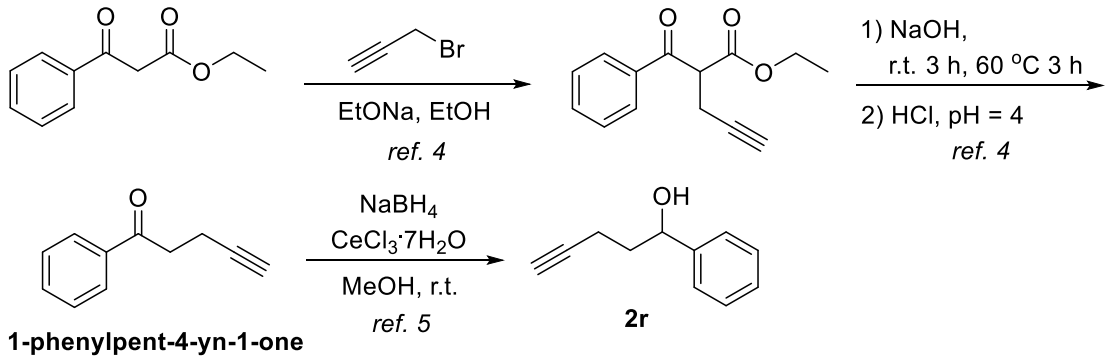
2o

1-Phenylbut-2-yn-1-ol (**2o**) was prepared through the reaction of 1-

propynylmagnesium bromide with benzaldehyde according to the literature procedure.[2]

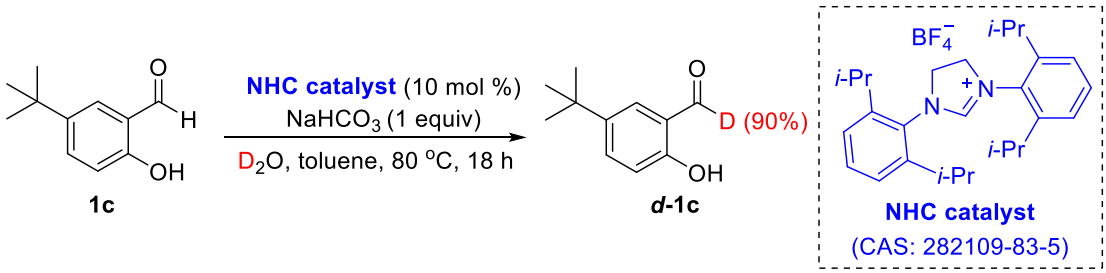


1,3-Diphenylprop-2-yn-1-ol (**2p**) was prepared through the Sonogashira cross-coupling reaction of 1-phenylprop-2-yn-1-ol (**2a**) with iodobenzene according to the literature procedure.[3]



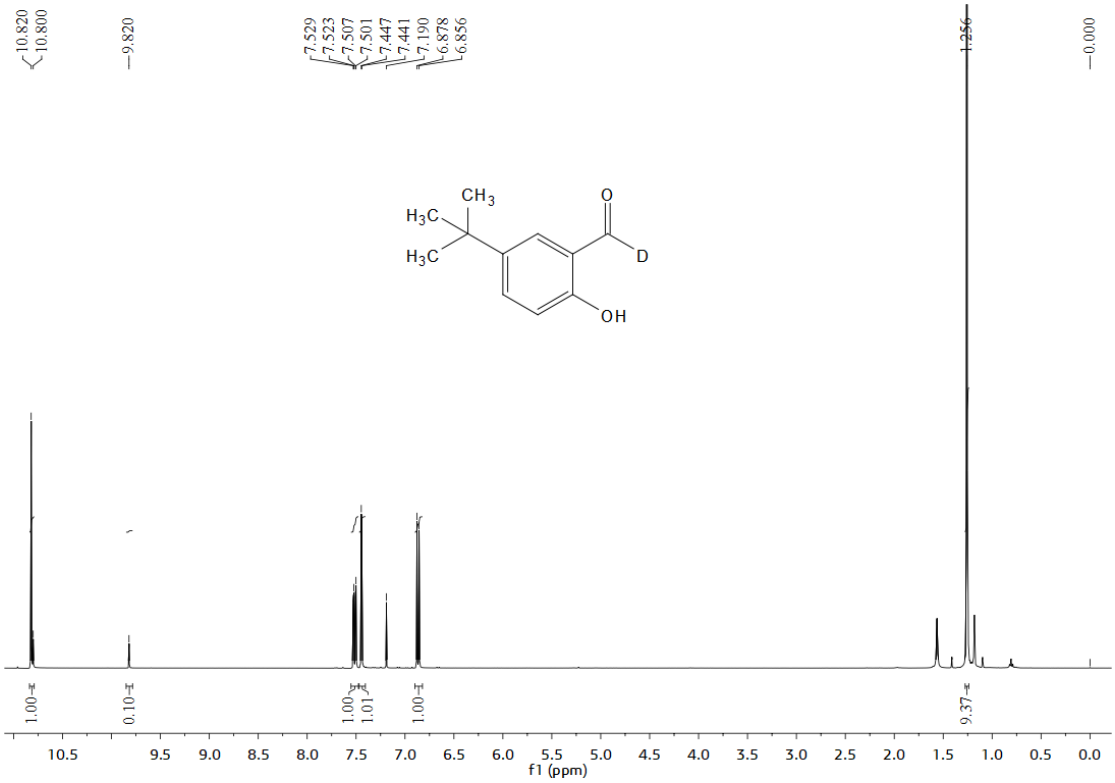
1-Phenylpent-4-yn-1-ol (**2r**) was prepared through the reaction of ethyl 3-oxo-3-phenylpropanoate with propargyl bromide[4] followed by reduction of the resulting 1-phenylpent-4-yn-1-one by $NaBH_4$ according to the literature procedure.[5] The spectrum was in accordance with the previously reported data.[4]

(4) Preparation of 5-*tert*-Butylsalicylaldehyde-*α*-*d*$_1$ (*d*-1c)[6]



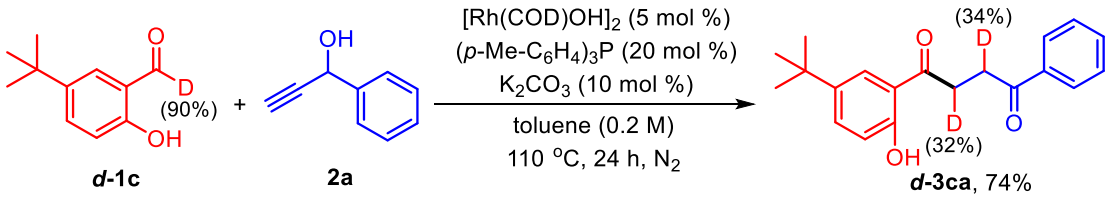
5-*tert*-Butylsalicylaldehyde (2 mmol, 356.4 mg), NHC catalyst (10 mol %, 95.7 mg) and $NaHCO_3$ (2 mmol, 168.0 mg) was dissolved in a mixture of D_2O (4 mL) and anhydrous toluene (1.0 mL) in a sealed tube (15 mL). Then the reaction mixture was vigorously stirred at 80 °C for 18 hours using a Heidolph MR Hei-Tec heating magnetic

stirrer (Heidolph Instruments, Germany). After cooling to room temperature, the reaction mixture was extracted with DCM, dried over anhydrous sodium sulfate, concentrated in vacuo, and purified by column chromatography using *n*-hexane/DCM (50:1) as the eluent to afford ***d*-1c** as a light-yellow oil (280.4 mg, 78%) with 90% D-incorporation.



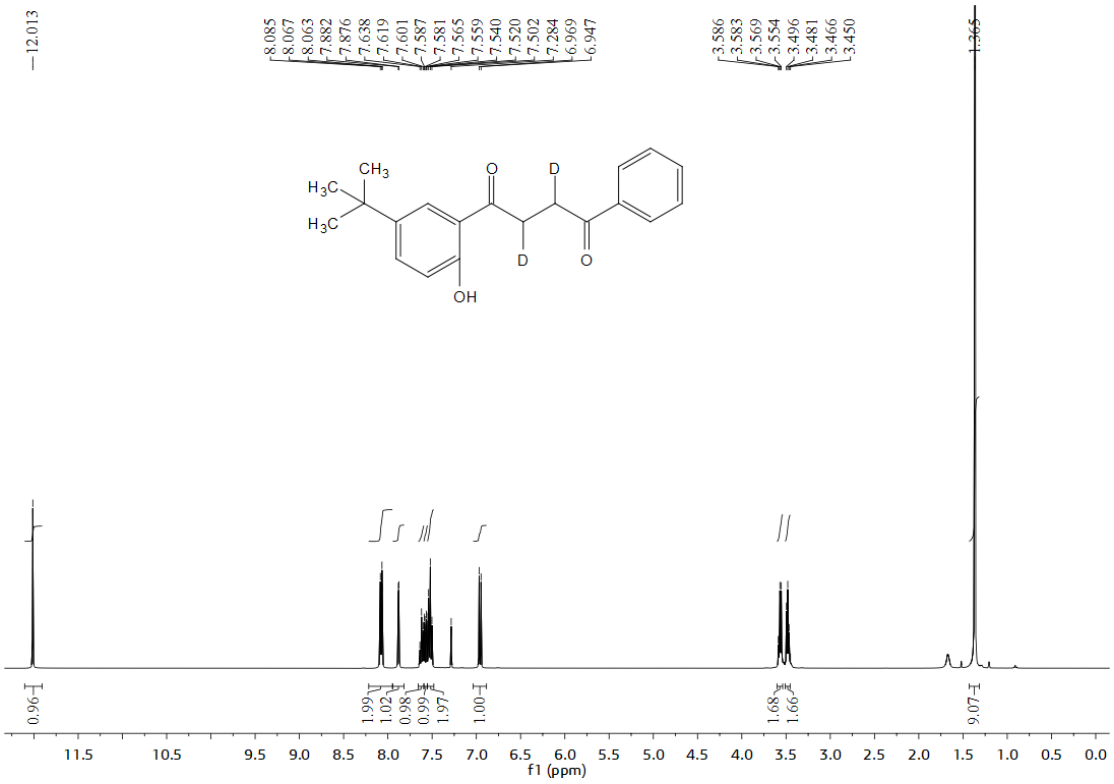
^{1}H NMR (400 MHz, $CDCl_3$) of **5-*tert*-Butylsalicylaldehyde-*α*-*d*$_1$ (*d*-1c)**

(5) Preparation of 1-(5-(*tert*-Butyl)-2-hydroxyphenyl)-4-phenylbutane-1,4-dione-2,3-*d*$_2$ (*d*-3ca)



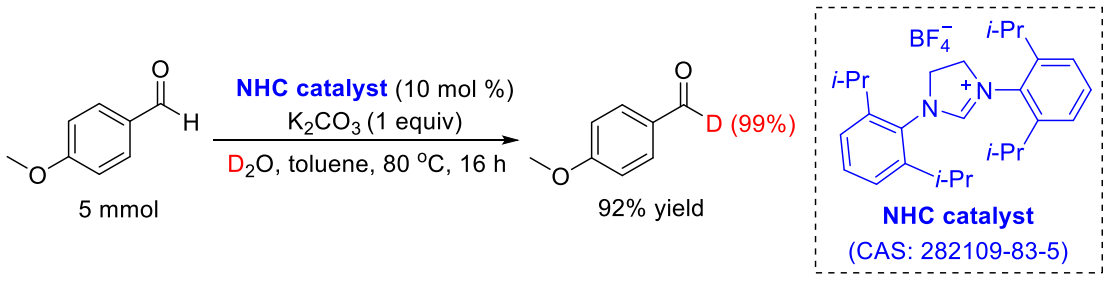
To an oven-dried sealed tube (10 mL) equipped with a stirrer bar in the glove box (filled with N_2) was added $[Rh(COD)OH]_2$ (4.6 mg, 0.01 mmol, 5 mol %), tri(*p*-tolyl)phosphine (12.2 mg, 0.04 mmol, 20 mol %), K_2CO_3 (2.8 mg, 0.02 mmol, 10 mol %), ***d*-1c** (0.2 mmol, 35.8 mg, 1.0 equiv), and 1-phenylprop-2-yn-1-ol **2a** (0.4 mmol, 52.9 mg, 2.0 equiv). Then anhydrous toluene (1.0 mL, 0.2 M) was added. The tube was

sealed and removed from the glove box. The resulting mixture was stirred at room temperature for 15 min, and heated at 110 °C for 24 h using a Heidolph MR Hei-Tec heating magnetic stirrer (Heidolph Instruments, Germany). Upon completion, the reaction mixture was cooled to room temperature and concentrated. The residue was purified by silica gel chromatography (*n*-hexane/EtOAc = 150:1) to afford the desired diketone ***d*-3ca** as a light-yellow solid (45.9 mg, 74%).



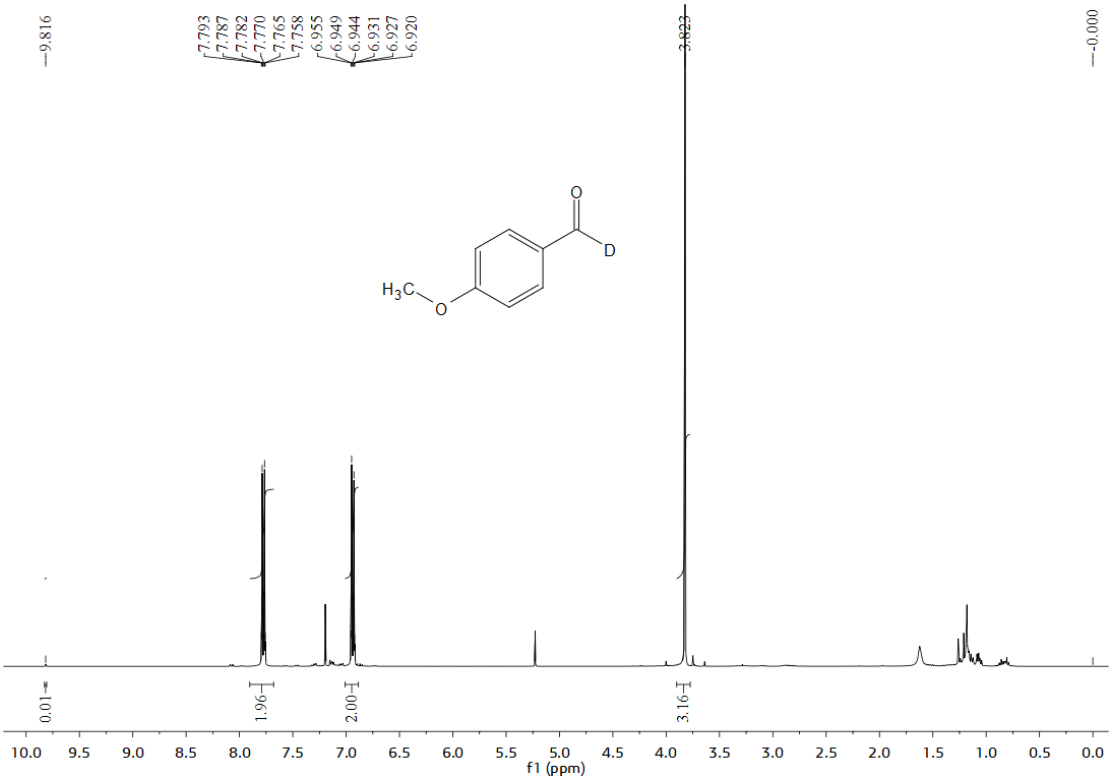
^{1}H NMR (400 MHz, $CDCl_3$) of ***d*-3ca**

(6) Preparation of 4-Methoxybenzaldehyde-*α*-*d*$_1$[6a]



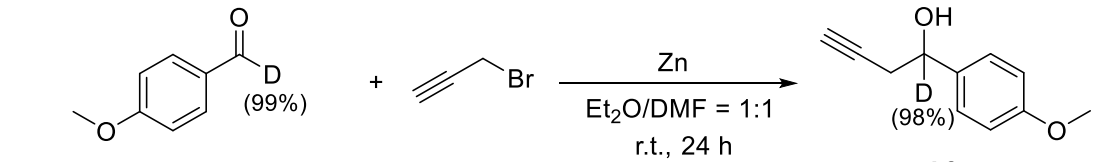
4-Methoxybenzaldehyde (5 mmol, 680.8 mg), NHC catalyst (10 mol %, 239.2 mg) and K_2CO_3 (5 mmol, 691.1 mg) was dissolved in a mixture of D_2O (10 mL) and anhydrous toluene (2.5 mL) in a sealed tube (35 mL). Then the reaction mixture was vigorously stirred at 80 °C for 16 hours using a Heidolph MR Hei-Tec heating magnetic

stirrer (Heidolph Instruments, Germany). After cooling to room temperature, the reaction mixture was extracted with DCM, dried over anhydrous sodium sulfate, concentrated in vacuo, and purified by column chromatography using *n*-hexane/DCM (6:1) as the eluent to afford **4-methoxybenzaldehyde-*α*-*d*$_1$** as a light-yellow oil (628.6 mg, 92%) with 99% D-incorporation. The spectrum was in accordance with the previously reported data.[6a]



^{1}H NMR (400 MHz, $CDCl_3$) of **4-methoxybenzaldehyde-*α*-*d*$_1$**

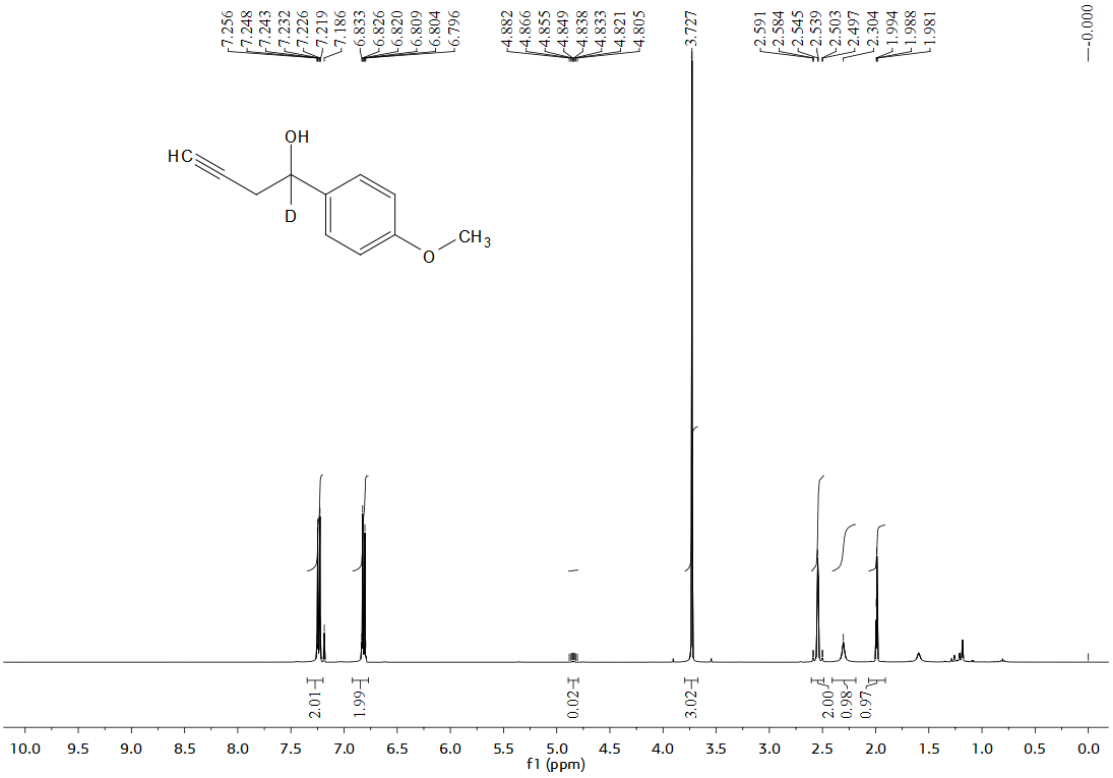
(7) Preparation of 1-(4-Methoxyphenyl)but-3-yn-1-*d*-1-ol (*d*-2s)[7]



4-methoxybenzaldehyde-*α*-*d*$_1$ ***d*-2s**

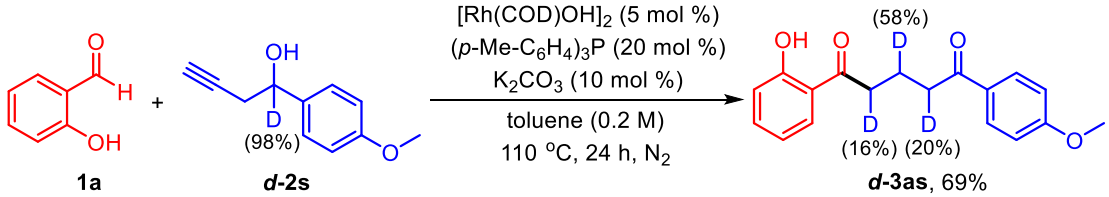
To a 100 mL round-bottom flask was added **4-methoxybenzaldehyde-*α*-*d*$_1$** (548.6 mg, 4 mmol, 1 equiv) in ether/DMF (1:1, 40 mL, tech grade, not anhydrous) and a solution of 80 wt% propargyl bromide in toluene (0.6 mL, 5.2 mmol, 1.3 equiv). The solution was cooled to 0 °C, and an activated zinc powder (0.785 g, 12 mmol, 3 equiv) was then added portion wise over 10 min. (Caution! Very exothermic reaction.) The

reaction was allowed to stir at room temperature for 24 h. Upon completion, the reaction was slowly quenched with saturated ammonium chloride (50 mL) and allowed to stir for another 30 min. The resulting mixture was decanted into a separatory funnel, and the organic layer was separated. The aqueous layer was extracted with ethyl acetate (3 × 25 mL), and the combined organic layers were washed with brine (3 × 30 mL), dried with anhydrous sodium sulfate, and concentrated in vacuo. The resulting crude product was purified via flash column chromatography (*n*-hexane/EtOAc = 50:1-30:1) to give ***d*-2s** as a colorless oil (411.4 mg, 58%) with 98% D-incorporation. The spectra can be compared with the previously reported data of non-deuterated product.[8]



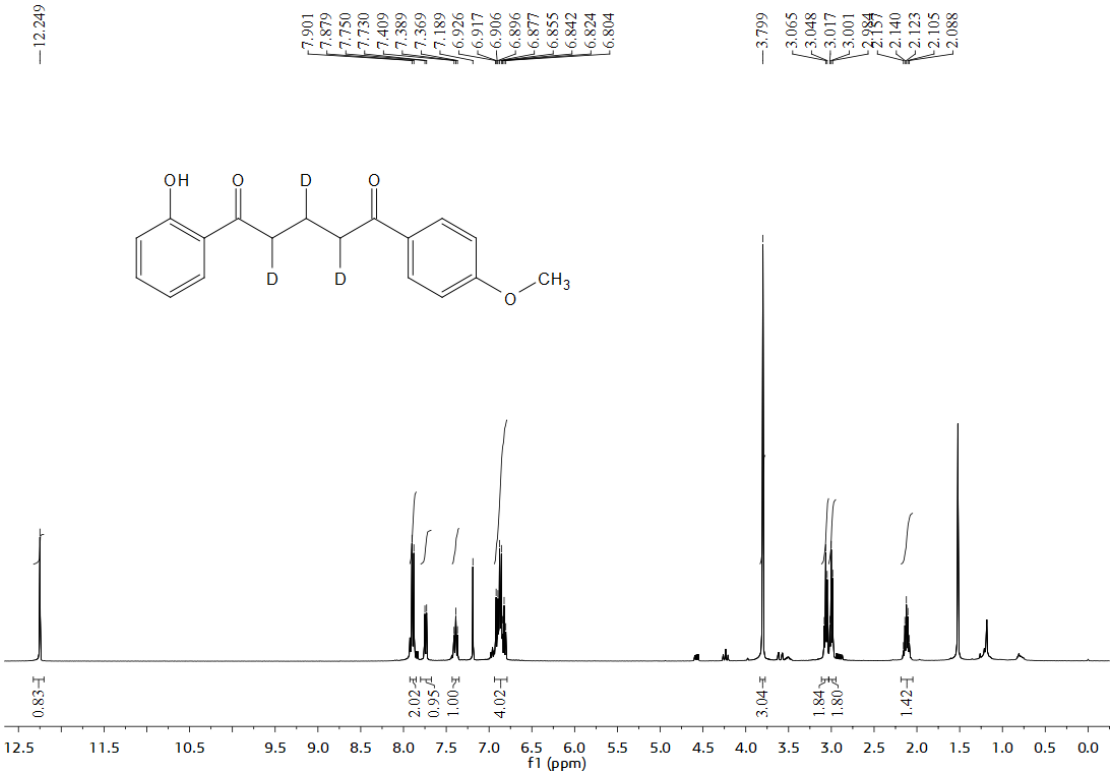
^{1}H NMR (400 MHz, $CDCl_3$) of **1-(4-Methoxyphenyl)but-3-yn-1-*d*-1-ol (*d*-2s)**

(8) Preparation of 1-(2-Hydroxyphenyl)-5-(4-methoxyphenyl)pentane-1,5-dione-2,3,4-*d*$_3$ (*d*-3as)



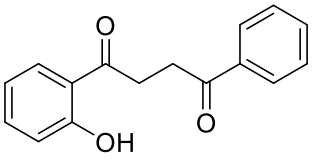
1a **d-2s** ***d*-3as**, 69%

To an oven-dried sealed tube (10 mL) equipped with a stirrer bar in the glove box (filled with N_2) was added $[Rh(COD)OH]_2$ (4.6 mg, 0.01 mmol, 5 mol %), tri(*p*-tolyl)phosphine (12.2 mg, 0.04 mmol, 20 mol %), K_2CO_3 (2.8 mg, 0.02 mmol, 10 mol %), **1a** (0.2 mmol, 24.4 mg, 1.0 equiv), and ***d*-2s** (0.4 mmol, 70.9 mg, 2.0 equiv). Then anhydrous toluene (1.0 mL, 0.2 M) was added. The tube was sealed and removed from the glove box. The resulting mixture was stirred at room temperature for 15 min, and heated at 110 ℃ for 24 h using a Heidolph MR Hei-Tec heating magnetic stirrer (Heidolph Instruments, Germany). Upon completion, the reaction mixture was cooled to room temperature and concentrated. The residue was purified by silica gel chromatography (*n*-hexane/EtOAc = 60:1-30:1) to afford the desired diketone ***d*-3as** as a light-yellow oil (41.3 mg, 69%).



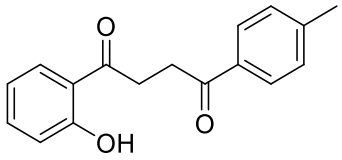
^{1}H NMR (400 MHz, $CDCl_3$) of ***d*-3as**

3. Characterization of Materials



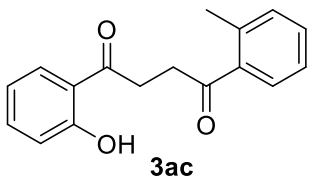
3aa

1-(2-hydroxyphenyl)-4-phenylbutane-1,4-dione[9] **(3aa):** the product was obtained as a white solid after column chromatography (*n*-hexane/EtOAc = 120:1-50:1), 36.1 mg (71%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.13 (s, 1H), 8.03-8.05 (m, 2H), 7.91 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 7.57-7.61 (m, 1H), 7.46-7.51 (m, 3H), 6.98 (dd, *J* = 8.4 Hz, 0.8 Hz, 1H), 6.91-6.95 (m, 1H), 3.49-3.53 (m, 2H), 3.43-3.46 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.7, 198.3, 162.3, 136.6, 136.5, 133.4, 130.0, 128.7, 128.2, 119.4, 119.1, 118.5, 32.22, 32.16; HRMS (ESI-TOF) calcd for $C_{16}H_{15}O_3$ $[M+H]^+$ (255.1021), found 255.1023.



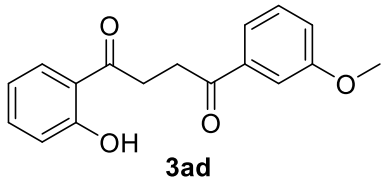
3ab

1-(2-hydroxyphenyl)-4-(*p*-tolyl)butane-1,4-dione[10] **(3ab):** the product was obtained as a light-yellow solid after column chromatography (*n*-hexane/EtOAc = 100:1-80:1), 37.9 mg (68%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.13 (s, 1H), 7.90-7.95 (m, 3H), 7.46-7.50 (m, 1H), 7.29 (d, *J* = 8.0 Hz, 2H), 6.98 (dd, *J* = 8.4 Hz, 0.8 Hz, 1H), 6.91-6.95 (m, 1H), 3.48-3.52 (m, 2H), 3.41-3.44 (m, 2H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.8, 197.9, 162.4, 144.2, 136.4, 134.2, 130.0, 129.4, 128.3, 119.4, 119.1, 118.5, 32.3, 32.1, 21.8; HRMS (ESI-TOF) calcd for $C_{17}H_{17}O_3$ $[M+H]^+$ (269.1178), found 269.1182.



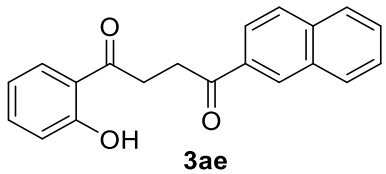
3ac

1-(2-hydroxyphenyl)-4-(*o*-tolyl)butane-1,4-dione (3ac): the product was obtained as a light-yellow oil after column chromatography (*n*-hexane/EtOAc = 120:1-100:1), 37.5 mg (70%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.06 (s, 1H), 7.83 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 7.74 (dd, *J* = 8.0 Hz, 0.8 Hz, 1H), 7.39-7.43 (m, 1H), 7.31-7.35 (m, 1H), 7.19-7.25 (m, 2H), 6.91 (dd, *J* = 8.4 Hz, 0.8 Hz, 1H), 6.84-6.88 (m, 1H), 3.41-3.44 (m, 2H), 3.26-3.29 (m, 2H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.6, 202.2, 162.3, 138.4, 137.6, 136.5, 132.1, 131.6, 130.0, 128.7, 125.8, 119.4, 119.1, 118.5, 34.8, 32.6, 21.4; HRMS (ESI-TOF) calcd for $C_{17}H_{17}O_3$ $[M+H]^+$ (269.1178), found 269.1182.



3ad

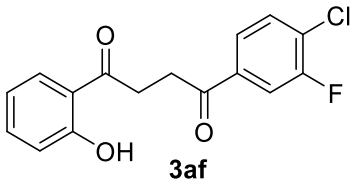
1-(2-hydroxyphenyl)-4-(3-methoxyphenyl)butane-1,4-dione (3ad): the product was obtained as a white solid after column chromatography (*n*-hexane/EtOAc = 60:1), 45.0 mg (79%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.12 (s, 1H), 7.90 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 7.63 (d, *J* = 7.6 Hz, 1H), 7.54-7.55 (m, 1H), 7.46-7.50 (m, 3H), 7.38-7.42 (m, 1H), 7.12-7.15 (m, 1H), 6.98 (dd, *J* = 8.4 Hz, 0.8 Hz, 1H), 6.91-6.95 (m, 1H), 3.86 (s, 3H), 3.48-3.51 (m, 2H), 3.41-3.45 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.6, 198.1, 162.3, 159.9, 138.0, 136.5, 130.0, 129.7, 120.8, 119.9, 119.4, 119.1, 118.5, 112.4, 55.5, 32.34, 32.27; HRMS (ESI-TOF) calcd for $C_{17}H_{17}O_4$ $[M+H]^+$ (285.1127), found 285.1129.



3ae

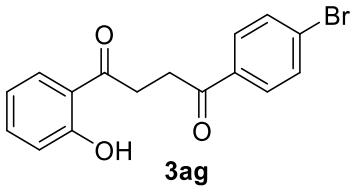
1-(2-hydroxyphenyl)-4-(naphthalen-2-yl)butane-1,4-dione (3ae): the product was obtained as a light-yellow solid after column chromatography (*n*-hexane/EtOAc = 150:1-80:1), 48.1 mg (79%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.07 (s, 1H), 8.50 (s, 1H), 8.00 (dd, *J* = 8.4 Hz, 1.6 Hz, 1H), 7.91 (d, *J* = 8.0 Hz, 1H), 7.80-7.87 (m, 3H), 7.47-

7.56 (m, 2H), 7.39-7.44 (m, 1H), 6.92 (dd, J = 8.4 Hz, 1.2 Hz, 1H), 6.85-6.89 (m, 1H), 3.46-3.53 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.8, 198.3, 162.4, 136.5, 135.8, 134.0, 132.6, 130.04, 129.95, 129.7, 128.64, 128.61, 127.9, 126.9, 123.9, 119.4, 119.1, 118.6, 32.4, 32.3; HRMS (ESI-TOF) calcd for $C_{20}H_{17}O_3$ $[M+H]^+$ (305.1178), found 305.1179.



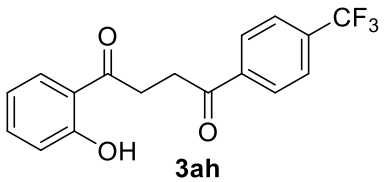
3af

1-(4-chloro-3-fluorophenyl)-4-(2-hydroxyphenyl)butane-1,4-dione (3af): the product was obtained as a white solid after column chromatography (*n*-hexane/EtOAc = 150:1-100:1), 47.6 mg (78%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.03 (s, 1H), 7.89 (dd, J = 8.0 Hz, 1.6 Hz, 1H), 7.77-7.81 (m, 2H), 7.47-7.56 (m, 2H), 6.99 (dd, J = 8.4 Hz, 0.8 Hz, 1H), 6.92-6.96 (m, 1H), 3.52 (t, J = 6.8 Hz, 2H), 3.38 (t, J = 6.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.2, 196.2, 162.3, 158.3 (d, J = 249.5 Hz), 136.9 (d, J = 5.2 Hz), 136.6, 131.1, 129.9, 126.8 (d, J = 17.7 Hz), 124.6 (d, J = 3.7 Hz), 119.3, 119.2, 118.6, 116.1 (d, J = 21.8 Hz), 32.22, 32.16; HRMS (ESI-TOF) calcd for $C_{16}H_{13}ClFO_3$ $[M+H]^+$ (307.0537), found 307.0540.



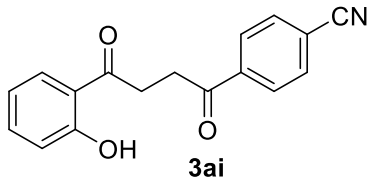
3ag

1-(4-bromophenyl)-4-(2-hydroxyphenyl)butane-1,4-dione[10] **(3ag):** the product was obtained as a white solid after column chromatography (*n*-hexane/EtOAc = 150:1-120:1), 44.8 mg (67%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.07 (s, 1H), 7.89-7.91 (m, 3H), 7.63 (d, J = 8.0 Hz, 2H), 7.46-7.50 (m, 1H), 6.98 (d, J = 8.4 Hz, 1H), 6.92-6.95 (m, 1H), 3.51 (t, J = 6.4 Hz, 2H), 3.38 (t, J = 6.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.4, 197.3, 162.3, 136.6, 135.4, 132.1, 130.0, 129.7, 128.6, 119.3, 119.1, 118.6, 32.2, 32.1; HRMS (ESI-TOF) calcd for $C_{16}H_{14}BrO_3$ $[M+H]^+$ (333.0126), found 333.0125.



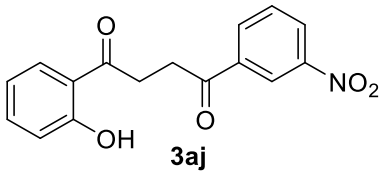
3ah

1-(2-hydroxyphenyl)-4-(4-(trifluoromethyl)phenyl)butane-1,4-dione (3ah): the product was obtained as a white solid after column chromatography (*n*-hexane/EtOAc = 100:1-80:1), 45.7 mg (71%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.04 (s, 1H), 8.14 (d, *J* = 8.0 Hz, 2H), 7.90 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 7.76 (d, *J* = 8.0 Hz, 2H), 7.47-7.51 (m, 1H), 6.99 (dd, *J* = 8.4 Hz, 0.8 Hz, 1H), 6.92-6.96 (m, 1H), 3.53-3.56 (m, 2H), 3.44-3.47 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.2, 197.5, 162.4, 139.3, 136.6, 134.7 (q, *J* = 32.5 Hz), 129.9, 128.5, 125.8 (q, *J* = 3.7 Hz), 123.7 (q, *J* = 271.0 Hz), 119.3, 119.2, 118.6, 32.4, 32.2; HRMS (ESI-TOF) calcd for $C_{17}H_{14}F_3O_3$ $[M+H]^+$ (323.0895), found 323.0897.



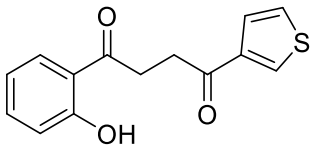
3ai

4-(4-(2-hydroxyphenyl)-4-oxobutanoyl)benzonitrile (3ai): the product was obtained as a white solid after column chromatography (*n*-hexane/EtOAc = 50:1-10:1), 32.5 mg (58%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.00 (s, 1H), 8.12 (dd, *J* = 6.8 Hz, 1.6 Hz, 2H), 7.88 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 7.80 (dd, *J* = 6.8 Hz, 1.6 Hz, 2H), 7.47-7.51 (m, 1H), 6.98 (dd, *J* = 8.4 Hz, 0.8 Hz, 1H), 6.92-6.96 (m, 1H), 3.52-3.55 (m, 2H), 3.41-3.45 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.0, 197.2, 162.3, 139.6, 136.7, 132.6, 129.9, 128.6, 119.20, 119.19, 118.6, 118.0, 116.6, 32.4, 32.2; HRMS (ESI-TOF) calcd for $C_{17}H_{14}NO_3$ $[M+H]^+$ (280.0974), found 280.0979.



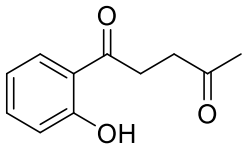
3aj

1-(2-hydroxyphenyl)-4-(3-nitrophenyl)butane-1,4-dione (3aj): the product was obtained as a white solid after column chromatography (*n*-hexane/EtOAc = 30:1-15:1), 14.3 mg (24%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.02 (s, 1H), 8.89-8.90 (m, 1H), 8.49 (ddd, *J* = 8.0 Hz, 2.4 Hz, 1.2 Hz, 1H), 8.38-8.41 (m, 1H), 7.93 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 7.73-7.77 (m, 1H), 7.51-7.55 (m, 1H), 7.02 (dd, *J* = 8.4 Hz, 0.8 Hz, 1H), 6.96-7.00 (m, 1H), 3.59-3.62 (m, 2H), 3.49-3.52 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.0, 196.3, 162.4, 148.6, 138.0, 136.7, 133.8, 130.1, 129.9, 127.7, 123.2, 119.3, 119.2, 118.7, 32.4, 32.3; HRMS (ESI-TOF) calcd for $C_{16}H_{14}NO_5$ $[M+H]^+$ (300.0872), found 300.0873.



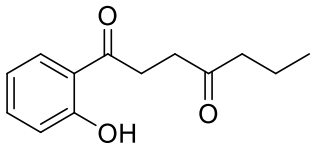
3al

1-(2-hydroxyphenyl)-4-(thiophen-3-yl)butane-1,4-dione (3al): the product was obtained as a white solid after column chromatography (*n*-hexane/EtOAc = 120:1-50:1), 35.4 mg (68%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.11 (s, 1H), 8.16 (dd, *J* = 2.8 Hz, 1.2 Hz, 1H), 7.89 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 7.59 (dd, *J* = 5.2 Hz, 1.2 Hz, 1H), 7.46-7.50 (m, 1H), 7.34 (dd, *J* = 5.2 Hz, 2.8 Hz, 1H), 6.98 (dd, *J* = 8.0 Hz, 0.8 Hz, 1H), 6.91-6.95 (m, 1H), 3.49 (t, *J* = 6.4 Hz, 2H), 3.36 (t, *J* = 6.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.6, 192.6, 162.3, 141.9, 136.5, 132.3, 130.0, 126.9, 126.6, 119.4, 119.1, 118.5, 33.3, 32.1; HRMS (ESI-TOF) calcd for $C_{14}H_{13}O_3S$ $[M+H]^+$ (261.0585), found 261.0588.



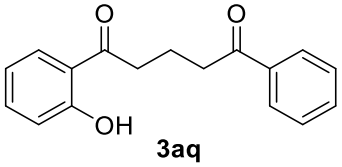
3am

1-(2-hydroxyphenyl)pentane-1,4-dione[10] **(3am):** the product was obtained as a colorless oil after column chromatography (*n*-hexane/EtOAc = 150:1-80:1), 15.0 mg (39%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.01 (s, 1H), 7.75 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 7.38-7.42 (m, 1H), 6.90 (dd, *J* = 8.4 Hz, 0.8 Hz, 1H), 6.82-6.86 (m, 1H), 3.26 (t, *J* = 6.0 Hz, 2H), 2.81 (t, *J* = 6.0 Hz, 2H), 2.20 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.0, 204.5, 162.3, 136.5, 129.9, 119.3, 119.1, 118.5, 36.7, 32.1, 30.1; HRMS (ESI-TOF) calcd for $C_{11}H_{13}O_3$ $[M+H]^+$ (193.0865), found 193.0866.



3an

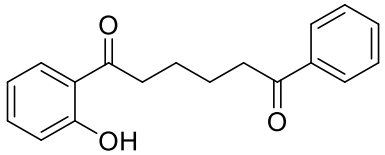
1-(2-hydroxyphenyl)heptane-1,4-dione (3an): the product was obtained as a colorless oil after column chromatography (*n*-hexane/EtOAc = 150:1-120:1), 15.8 mg (36%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.03 (s, 1H), 7.76 (dd, *J* = 8.4 Hz, 1.6 Hz, 1H), 7.38-7.42 (m, 1H), 6.90 (dd, *J* = 8.4 Hz, 0.8 Hz, 1H), 6.82-6.86 (m, 1H), 3.26 (t, *J* = 6.4 Hz, 2H), 2.78 (t, *J* = 6.4 Hz, 2H), 2.45 (t, *J* = 7.2 Hz, 2H), 1.55-1.64 (m, 2H), 0.88 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.4, 204.7, 162.3, 136.4, 130.0, 119.3, 119.1, 118.5, 44.9, 35.9, 32.1, 17.4, 13.8; HRMS (ESI-TOF) calcd for $C_{13}H_{17}O_3$ $[M+H]^+$ (221.1178), found 221.1180.



3aq

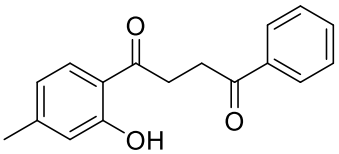
1-(2-hydroxyphenyl)-5-phenylpentane-1,5-dione (3aq): the product was obtained as a white solid after column chromatography (*n*-hexane/EtOAc = 100:1-50:1), 39.0 mg

(73%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.25 (s, 1H), 7.90-7.92 (m, 2H), 7.74 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 7.48-7.52 (m, 1H), 7.37-7.42 (m, 3H), 6.91 (dd, *J* = 8.4 Hz, 0.8 Hz, 1H), 6.81-6.85 (m, 1H), 3.05-3.10 (m, 4H), 2.11-2.18 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.3, 199.7, 162.5, 136.9, 136.5, 133.3, 130.1, 128.7, 128.1, 119.4, 119.1, 118.6, 37.49, 37.45, 18.8; HRMS (ESI-TOF) calcd for $C_{17}H_{17}O_3$ $[M+H]^+$ (269.1178), found 269.1182.



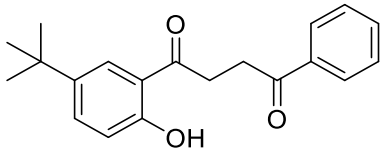
3ar

1-(2-hydroxyphenyl)-6-phenylhexane-1,6-dione (3ar): the product was obtained as a white solid after column chromatography (*n*-hexane/EtOAc = 150:1-100:1), 27.7 mg (49%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.25 (s, 1H), 7.87-7.90 (m, 2H), 7.69 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 7.47-7.51 (m, 1H), 7.37-7.41 (m, 3H), 6.91 (dd, *J* = 8.4 Hz, 0.8 Hz, 1H), 6.80-6.84 (m, 1H), 2.95-3.01 (m, 4H), 1.74-1.82 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.4, 199.9, 162.6, 137.0, 136.4, 133.1, 130.0, 128.7, 128.1, 119.4, 119.0, 118.6, 38.4, 38.2, 24.0, 23.9; HRMS (ESI-TOF) calcd for $C_{18}H_{19}O_3$ $[M+H]^+$ (283.1334), found 283.1337.



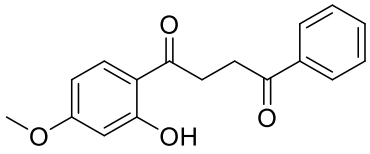
3ba

1-(2-hydroxy-4-methylphenyl)-4-phenylbutane-1,4-dione (3ba): the product was obtained as a white solid after column chromatography (*n*-hexane/EtOAc = 100:1-80:1), 39.1 mg (73%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.06 (s, 1H), 7.94-7.97 (m, 2H), 7.70 (d, *J* = 8.0 Hz, 1H), 7.49-7.53 (m, 1H), 7.39-7.43 (m, 2H), 6.71 (s, 1H), 6.66 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 3.33-3.41 (m, 4H), 2.27 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.0, 198.4, 162.5, 148.1, 136.7, 133.3, 129.9, 128.7, 128.2, 120.4, 118.5, 117.2, 32.3, 32.1, 22.0; HRMS (ESI-TOF) calcd for $C_{17}H_{17}O_3$ $[M+H]^+$ (269.1178), found 269.1182.



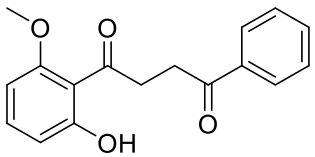
3ca

1-(5-(*tert*-butyl)-2-hydroxyphenyl)-4-phenylbutane-1,4-dione (3ca): the product was obtained as a light-yellow solid after column chromatography (*n*-hexane/EtOAc = 150:1-80:1), 50.8 mg (82%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.99 (s, 1H), 8.04-8.07 (m, 2H), 7.85 (d, *J* = 2.4 Hz, 1H), 7.58-7.62 (m, 1H), 7.55 (dd, *J* = 8.8 Hz, 2.4 Hz, 1H), 7.48-7.52 (m, 2H), 6.93 (d, *J* = 8.8 Hz, 1H), 3.53-3.56 (m, 2H), 3.44-3.47 (m, 2H), 1.34 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.6, 198.5, 160.2, 141.8, 136.7, 134.3, 133.4, 128.8, 128.2, 125.7, 118.7, 118.1, 34.2, 32.3, 32.2, 31.4; HRMS (ESI-TOF) calcd for $C_{20}H_{23}O_3$ $[M+H]^+$ (311.1647), found 311.1651.



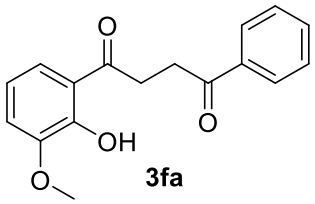
3da

1-(2-hydroxy-4-methoxyphenyl)-4-phenylbutane-1,4-dione[10] **(3da):** the product was obtained as a white solid after column chromatography (*n*-hexane/EtOAc = 80:1-50:1), 48.0 mg (84%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.59 (s, 1H), 8.02-8.04 (m, 2H), 7.80 (d, *J* = 8.8 Hz, 1H), 7.57-7.60 (m, 1H), 7.47-7.50 (m, 2H), 6.47 (dd, *J* = 8.8 Hz, 2.4 Hz, 1H), 6.42 (d, *J* =2.4 Hz, 1H), 3.84 (s, 3H), 3.39-3.46 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.7, 198.5, 166.1, 165.2, 136.7, 133.3, 131.6, 128.7, 128.2, 113.5, 107.8, 101.0, 55.6, 32.3, 31.8; HRMS (ESI-TOF) calcd for $C_{17}H_{17}O_4$ $[M+H]^+$ (285.1127), found 285.1129.



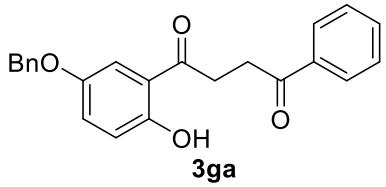
3ea

1-(2-hydroxy-6-methoxyphenyl)-4-phenylbutane-1,4-dione (3ea): the product was obtained as a light-yellow oil after column chromatography (*n*-hexane/EtOAc = 80:1-50:1), 27.8 mg (49%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.02 (s, 1H), 7.96-7.99 (m, 2H), 7.49-7.53 (m, 1H), 7.40-7.44 (m, 2H), 7.26-7.30 (m, 1H), 6.50 (dd, *J* = 8.0 Hz, 0.8 Hz, 1H), 6.34 (dd, *J* = 8.0 Hz, 0.8 Hz, 1H), 3.86 (s, 3H), 3.50 (t, *J* = 6.4 Hz, 2H), 3.30 (t, *J* = 6.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.6, 199.1, 164.7, 161.6, 137.0, 136.1, 133.2, 128.7, 128.2, 111.12, 110.88, 101.3, 55.8, 39.1, 32.7; HRMS (ESI-TOF) calcd for $C_{17}H_{17}O_4$ $[M+H]^+$ (285.1127), found 285.1129.



3fa

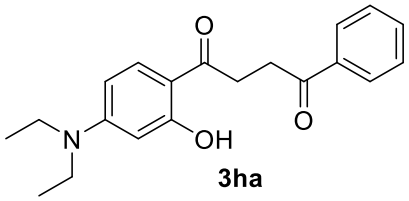
1-(2-hydroxy-3-methoxyphenyl)-4-phenylbutane-1,4-dione (3fa): the product was obtained as a light-yellow solid after column chromatography (*n*-hexane/EtOAc = 50:1-25:1), 23.5 mg (41%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.35 (s, 1H); 7.96-7.99 (m, 2H), 7.50-7.55 (m, 1H), 7.40-7.46 (m, 3H), 7.00 (d, *J* = 8.0 Hz, 1H), 6.80-6.84 (m, 1H), 3.84 (s, 3H), 3.44-3.47 (m, 2H), 3.37-3.40 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.1, 198.3, 152.8, 149.0, 136.6, 133.4, 128.8, 128.2, 121.2, 119.4, 118.5, 117.0, 56.3, 32.7, 32.2; HRMS (ESI-TOF) calcd for $C_{17}H_{17}O_4$ $[M+H]^+$ (285.1127), found 285.1129.



3ga

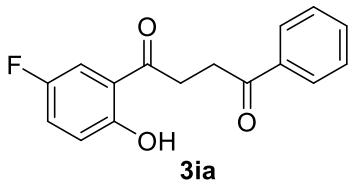
1-(5-(benzyloxy)-2-hydroxyphenyl)-4-phenylbutane-1,4-dione (3ga): the product was obtained as a light-yellow solid after column chromatography (*n*-hexane/EtOAc = 100:1-50:1), 43.2 mg (60%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.75 (s, 1H), 8.03-8.05 (m, 2H), 7.58-7.62 (m, 1H), 7.38-7.52 (m, 7H), 7.32-7.36 (m, 1H), 7.19 (dd, *J* = 9.2 Hz, 2.8 Hz, 1H), 6.93 (d, *J* = 9.2 Hz, 1H), 5.07 (s, 2H), 3.41-3.47 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.2, 198.3, 156.9, 151.0, 136.8, 136.6, 133.4, 128.8, 128.23, 128.19,

127.7, 125.3, 119.4, 118.9, 114.3, 71.2, 32.4, 32.2; HRMS (ESI-TOF) calcd for $C_{23}H_{21}O_4$ $[M+H]^+$ (361.1440), found 361.1444.



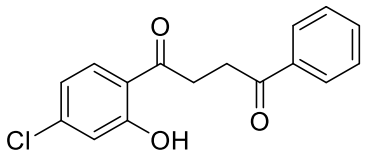
3ha

1-(4-(diethylamino)-2-hydroxyphenyl)-4-phenylbutane-1,4-dione (3ha): the product was obtained as a light-yellow solid after column chromatography (*n*-hexane/EtOAc = 70:1-30:1), 41.4 mg (64%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.70 (s, 1H), 7.96-7.98 (m, 2H), 7.60 (d, *J* = 9.2 Hz, 1H), 7.48-7.53 (m, 1H), 7.39-7.43 (m, 2H), 6.15 (dd, *J* = 9.2 Hz, 2.4 Hz, 1H), 6.00 (d, *J* = 2.4 Hz, 1H), 3.26-3.37 (m, 8H), 1.13 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.5, 199.0, 165.1, 153.8, 136.8, 133.2, 131.9, 128.7, 128.2, 109.4, 103.8, 97.2, 44.7, 32.8, 31.1, 12.7; HRMS (ESI-TOF) calcd for $C_{20}H_{24}NO_3$ $[M+H]^+$ (326.1756), found 326.1758.



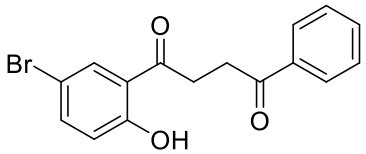
3ia

1-(5-fluoro-2-hydroxyphenyl)-4-phenylbutane-1,4-dione (3ia): the product was obtained as a white solid after column chromatography (*n*-hexane/EtOAc = 100:1-80:1), 34.0 mg (62%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.84 (s, 1H), 8.02-8.05 (m, 2H), 7.56-7.62 (m, 2H), 7.48-7.51 (m, 2H), 7.20-7.25 (m, 1H), 6.95 (dd, *J* = 9.2 Hz, 4.4 Hz, 1H), 3.42-3.48 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.8 (d, *J* = 2.7 Hz), 198.1, 158.5(d, *J* = 1.1 Hz), 154.9 (d, *J* = 237.4 Hz), 136.5, 133.5, 128.8, 128.2, 124.1 (d, *J* = 23.6 Hz), 119.9 (d, *J* = 7.3 Hz), 118.8 (d, *J* = 6.0 Hz), 114.9 (d, *J* = 23.1 Hz), 32.3, 32.1; HRMS (ESI-TOF) calcd for $C_{16}H_{14}FO_3$ $[M+H]^+$ (273.0927), found 273.0931.



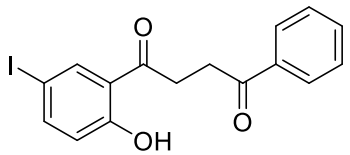
3ja

1-(4-chloro-2-hydroxyphenyl)-4-phenylbutane-1,4-dione[10] **(3ja):** the product was obtained as a white solid after column chromatography (*n*-hexane/EtOAc = 100:1-80:1), 43.5 mg (75%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.25 (s, 1H), 8.01-8.04 (m, 2H), 7.83 (d, *J* = 8.4 Hz, 1H), 7.57-7.62 (m, 1H), 7.47-7.51 (m, 2H), 7.00 (d, *J* = 2.0 Hz, 1H), 6.91 (dd, *J* = 8.8 Hz, 2.0 Hz, 1H), 3.42-3.48 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.0, 198.2, 163.0, 142.2, 136.5, 133.5, 131.0, 128.8, 128.2, 119.8, 118.6, 118.0, 32.3, 32.1; HRMS (ESI-TOF) calcd for $C_{16}H_{14}ClO_3$ $[M+H]^+$ (289.0631), found 289.0637.



3ka

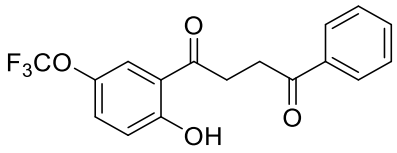
1-(5-bromo-2-hydroxyphenyl)-4-phenylbutane-1,4-dione (3ka): the product was obtained as a light-yellow solid after column chromatography (*n*-hexane/EtOAc = 100:1), 31.0 mg (47%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.02 (s, 1H), 8.01-8.04 (m, 3H), 7.48-7.62 (m, 4H), 6.89 (d, *J* = 8.8 Hz, 1H), 3.40-3.49 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.8, 198.0, 161.2, 139.1, 136.5, 133.5, 132.3, 128.8, 128.2, 120.62, 120.58, 110.7, 32.3, 32.1; HRMS (ESI-TOF) calcd for $C_{16}H_{14}BrO_3$ $[M+H]^+$ (333.0126), found 333.0125.



3la

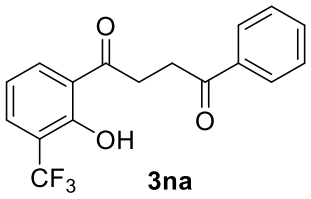
1-(2-hydroxy-5-iodophenyl)-4-phenylbutane-1,4-dione (3la): the product was obtained as a white solid after column chromatography (*n*-hexane/EtOAc = 100:1), 37.0 mg (49%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.05 (s, 1H), 8.19 (d, *J* = 2.0 Hz, 1H), 8.03

(d, J = 7.6 Hz, 1H), 7.72 (dd, J = 8.8 Hz, 1.6 Hz, 1H), 7.58-7.62 (m, 1H), 7.48-7.52 (m, 2H), 6.78 (d, J = 8.4 Hz, 1H), 3.43-3.52 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.6, 198.0, 161.8, 144.7, 138.4, 136.5, 133.4, 128.7, 128.2, 121.4, 121.0, 79.9, 32.3, 32.0; HRMS (ESI-TOF) calcd for $C_{16}H_{14}IO_3$ $[M+H]^+$ (380.9988), found 380.9992.



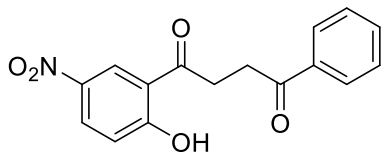
3ma

1-(2-hydroxy-5-(trifluoromethoxy)phenyl)-4-phenylbutane-1,4-dione (3ma): the product was obtained as a light-yellow solid after column chromatography (*n*-hexane/EtOAc = 100:1-80:1), 31.5 mg (47%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.05 (s, 1H), 8.03-8.05 (m, 2H), 7.75 (d, J = 2.8 Hz, 1H), 7.59-7.63 (m, 1H), 7.48-7.52 (m, 2H), 7.37 (dd, J = 9.2 Hz, 2.4 Hz, 1H), 7.01 (d, J = 9.2 Hz, 1H), 3.43-3.52 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.9, 198.0, 160.9, 140.6 (q, J = 1.9 Hz), 136.5, 133.5, 130.0, 128.8, 128.2, 122.5, 120.6 (q, J = 255.5 Hz), 120.0, 119.1, 32.3, 32.1; HRMS (ESI-TOF) calcd for $C_{17}H_{14}F_3O_4$ $[M+H]^+$ (339.0844), found 339.0843.



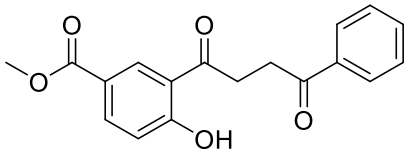
3na

1-(2-hydroxy-3-(trifluoromethyl)phenyl)-4-phenylbutane-1,4-dione (3na): the product was obtained as a white solid after column chromatography (*n*-hexane/EtOAc = 60:1-40:1), 55.2 mg (86%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.91 (s, 1H), 8.12 (dd, J = 8.4 Hz, 1.2 Hz, 1H), 8.02-8.04 (m, 2H), 7.79 (d, J = 7.6 Hz, 1H), 7.58-7.62 (m, 1H), 7.48-7.51 (m, 2H), 7.00-7.04 (m, 1H), 3.46-3.53 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.8, 198.0, 160.6 (q, J = 1.3 Hz), 136.4, 133.8, 133.6 (q, J = 4.9 Hz), 133.5, 128.8, 128.2, 123.2 (q, J = 271.0 Hz), 120.1, 119.3 (q, J = 31.2 Hz), 118.2, 32.4, 32.1; HRMS (ESI-TOF) calcd for $C_{17}H_{14}F_3O_3$ $[M+H]^+$ (323.0895), found 323.0897.



3oa

1-(2-hydroxy-5-nitrophenyl)-4-phenylbutane-1,4-dione (3oa): the product was obtained as a yellow solid after column chromatography (*n*-hexane/EtOAc = 40:1-25:1), 48.5 mg (81%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.67 (s, 1H), 8.83 (d, *J* = 2.8 Hz, 1H), 8.30 (dd, *J* = 9.2 Hz, 2.8 Hz, 1H), 7.95-7.97 (m, 2H), 7.52-7.57 (m, 1H), 7.42-7.46 (m, 2H), 7.02 (d, *J* = 9.2 Hz, 1H), 3.48-3.51 (m, 2H), 3.43-3.46 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.4, 197.7, 167.1, 139.7, 136.3, 133.6, 131.1, 128.8, 128.2, 126.6, 119.7, 118.3, 32.3, 32.2; HRMS (ESI-TOF) calcd for $C_{16}H_{14}NO_5$ $[M+H]^+$ (300.0872), found 300.0873.



3pa

methyl 4-hydroxy-3-(4-oxo-4-phenylbutanoyl)benzoate (3pa): the product was obtained as a white solid after column chromatography (*n*-hexane/EtOAc = 30:1-25:1), 57.3 mg (92%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.53 (s, 1H), 8.64 (d, *J* = 1.2 Hz, 1H), 8.13 (dd, *J* = 8.8 Hz, 1.6 Hz, 1H), 8.02 (d, *J* = 7.6 Hz, 2H), 7.57-7.60 (m, 1H), 7.46-7.50 (m, 2H), 7.00 (d, *J* = 8.8 Hz, 1H), 3.92 (s, 3H), 3.56 (t, *J* = 6.4 Hz, 2H), 3.46 (t, *J* = 6.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.7, 198.0, 166.0, 165.9, 137.2, 136.5, 133.4, 132.5, 128.7, 128.1, 121.2, 118.8, 118.7, 52.2, 32.3, 32.1; HRMS (ESI-TOF) calcd for $C_{18}H_{17}O_5$ $[M+H]^+$ (313.1076), found 313.1080.

4. References

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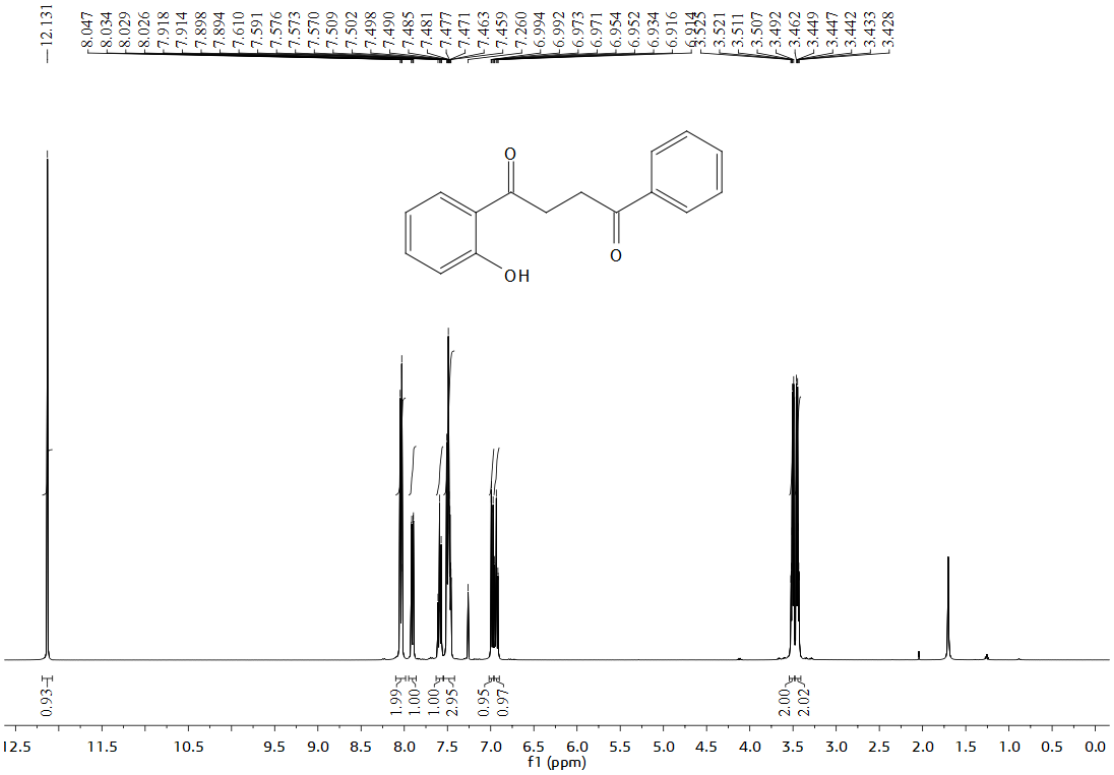
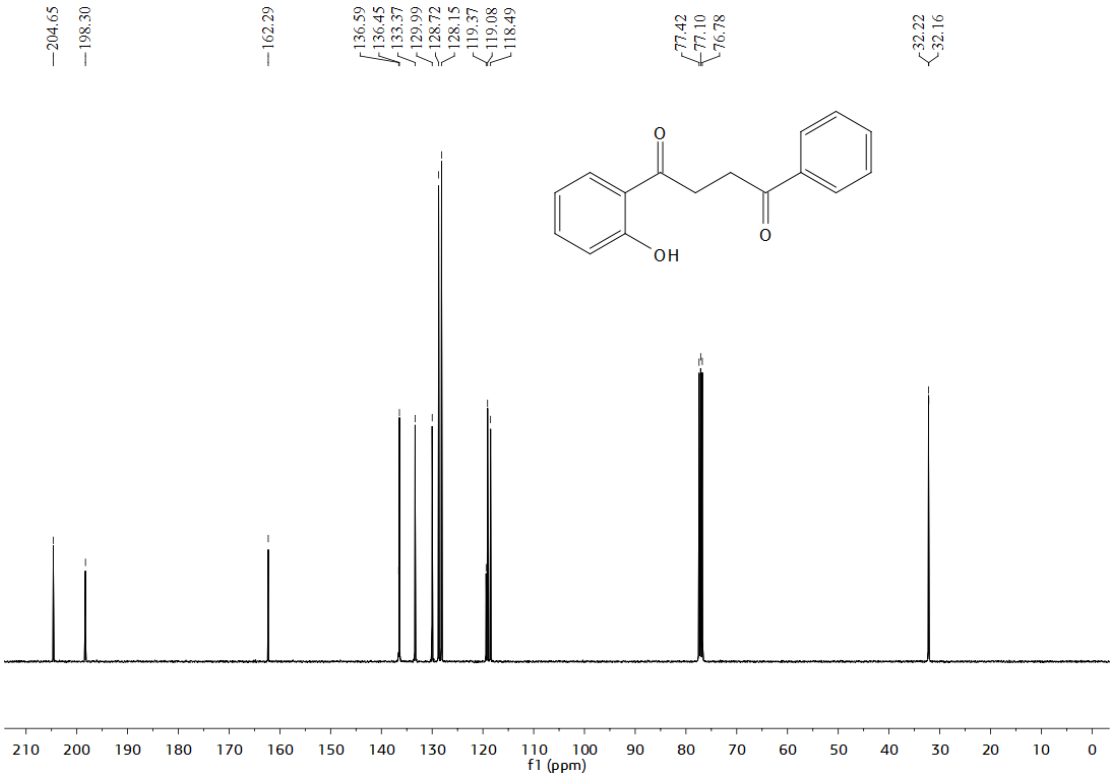
5. Copies of NMR Spectra

1-(2-hydroxyphenyl)-4-phenylbutane-1,4-dione (3aa):

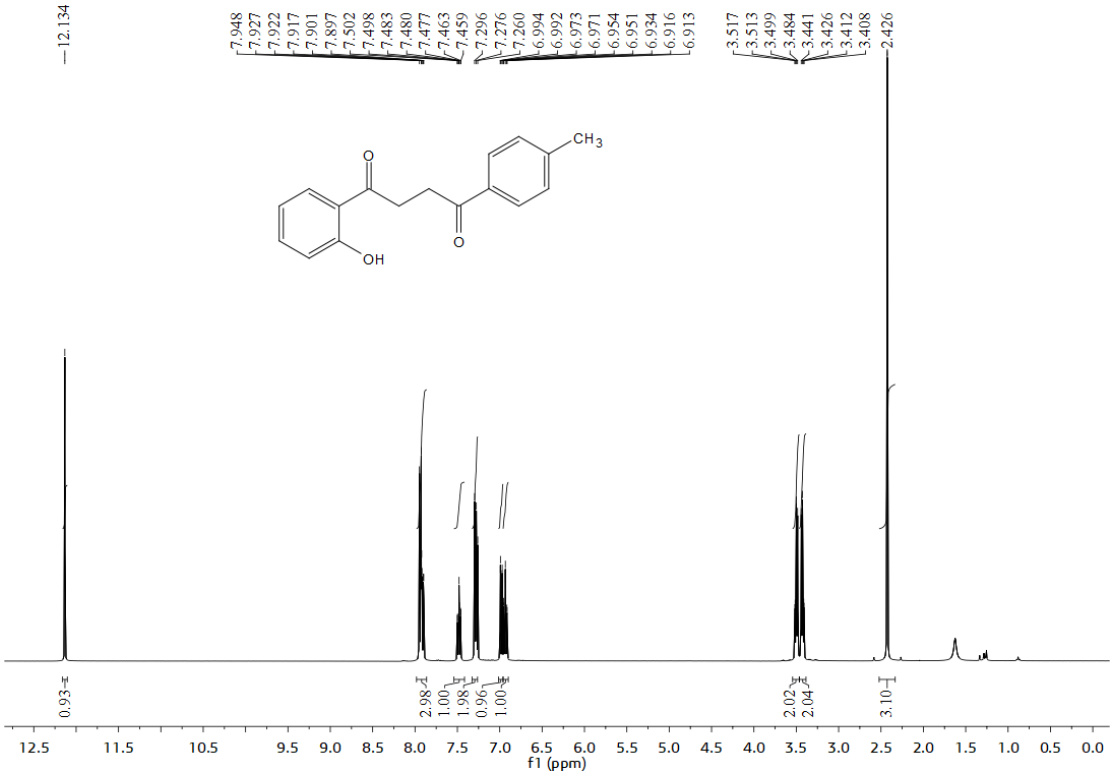


^{1}H NMR (400 MHz, $CDCl_3$) of **3aa**

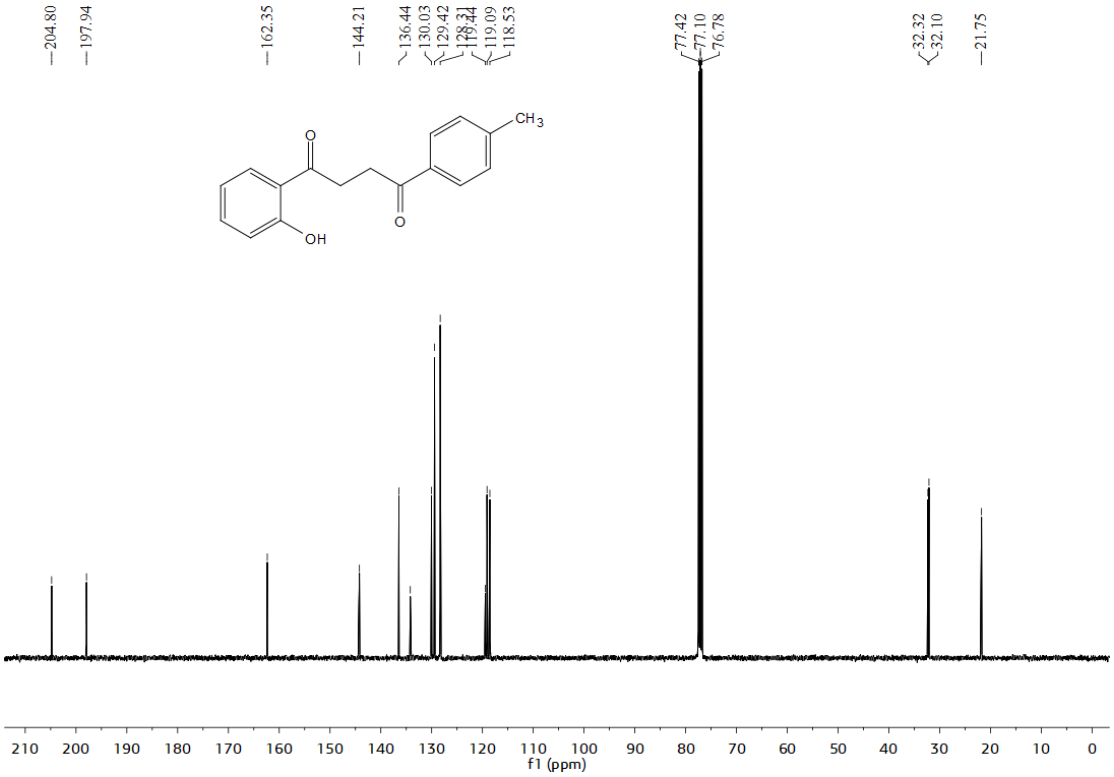


^{13}C NMR (100 MHz, $CDCl_3$) of **3aa**

1-(2-hydroxyphenyl)-4-(*p*-tolyl)butane-1,4-dione (3ab):

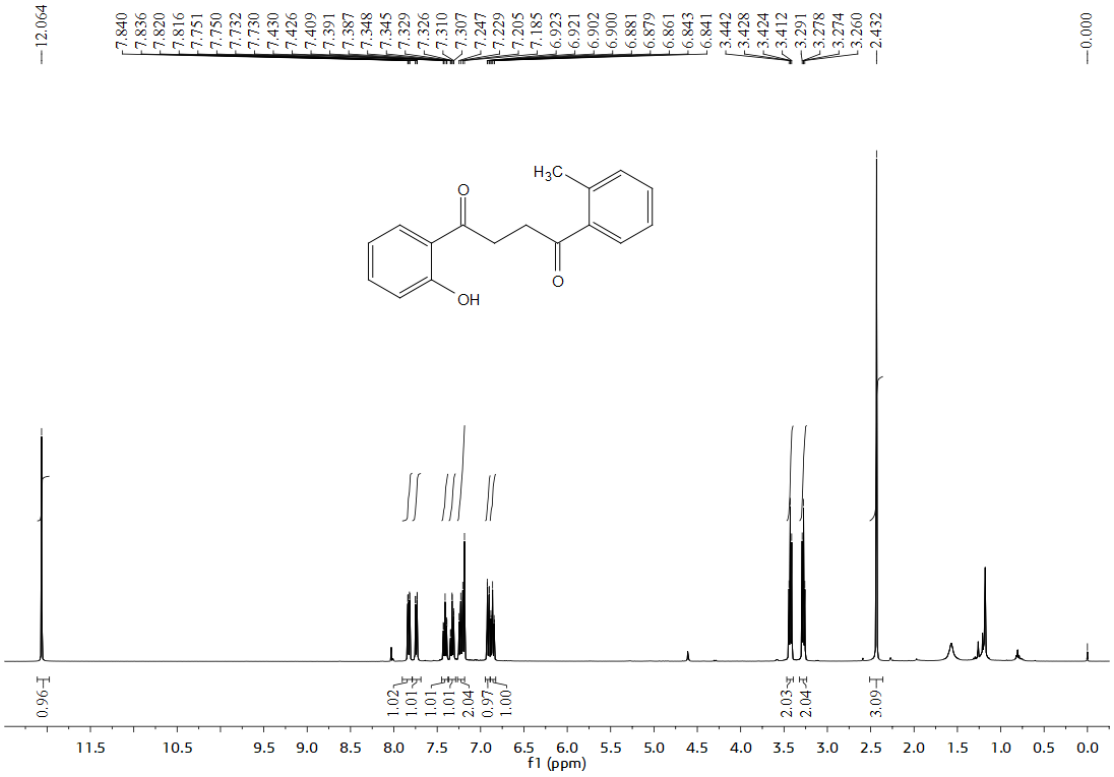


^{1}H NMR (400 MHz, $CDCl_3$) of **3ab**

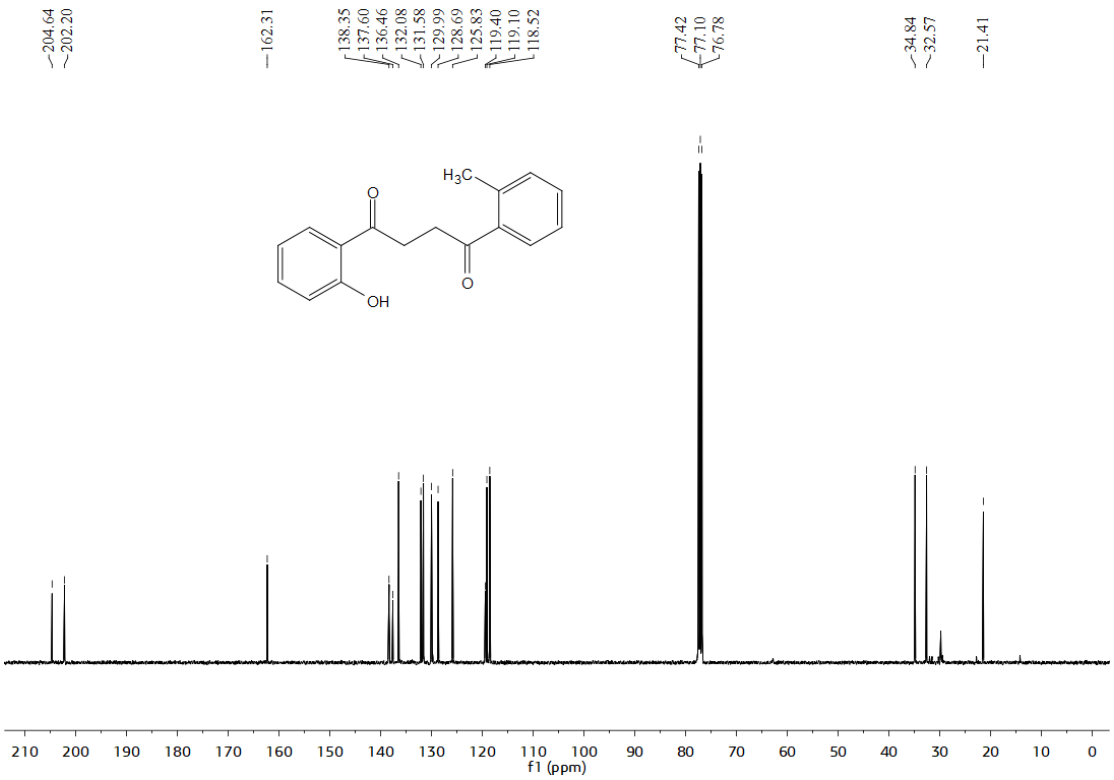


^{13}C NMR (100 MHz, $CDCl_3$) of **3ab**

1-(2-hydroxyphenyl)-4-(*o*-tolyl)butane-1,4-dione (3ac):

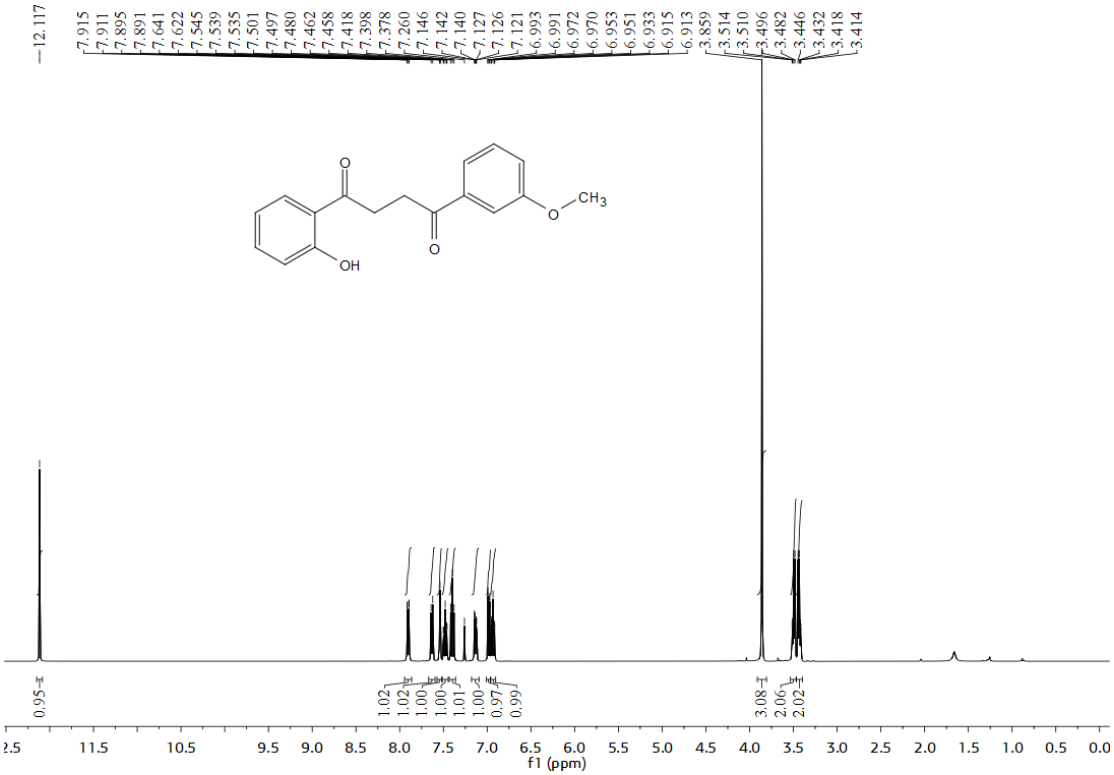


^{1}H NMR (400 MHz, $CDCl_3$) of **3ac**

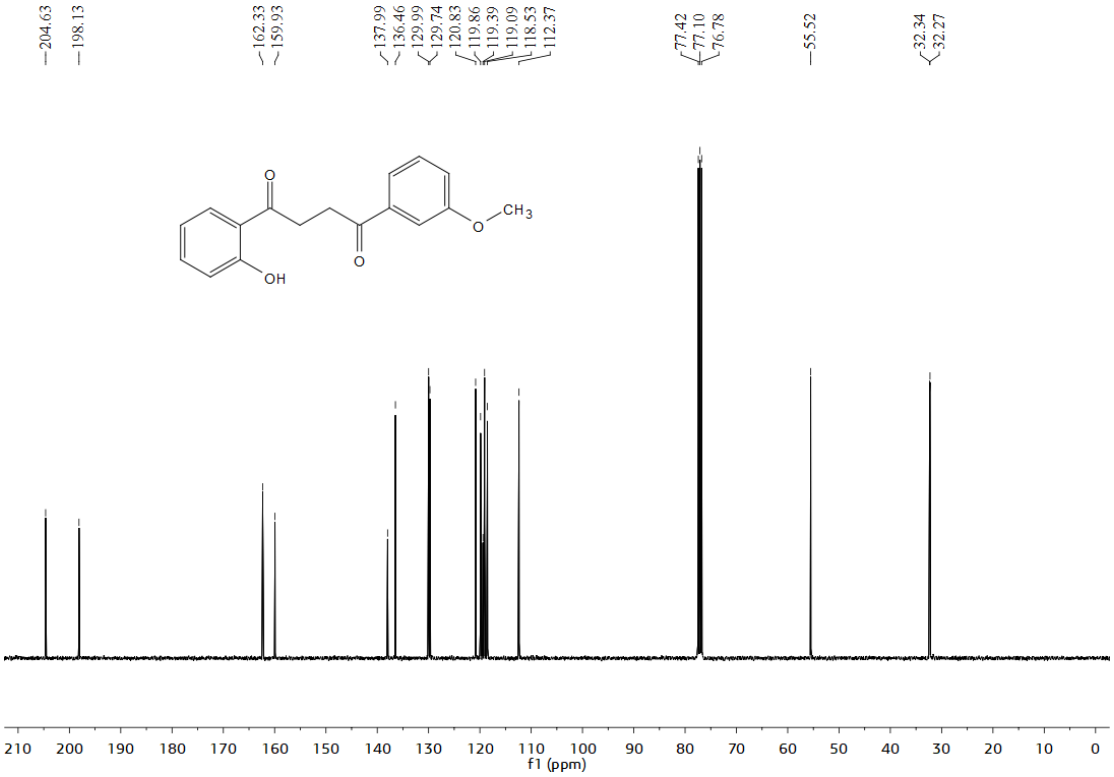


^{13}C NMR (100 MHz, $CDCl_3$) of **3ac**

1-(2-hydroxyphenyl)-4-(3-methoxyphenyl)butane-1,4-dione (3ad):

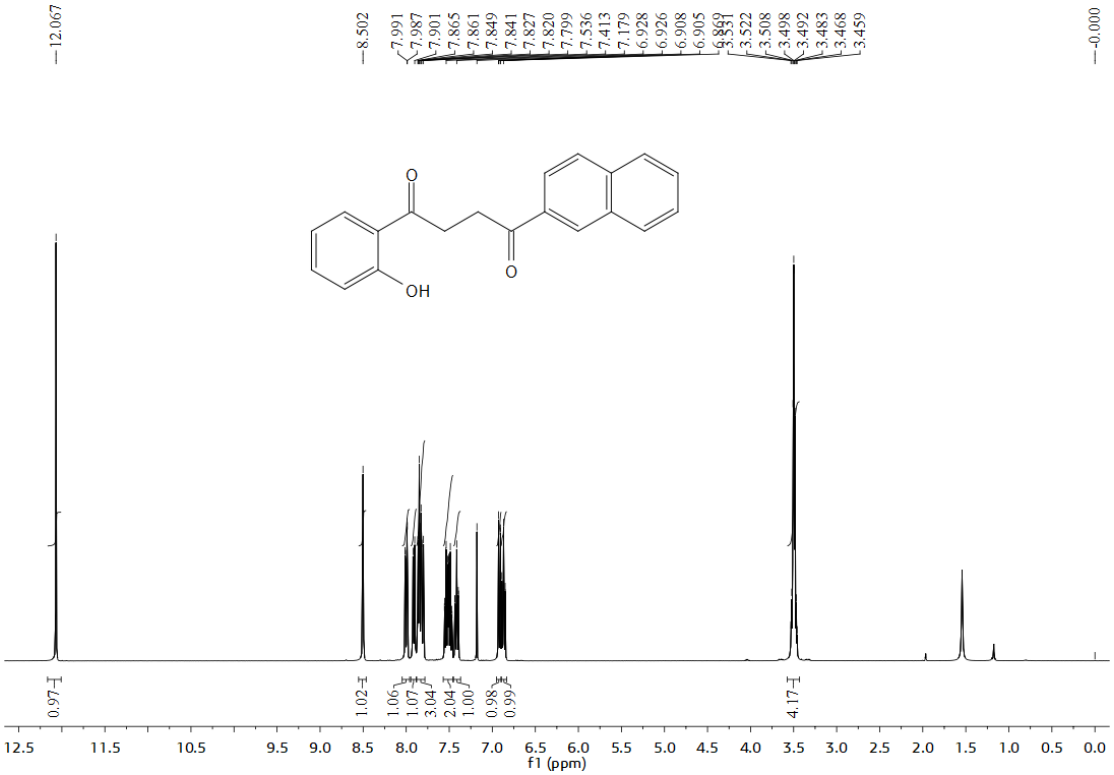


^{1}H NMR (400 MHz, $CDCl_3$) of **3ad**

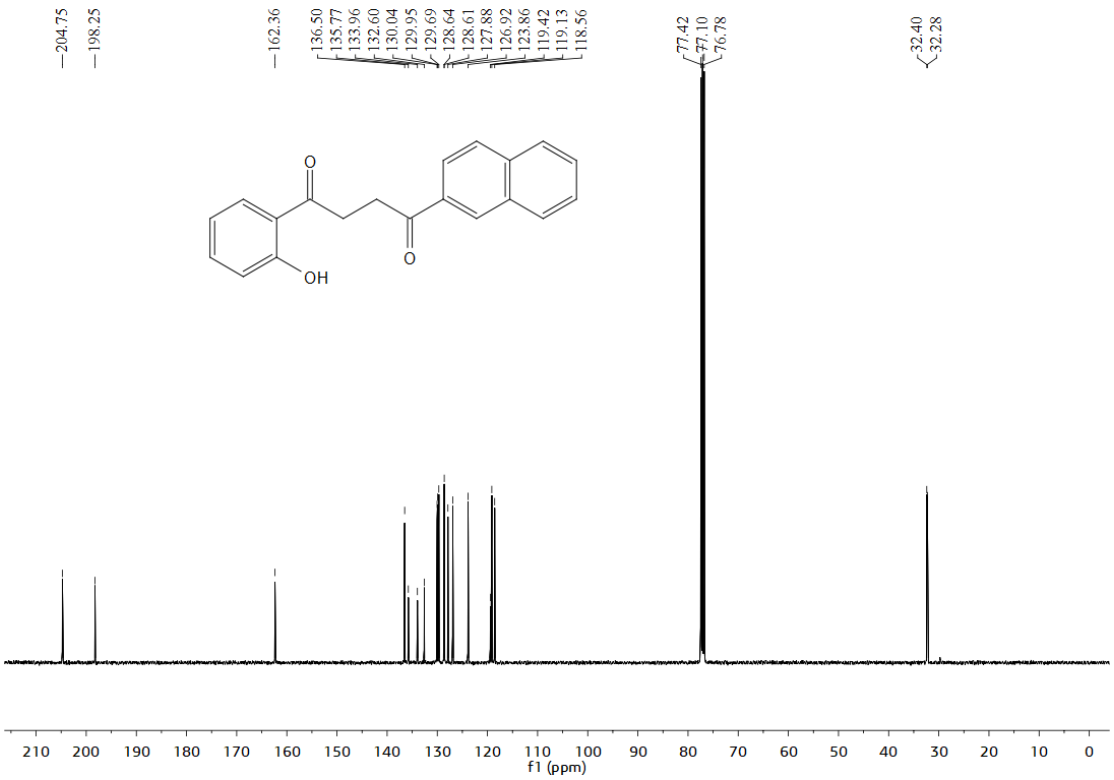


^{13}C NMR (100 MHz, $CDCl_3$) of **3ad**

1-(2-hydroxyphenyl)-4-(naphthalen-2-yl)butane-1,4-dione (3ae):

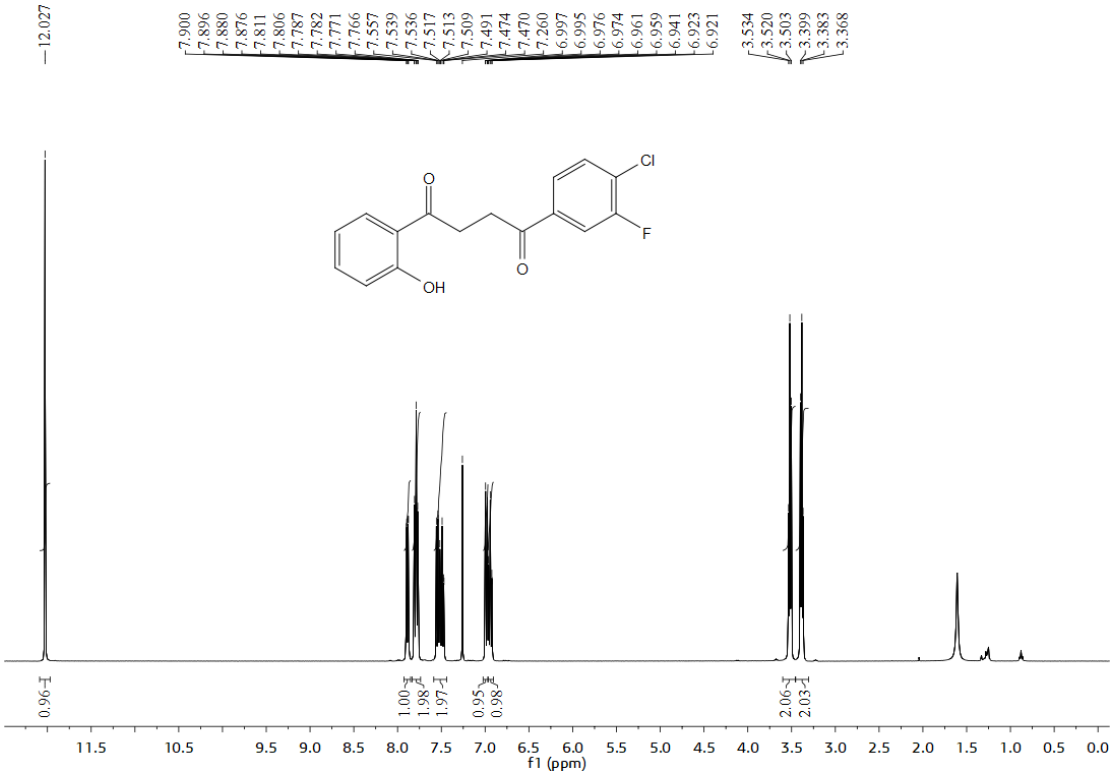


^{1}H NMR (400 MHz, $CDCl_3$) of **3ae**



^{13}C NMR (100 MHz, $CDCl_3$) of **3ae**

1-(4-chloro-3-fluorophenyl)-4-(2-hydroxyphenyl)butane-1,4-dione (3af):



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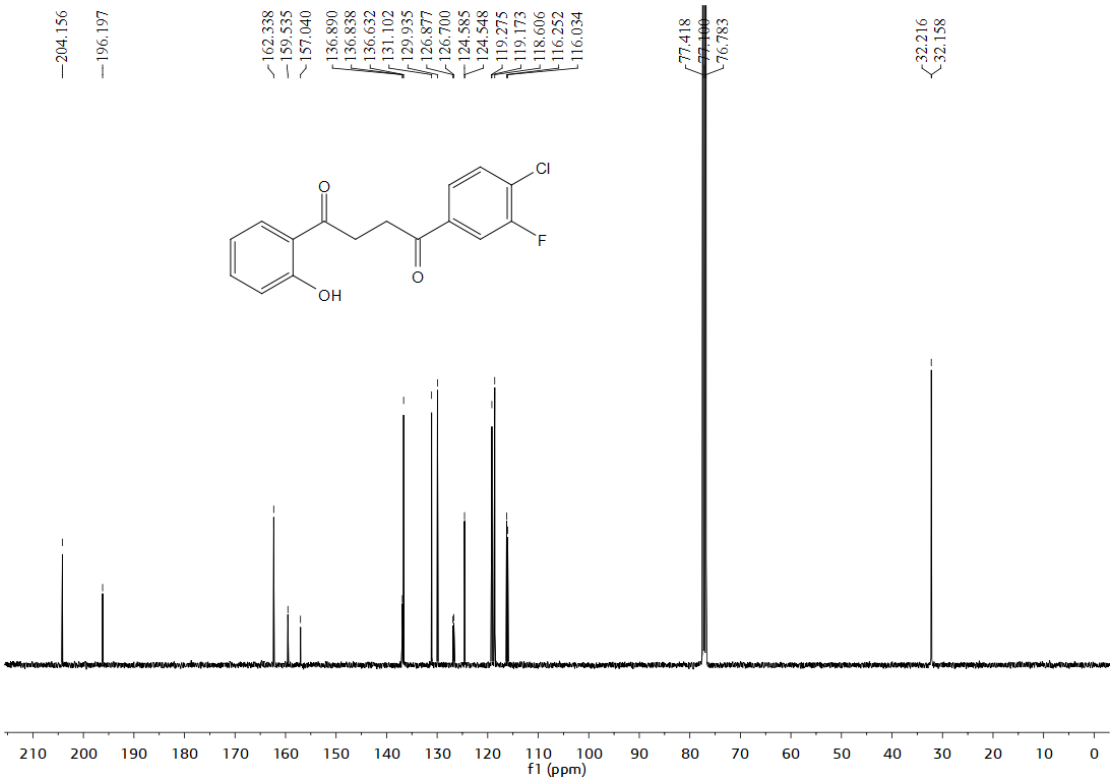
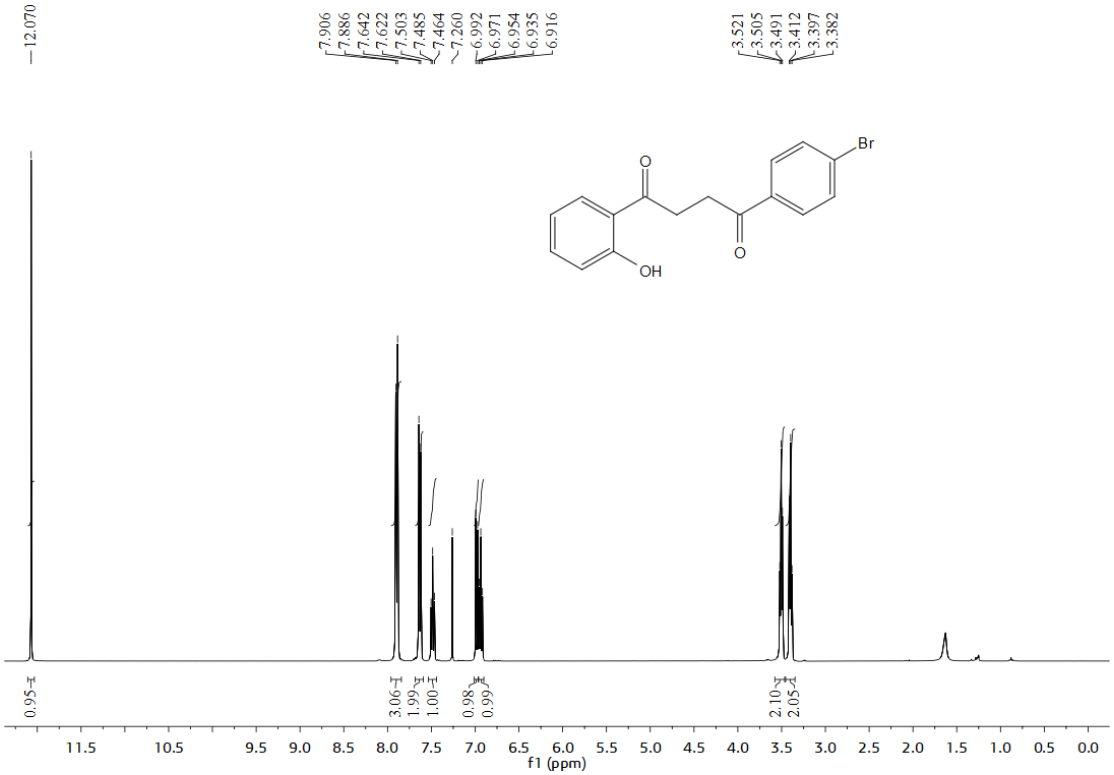
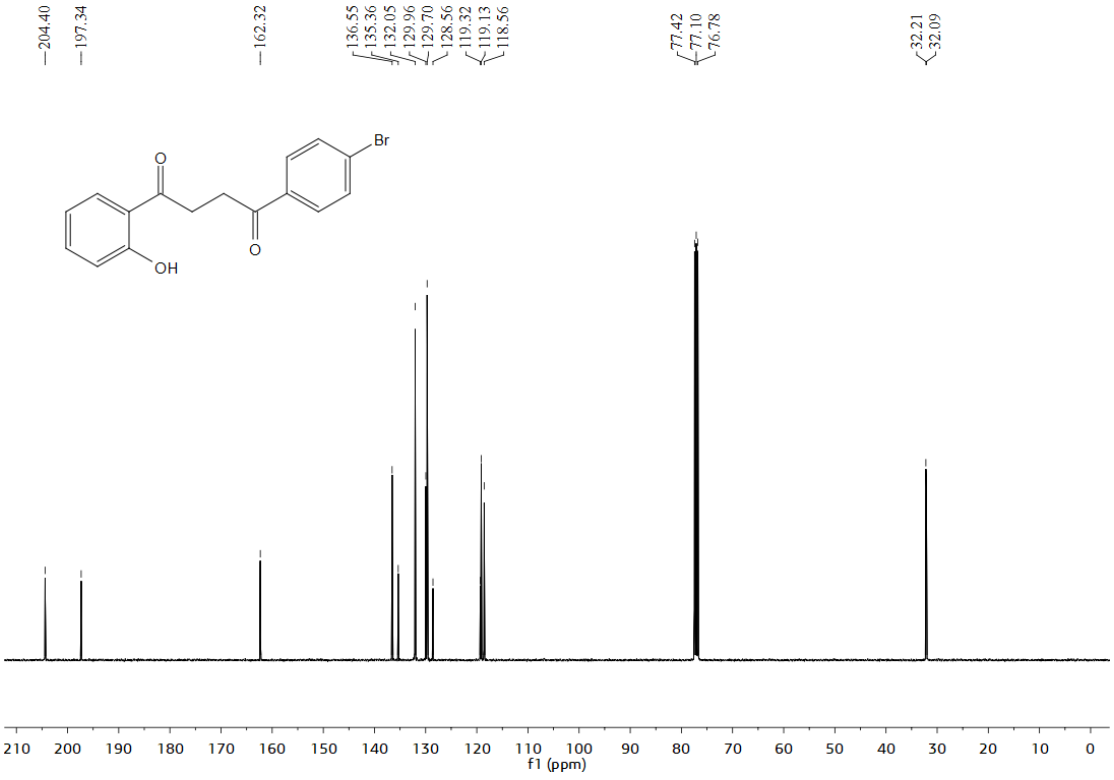


^{13}C NMR (100 MHz, $CDCl_3$) of **3af**

1-(4-bromophenyl)-4-(2-hydroxyphenyl)butane-1,4-dione (3ag):

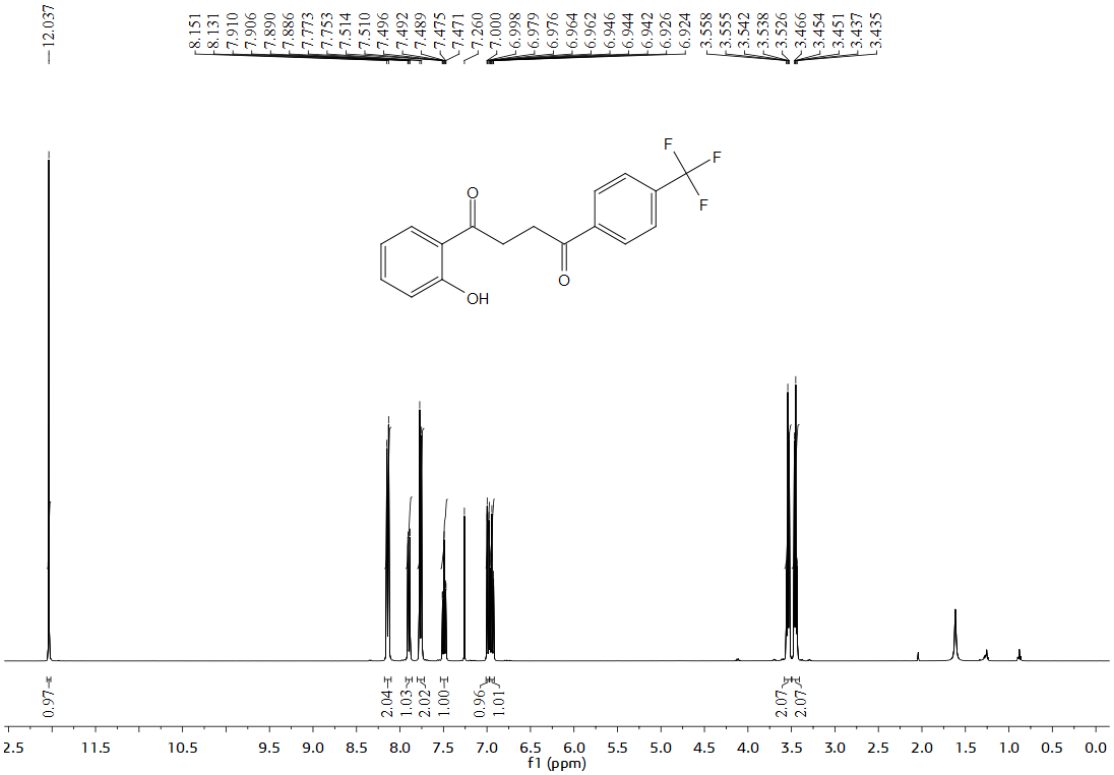


^{1}H NMR (400 MHz, $CDCl_3$) of **3ag**

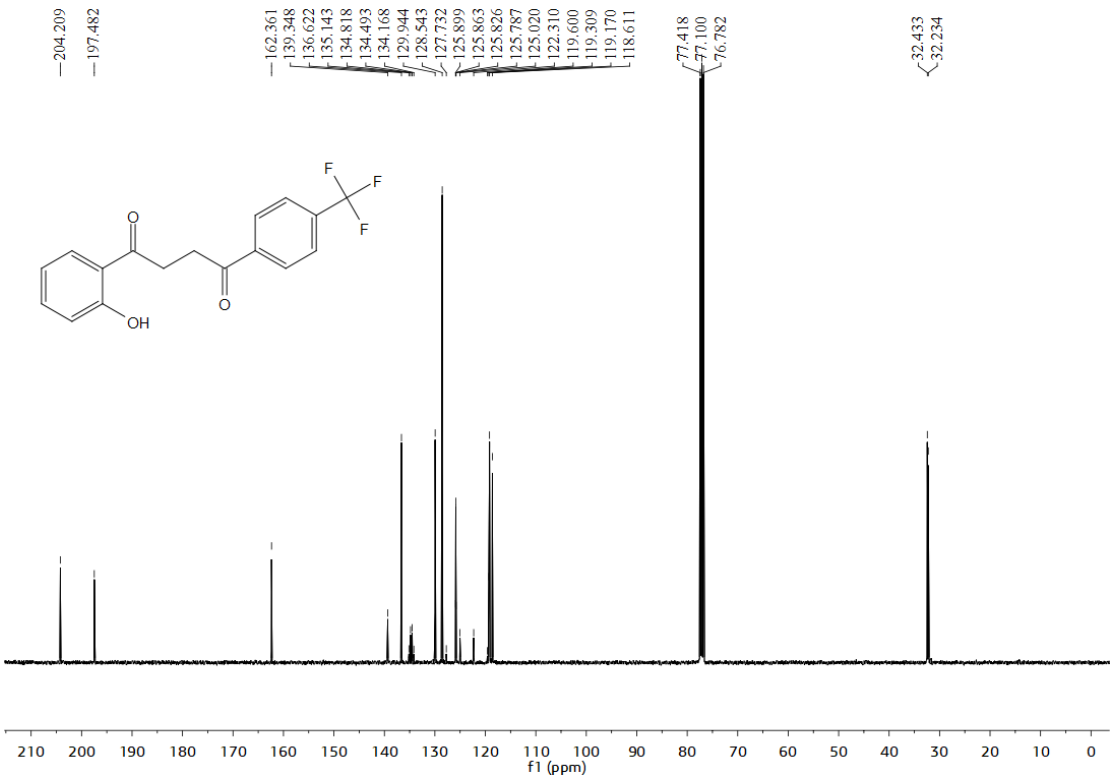


^{13}C NMR (100 MHz, $CDCl_3$) of **3ag**

1-(2-hydroxyphenyl)-4-(4-(trifluoromethyl)phenyl)butane-1,4-dione (3ah):

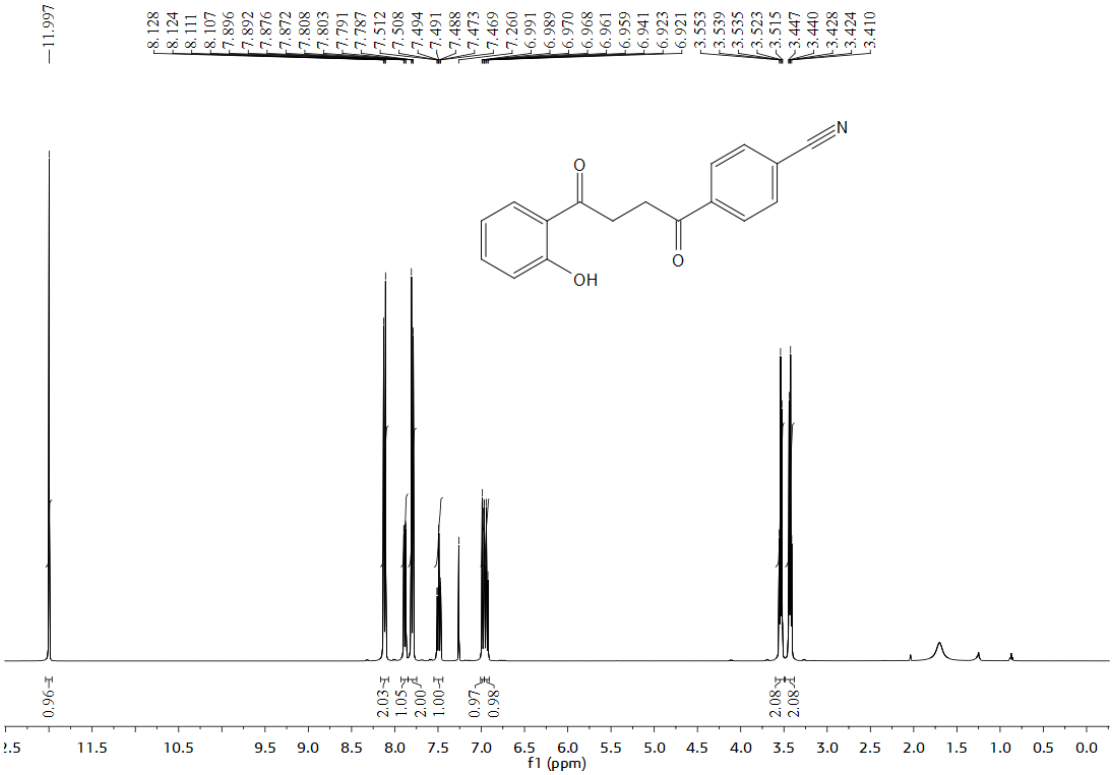


^{1}H NMR (400 MHz, $CDCl_3$) of **3ah**



^{13}C NMR (100 MHz, $CDCl_3$) of **3ah**

4-(4-(2-hydroxyphenyl)-4-oxobutanoyl)benzonitrile (3ai):



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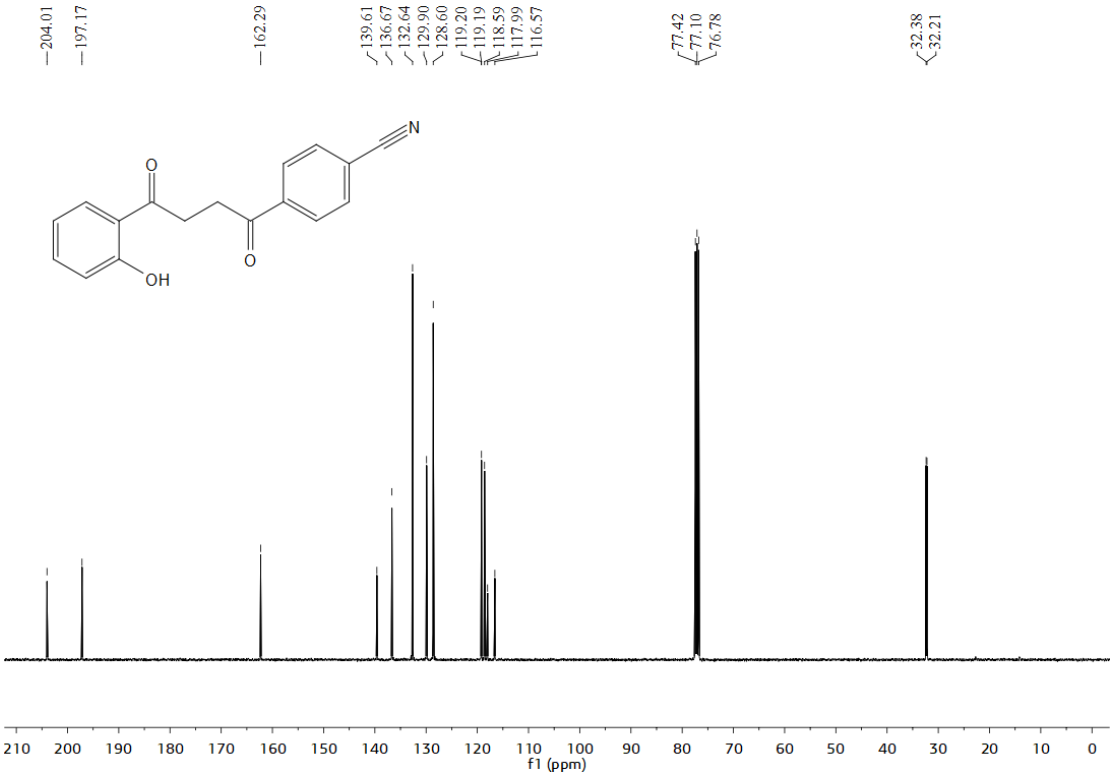
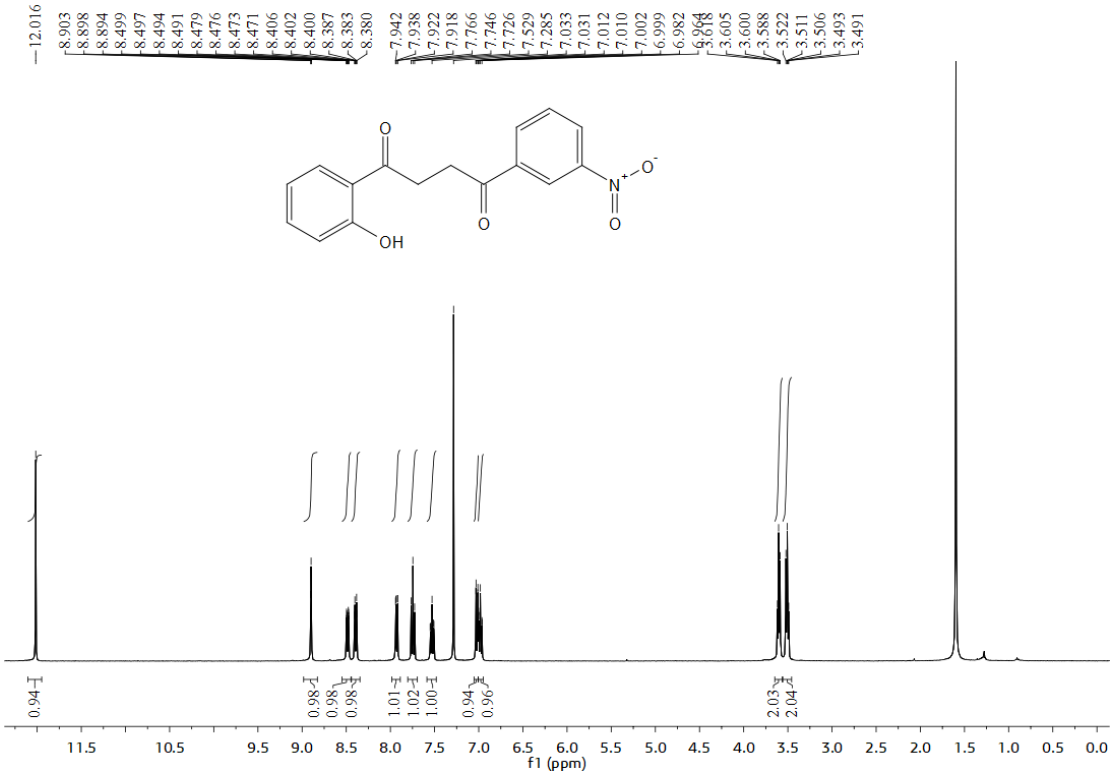
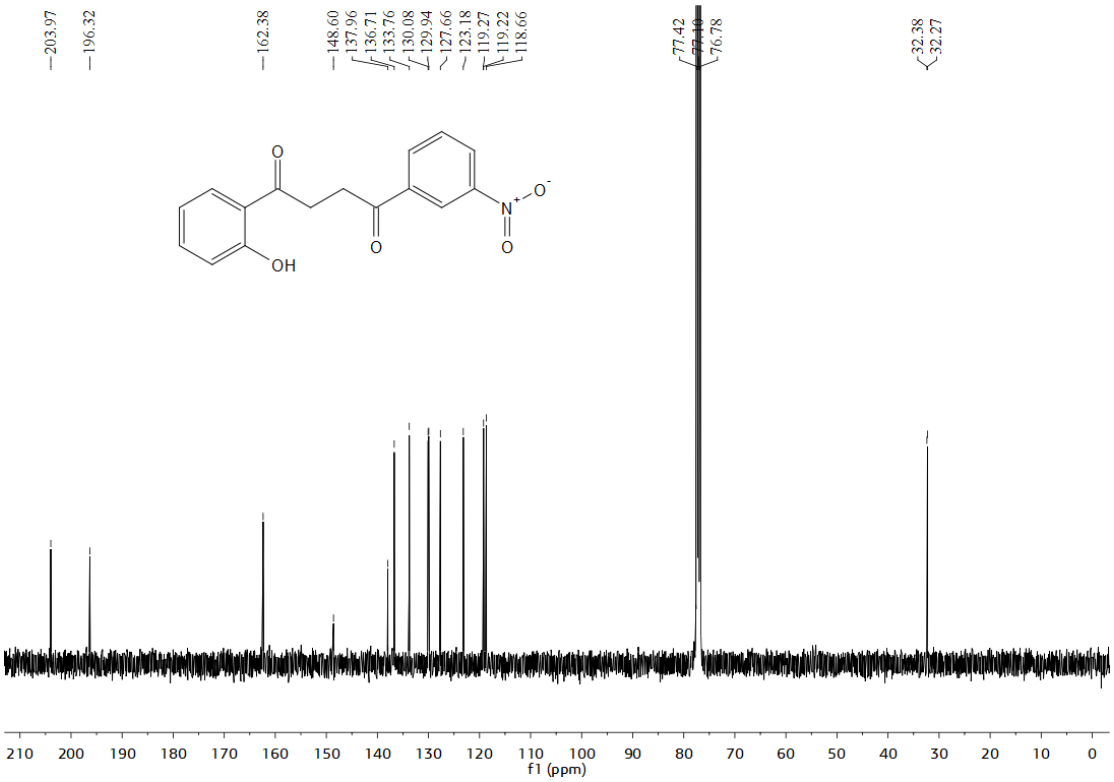


^{13}C NMR (100 MHz, $CDCl_3$) of **3ai**

1-(2-hydroxyphenyl)-4-(3-nitrophenyl)butane-1,4-dione (3aj):

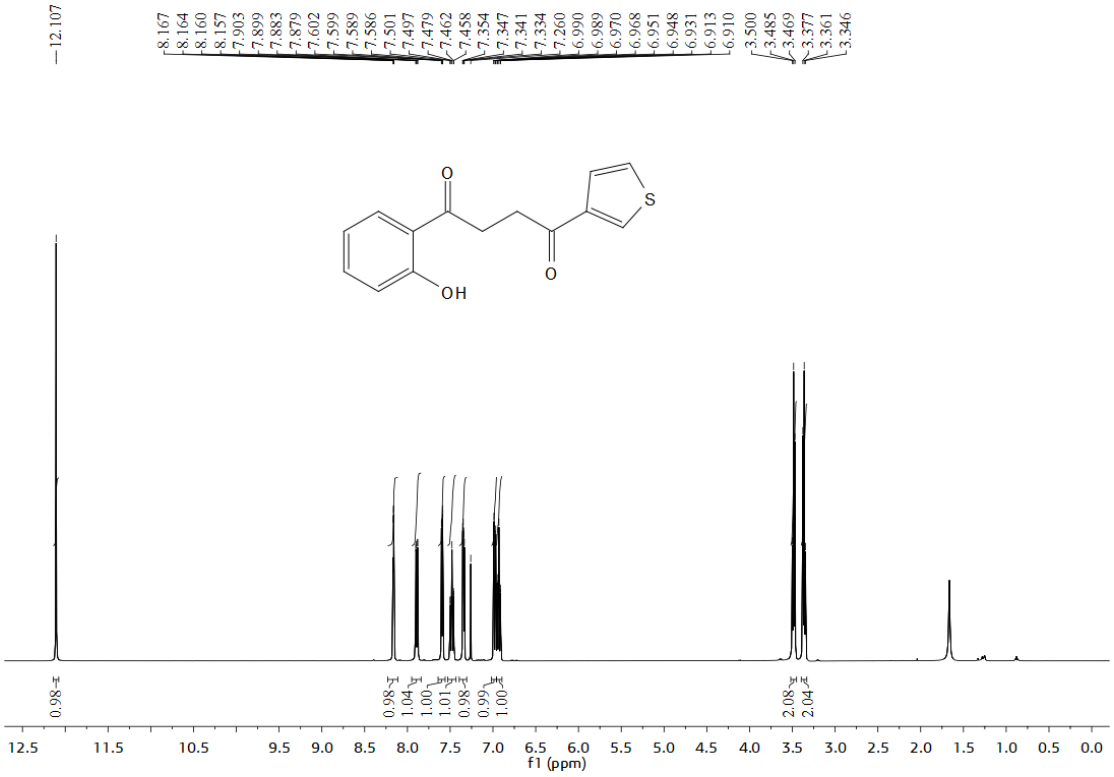


^{1}H NMR (400 MHz, $CDCl_3$) of **3aj**

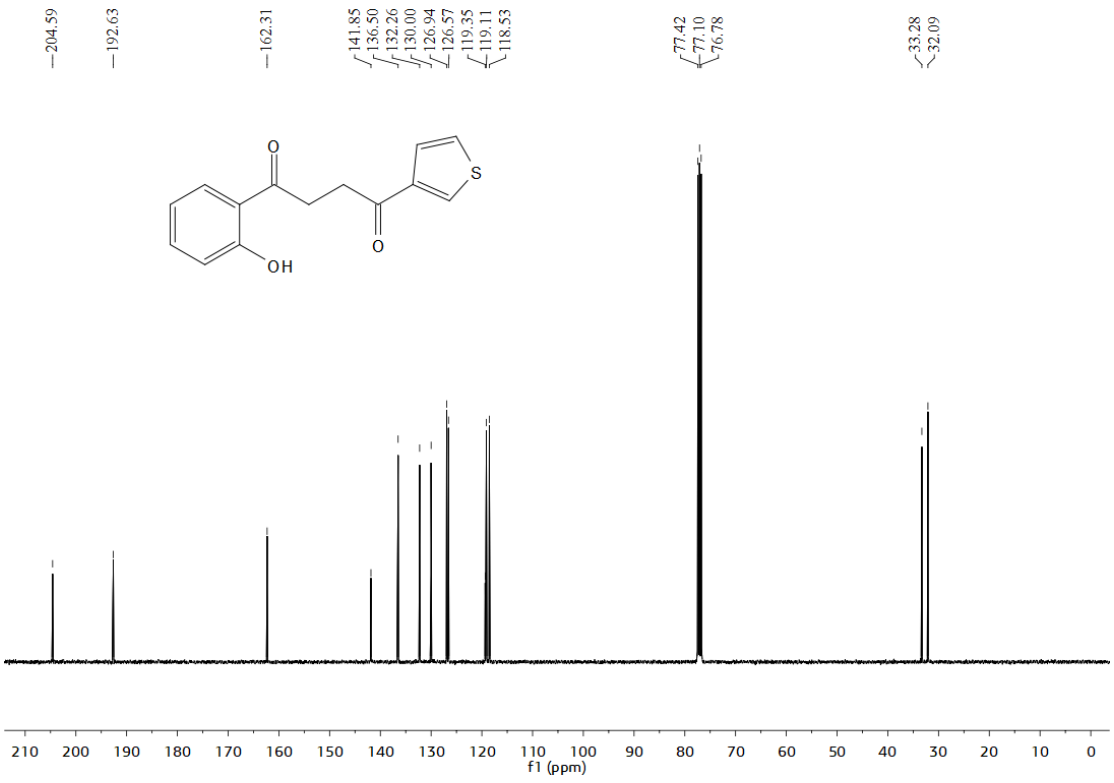


^{13}C NMR (100 MHz, $CDCl_3$) of **3aj**

1-(2-hydroxyphenyl)-4-(thiophen-3-yl)butane-1,4-dione (3al):

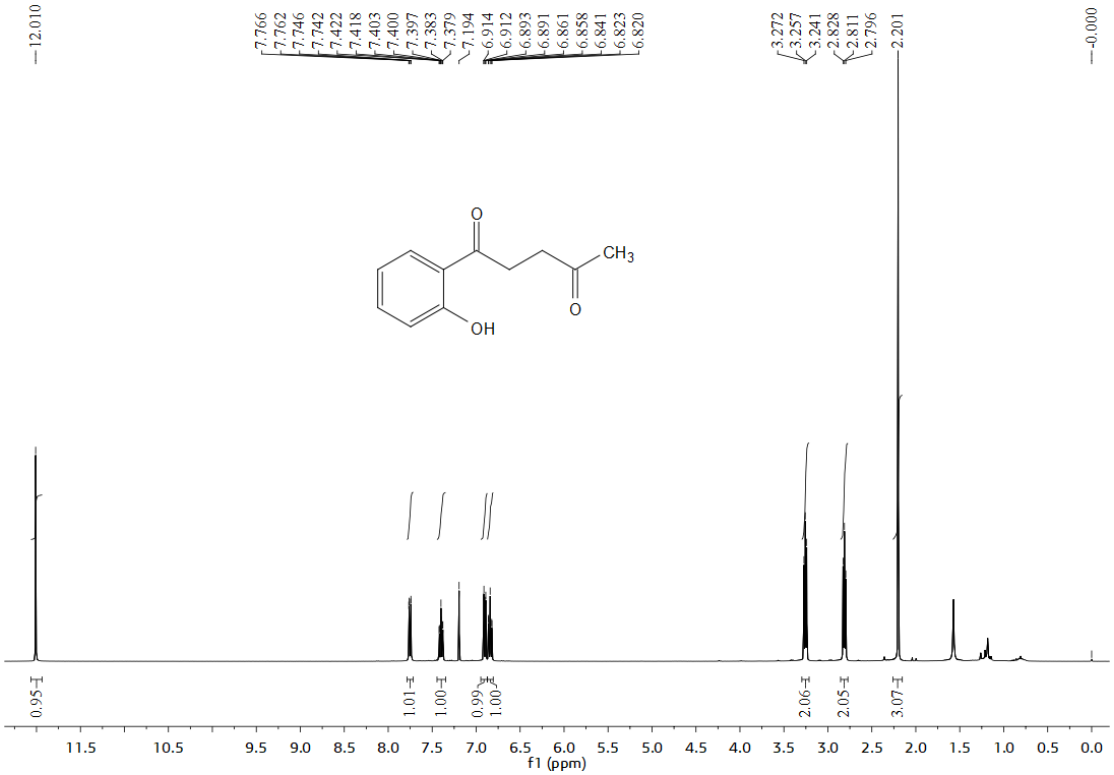


^{1}H NMR (400 MHz, $CDCl_3$) of **3al**

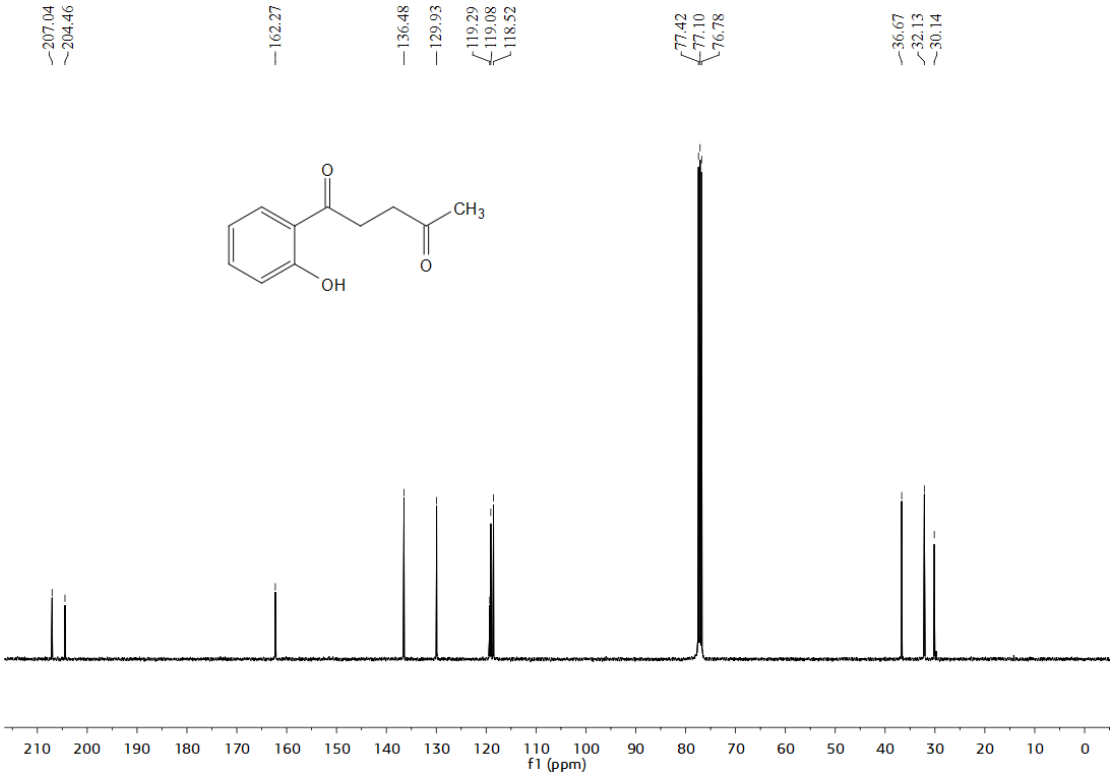


^{13}C NMR (100 MHz, $CDCl_3$) of **3al**

1-(2-hydroxyphenyl)pentane-1,4-dione (3am):

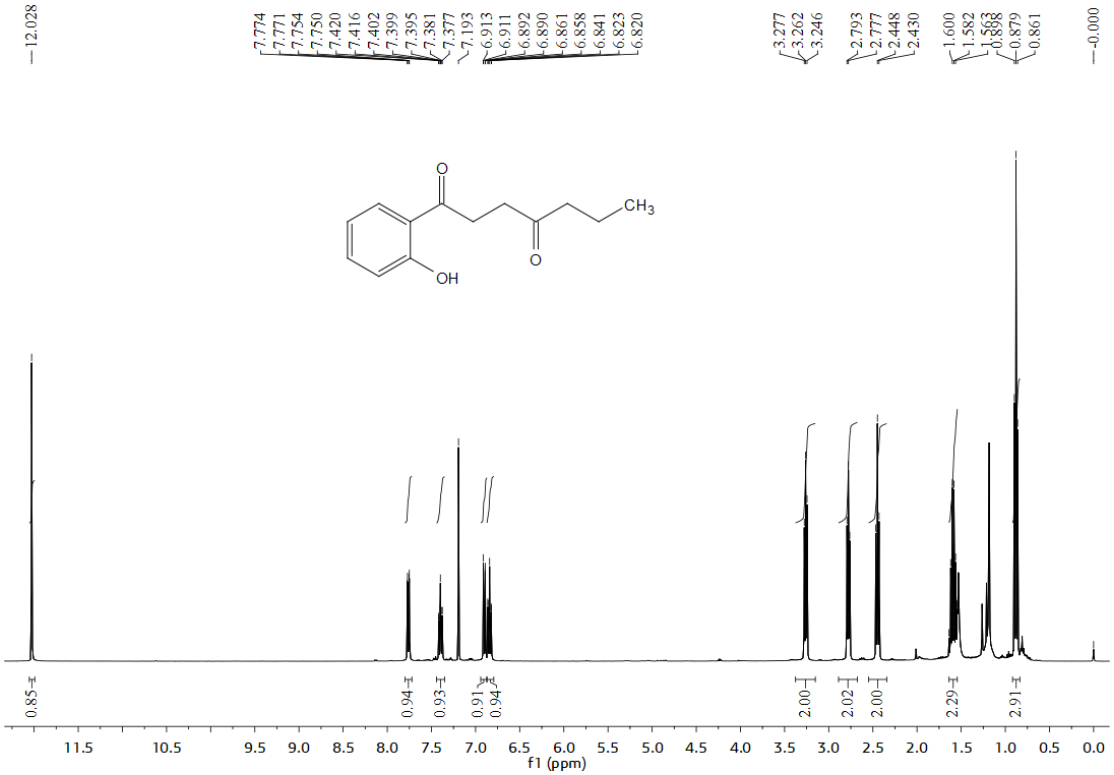


^{1}H NMR (400 MHz, $CDCl_3$) of **3am**

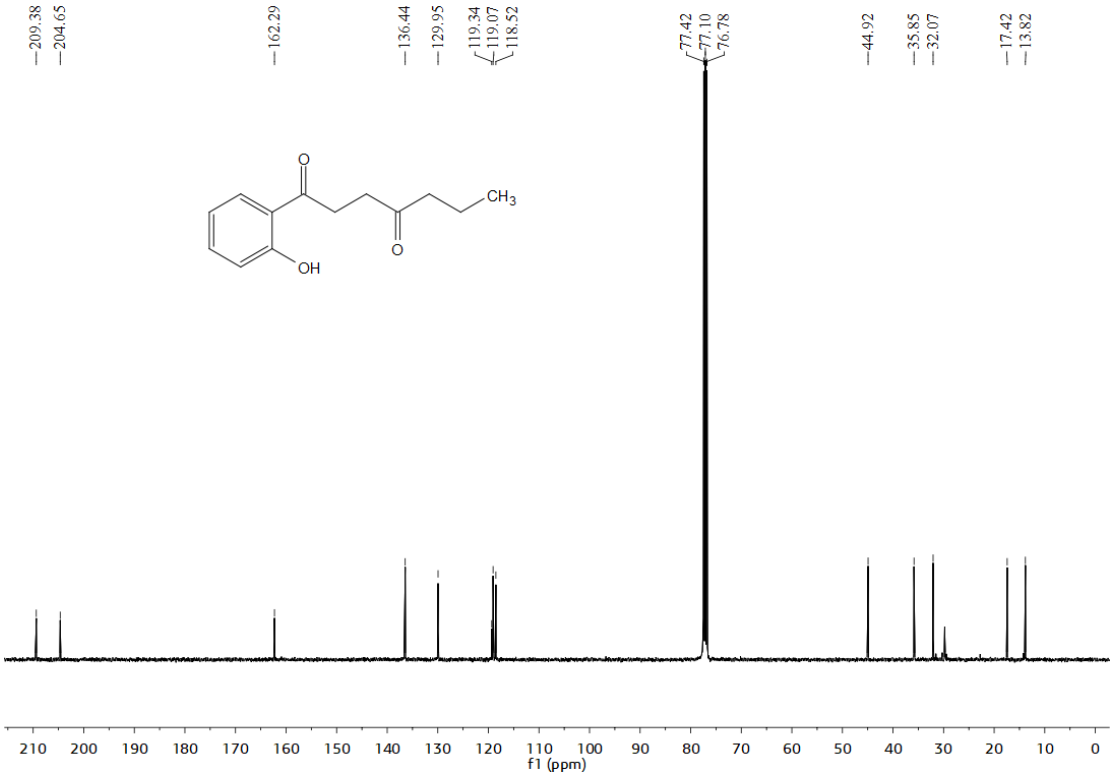


^{13}C NMR (100 MHz, $CDCl_3$) of **3am**

1-(2-hydroxyphenyl)heptane-1,4-dione (3an):

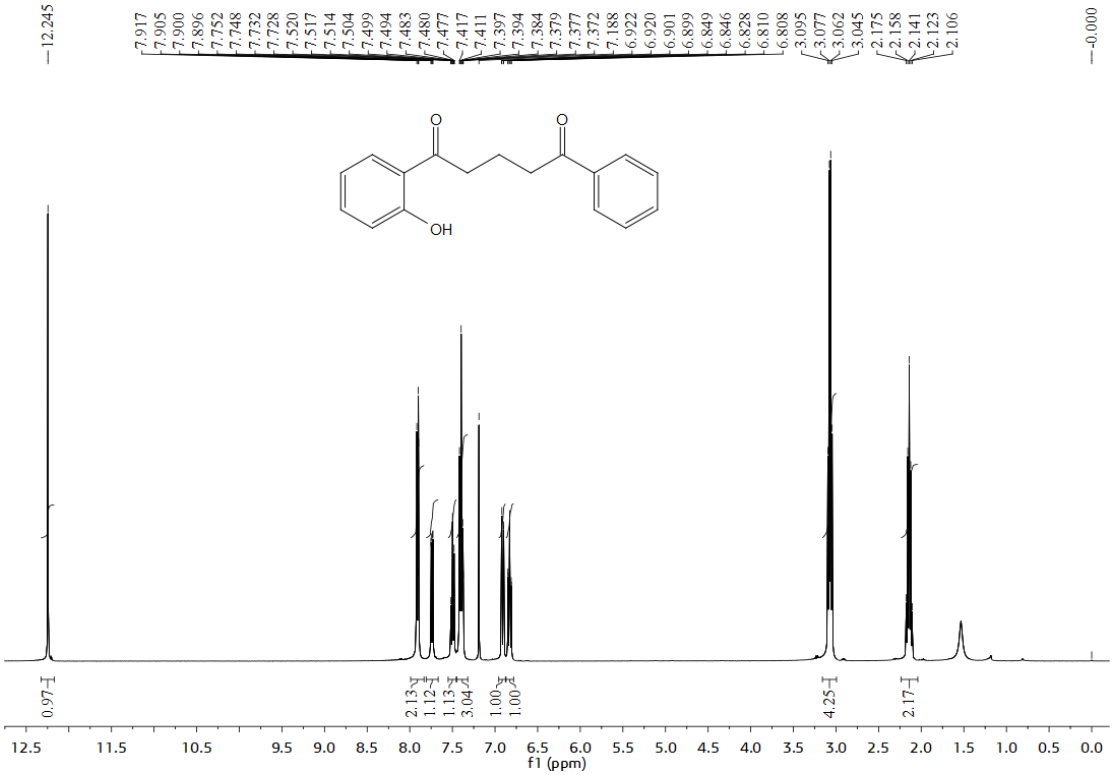


^{1}H NMR (400 MHz, $CDCl_3$) of **3an**

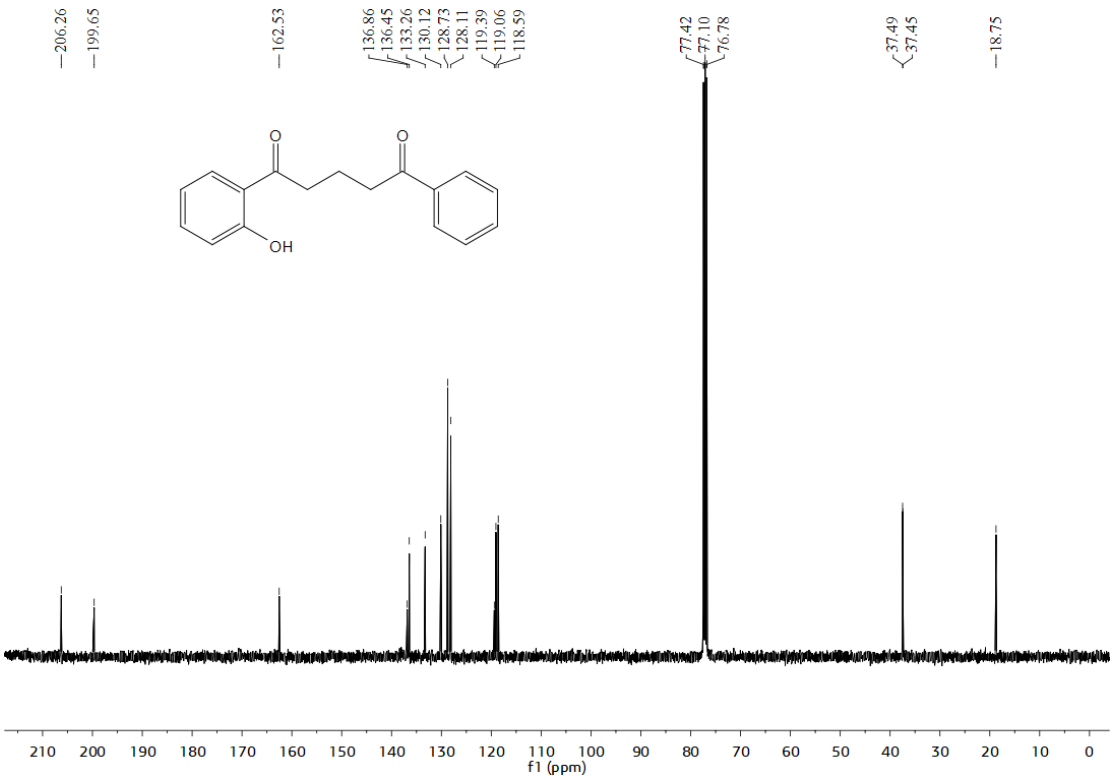


^{13}C NMR (100 MHz, $CDCl_3$) of **3an**

1-(2-hydroxyphenyl)-5-phenylpentane-1,5-dione (3aq):

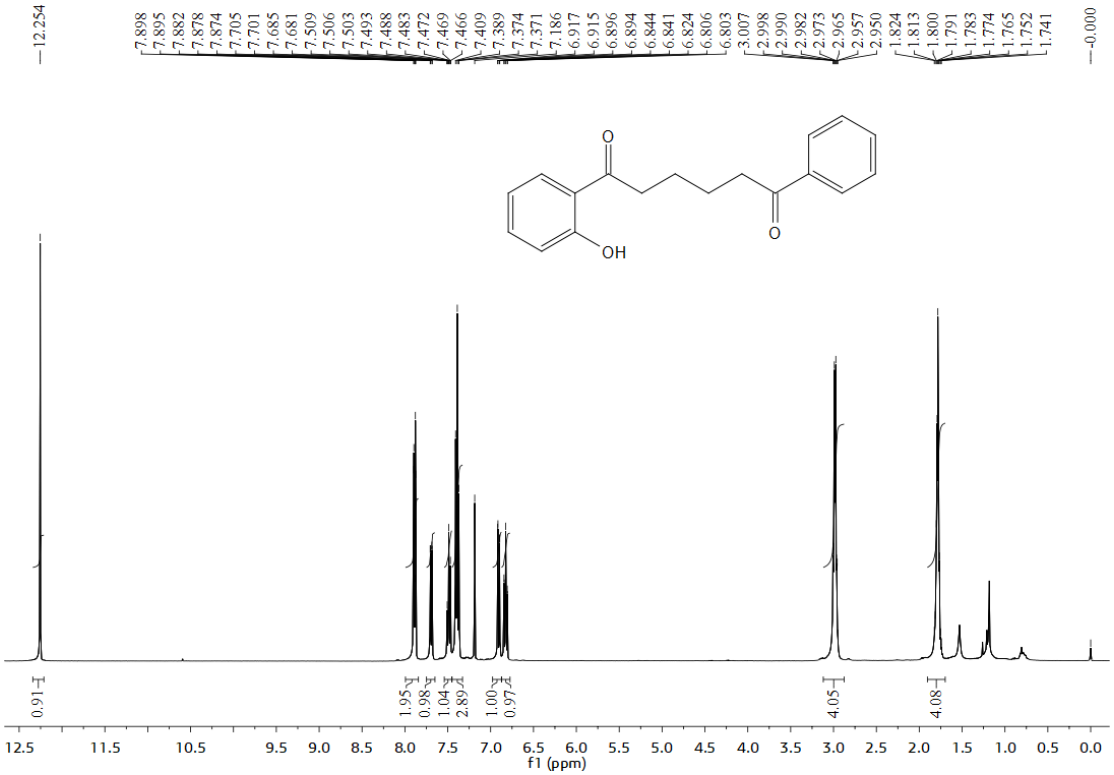


^{1}H NMR (400 MHz, $CDCl_3$) of **3aq**

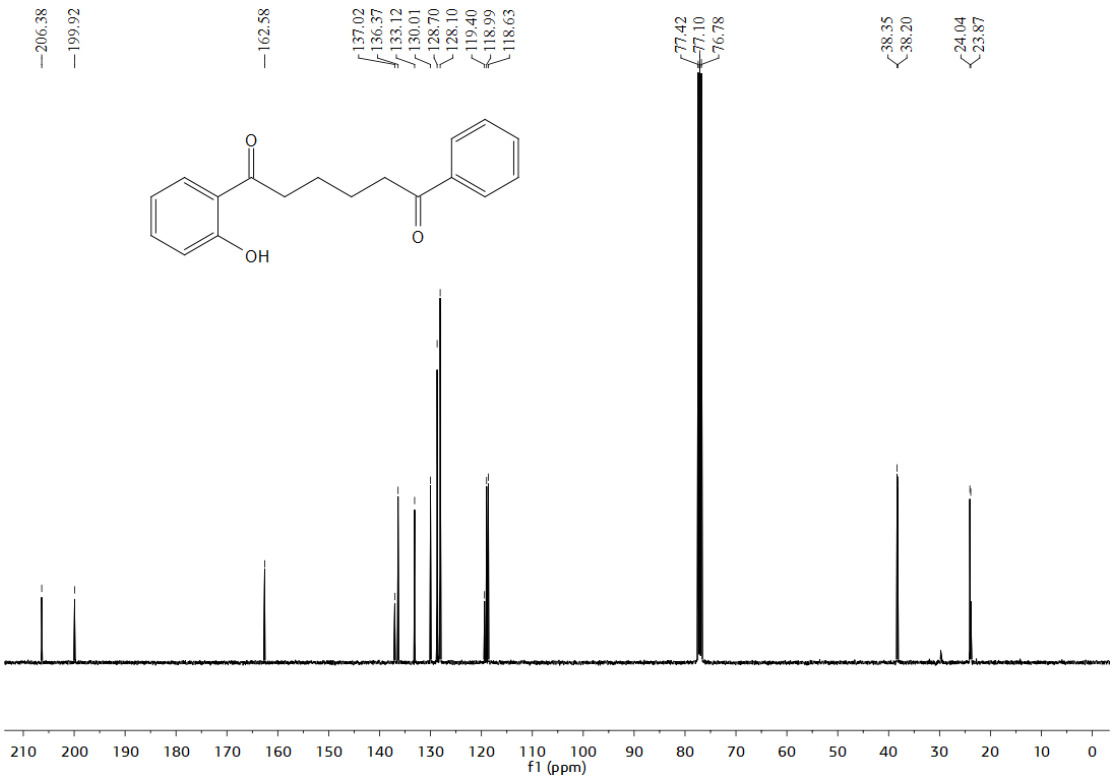


^{13}C NMR (100 MHz, $CDCl_3$) of **3aq**

1-(2-hydroxyphenyl)-6-phenylhexane-1,6-dione (3ar):

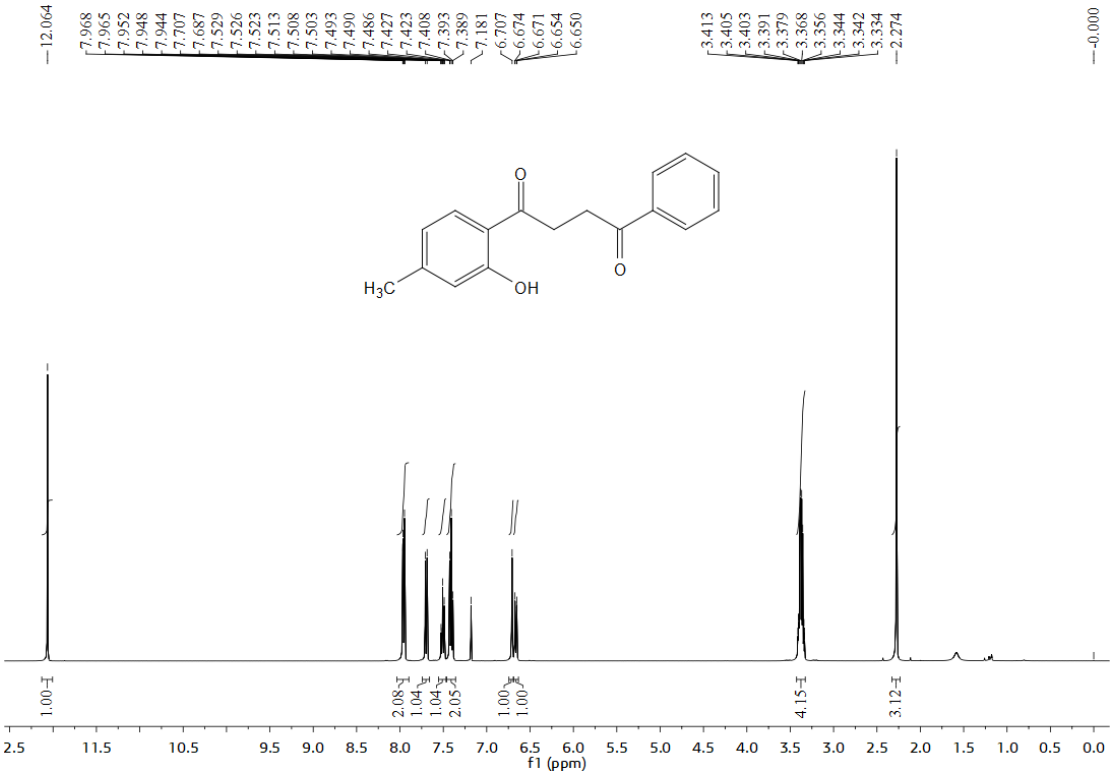


^{1}H NMR (400 MHz, $CDCl_3$) of **3ar**

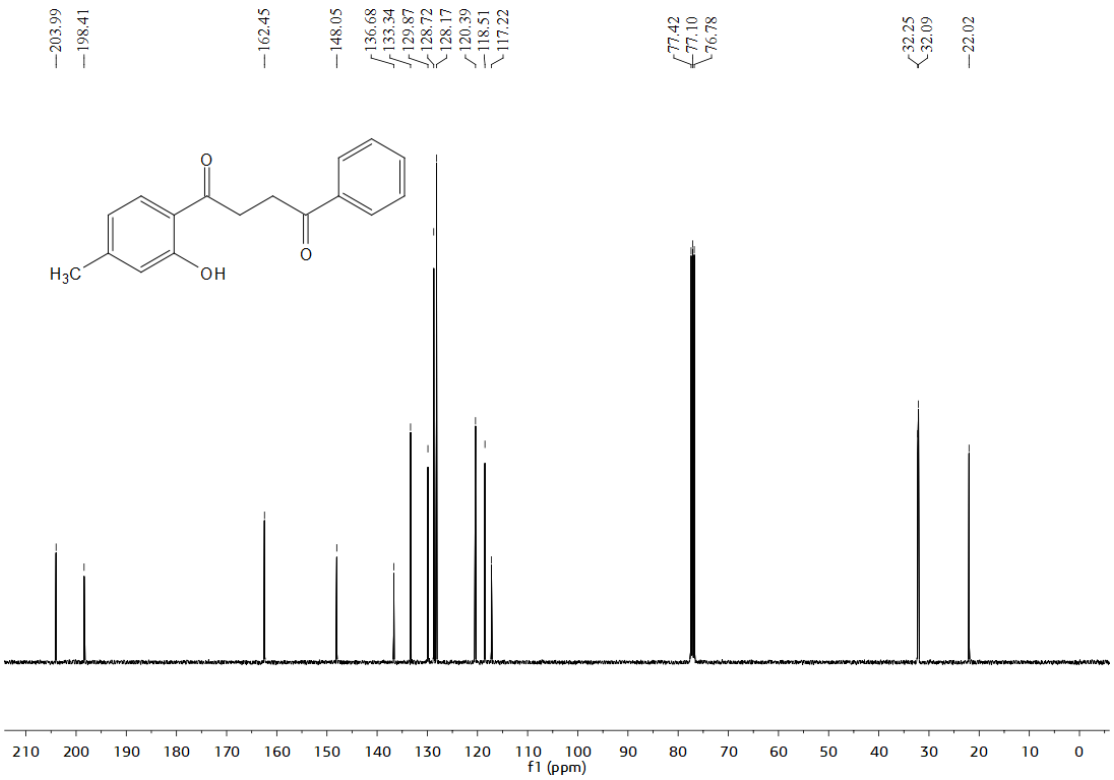


^{13}C NMR (100 MHz, $CDCl_3$) of **3ar**

1-(2-hydroxy-4-methylphenyl)-4-phenylbutane-1,4-dione (3ba):

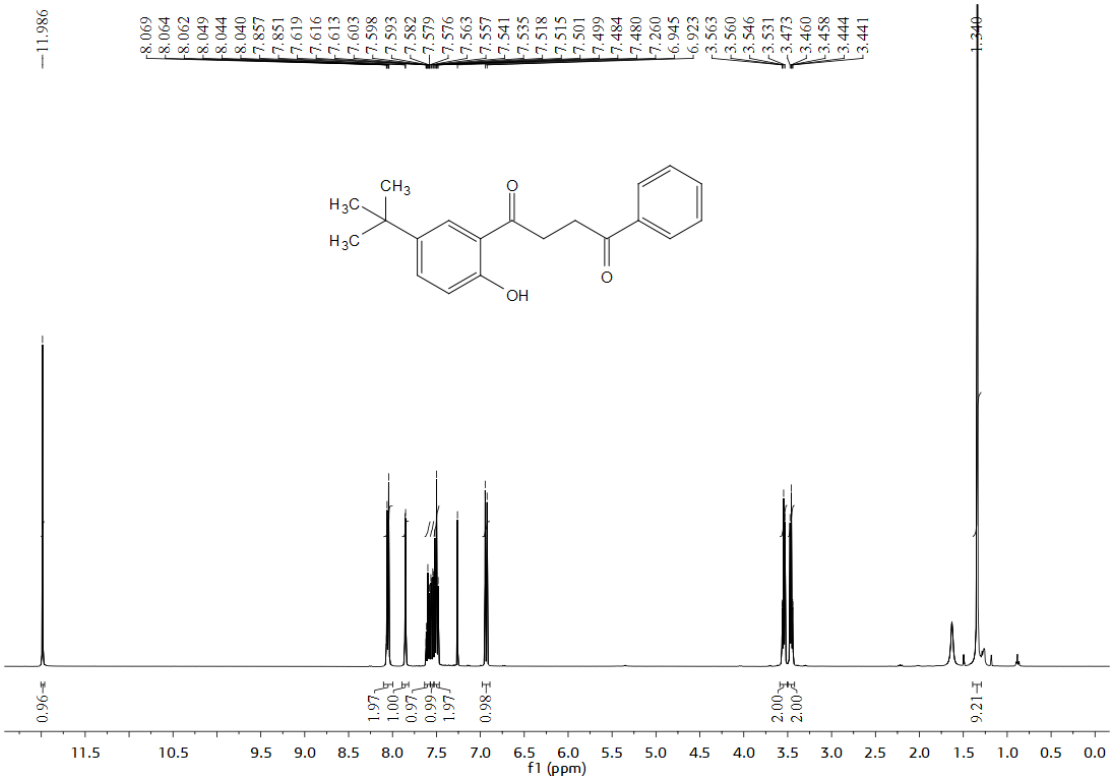


^{1}H NMR (400 MHz, $CDCl_3$) of **3ba**

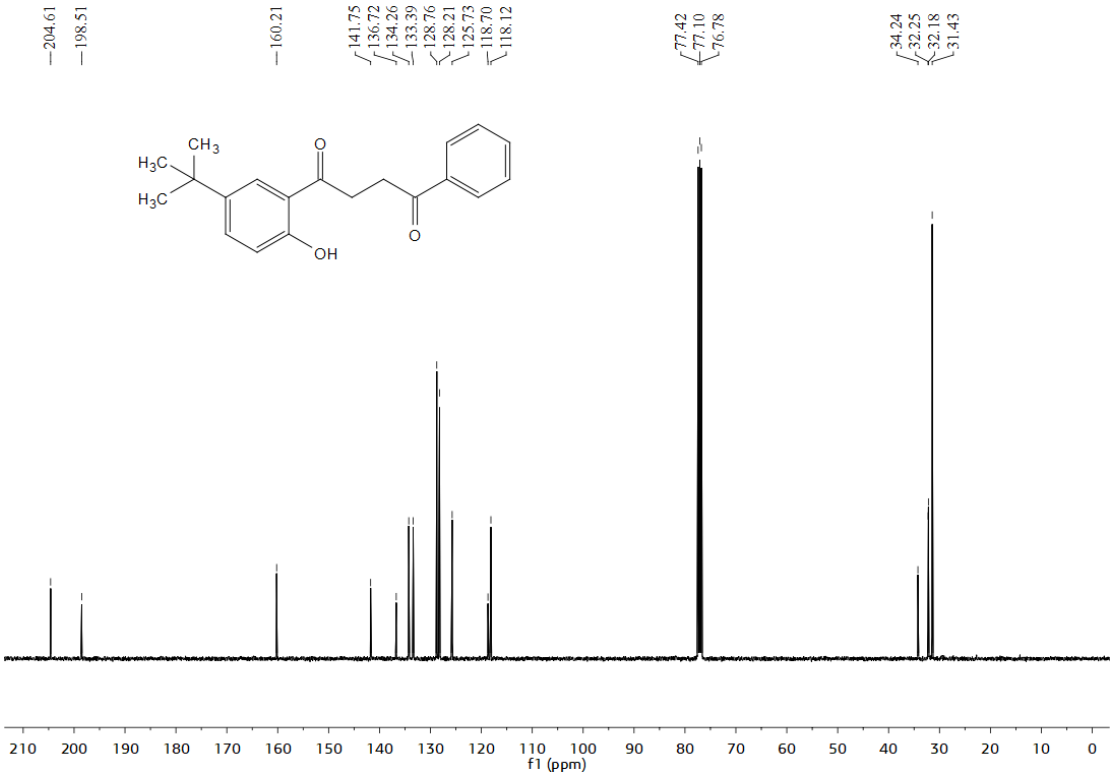


^{13}C NMR (100 MHz, $CDCl_3$) of **3ba**

1-(5-(*tert*-butyl)-2-hydroxyphenyl)-4-phenylbutane-1,4-dione (3ca):

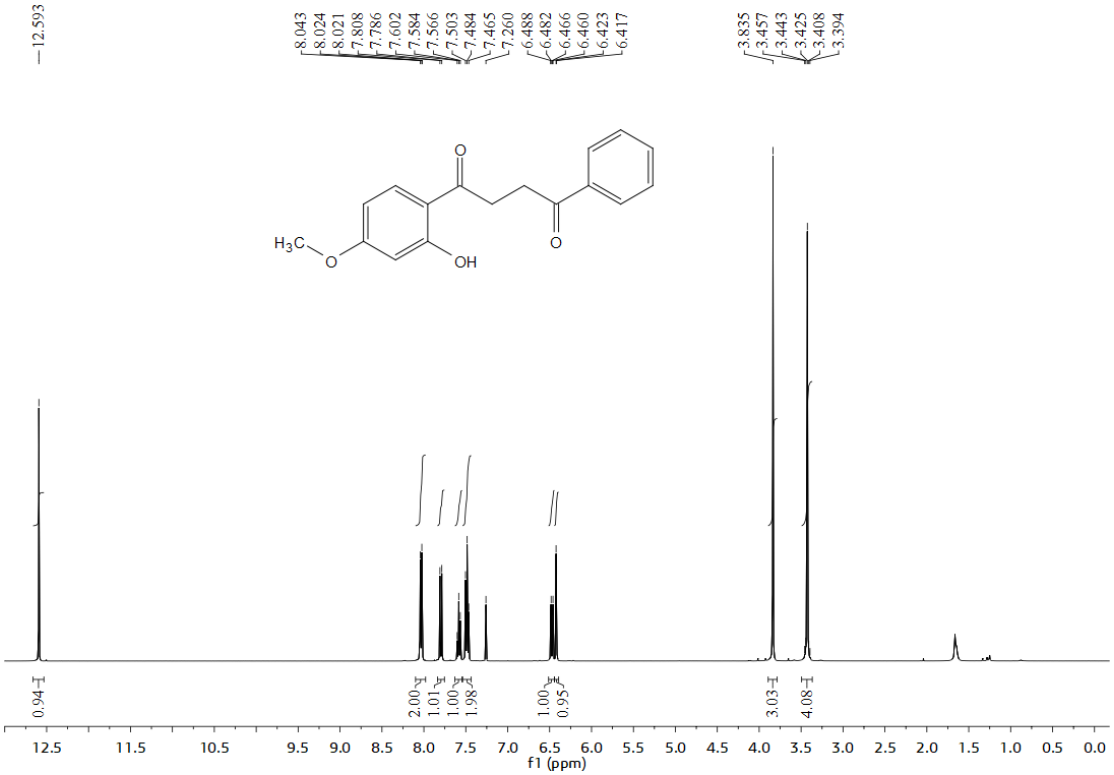


^{1}H NMR (400 MHz, $CDCl_3$) of **3ca**

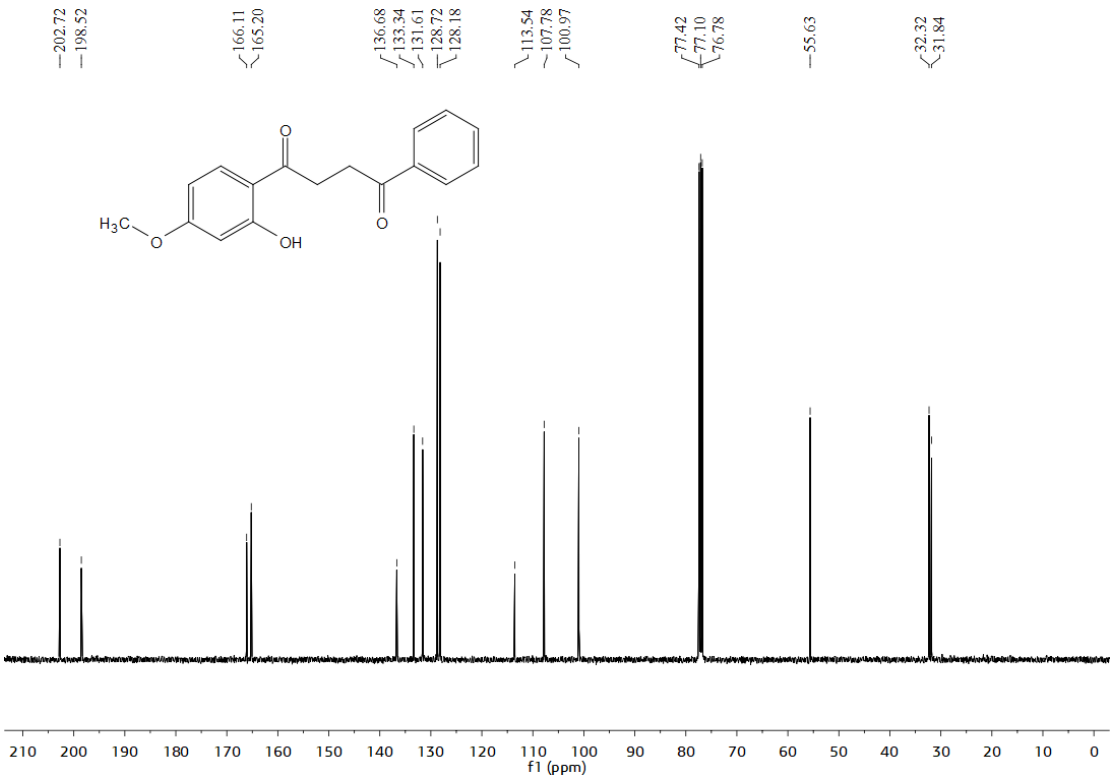


^{13}C NMR (100 MHz, $CDCl_3$) of **3ca**

1-(2-hydroxy-4-methoxyphenyl)-4-phenylbutane-1,4-dione (3da):

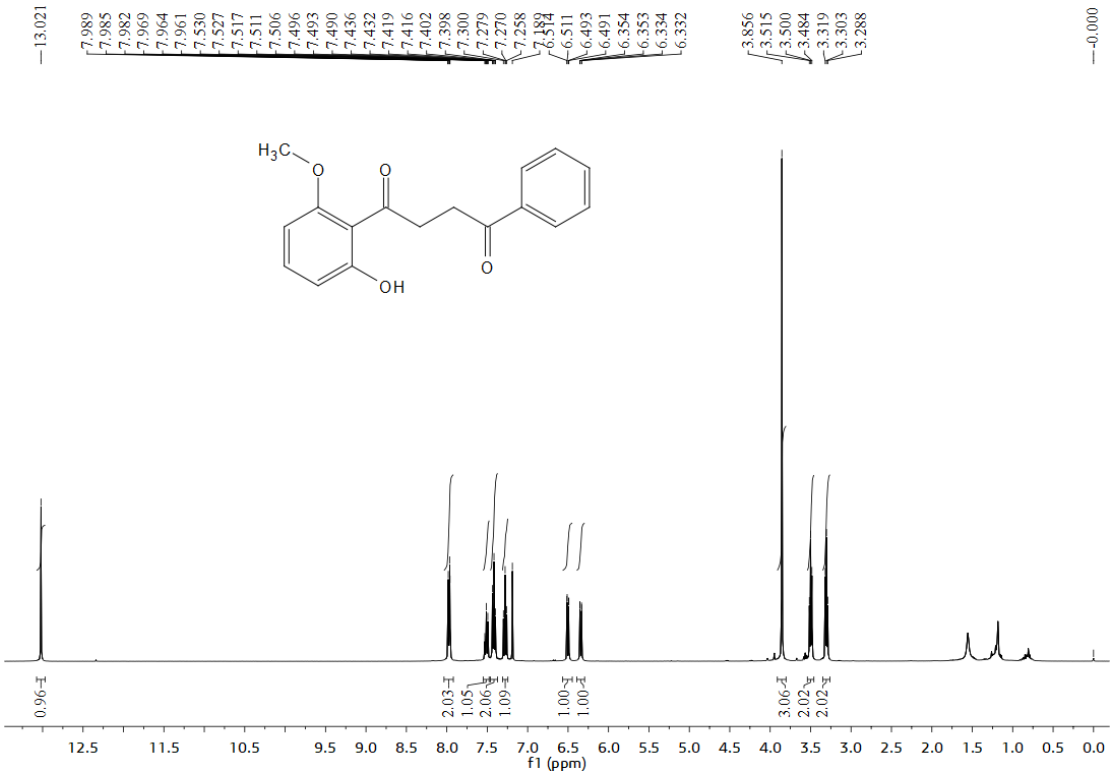


^{1}H NMR (400 MHz, $CDCl_3$) of **3da**

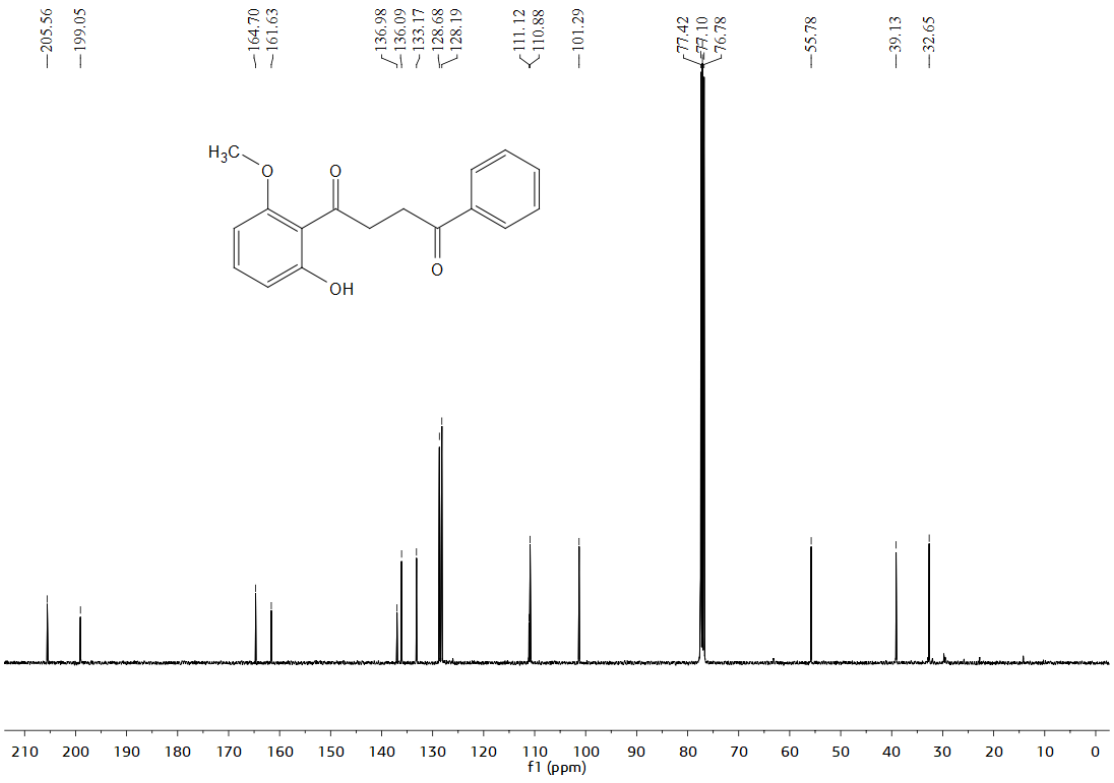


^{13}C NMR (100 MHz, $CDCl_3$) of **3da**

1-(2-hydroxy-6-methoxyphenyl)-4-phenylbutane-1,4-dione (3ea):

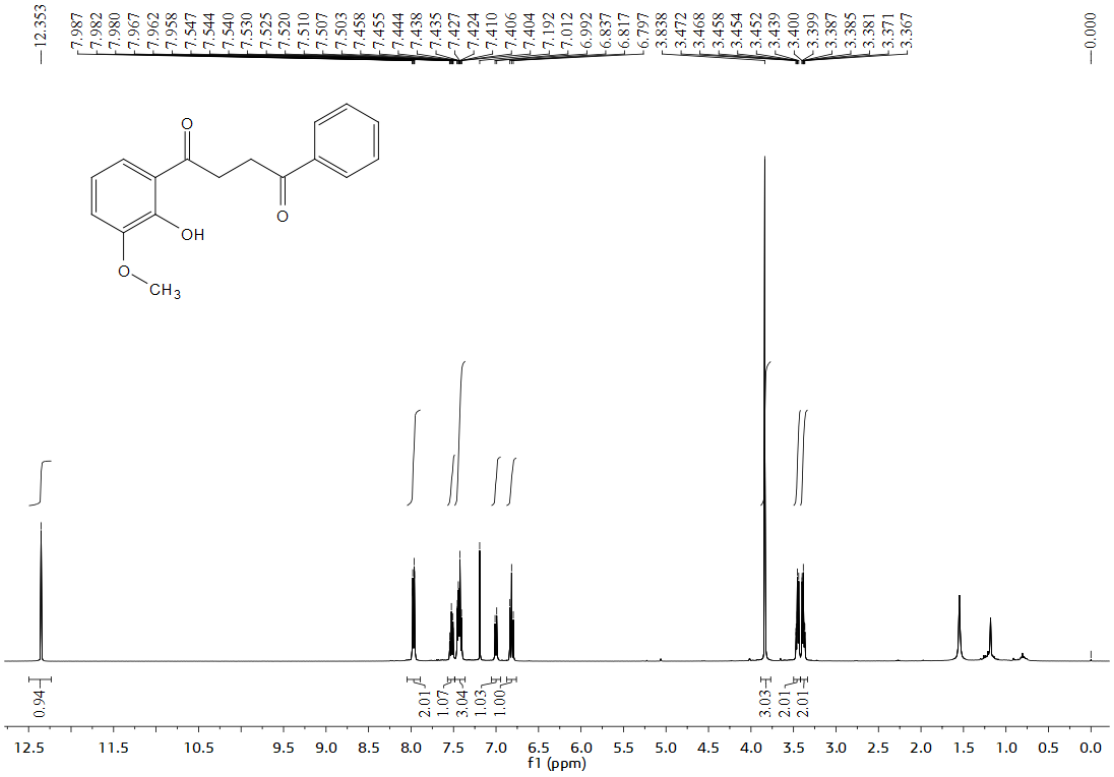


^{1}H NMR (400 MHz, $CDCl_3$) of **3ea**

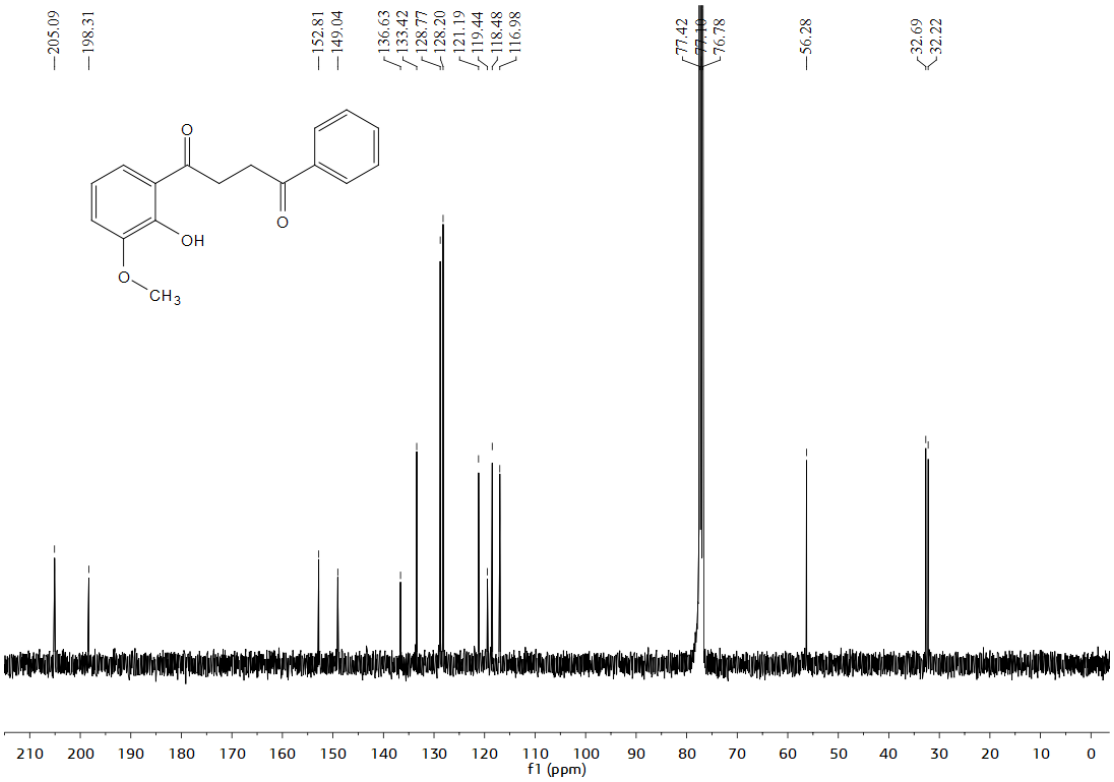


^{13}C NMR (100 MHz, $CDCl_3$) of **3ea**

1-(2-hydroxy-3-methoxyphenyl)-4-phenylbutane-1,4-dione (3fa):

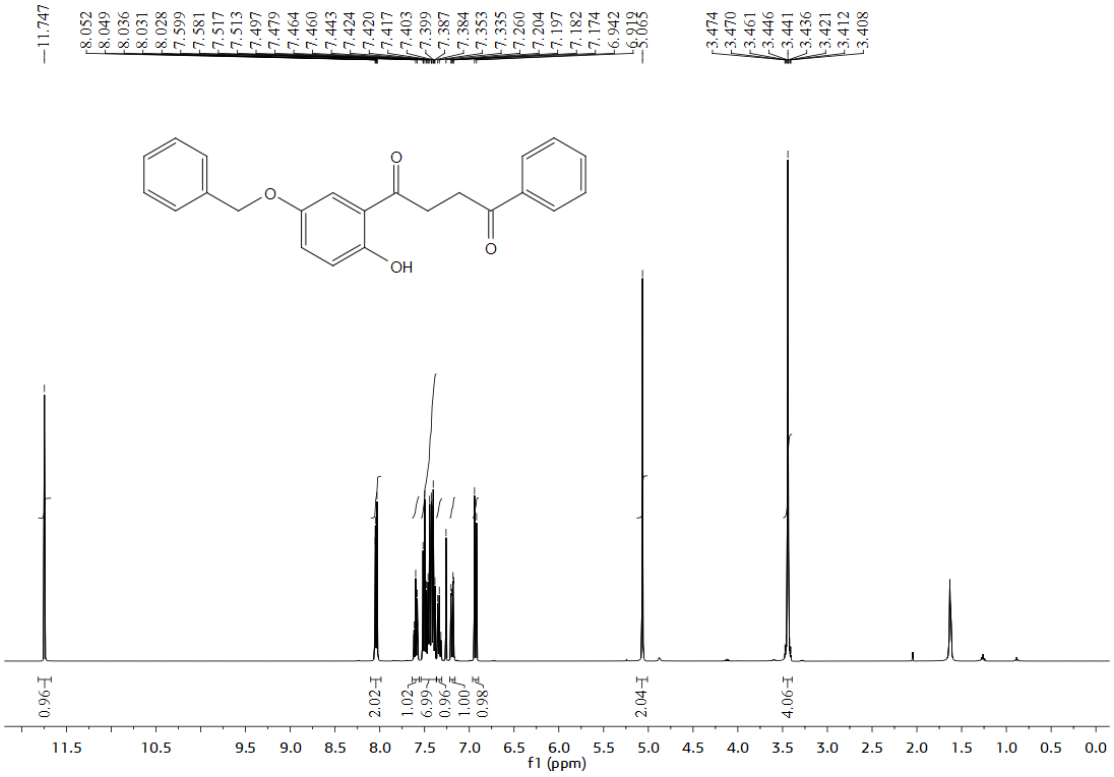


^{1}H NMR (400 MHz, $CDCl_3$) of **3fa**

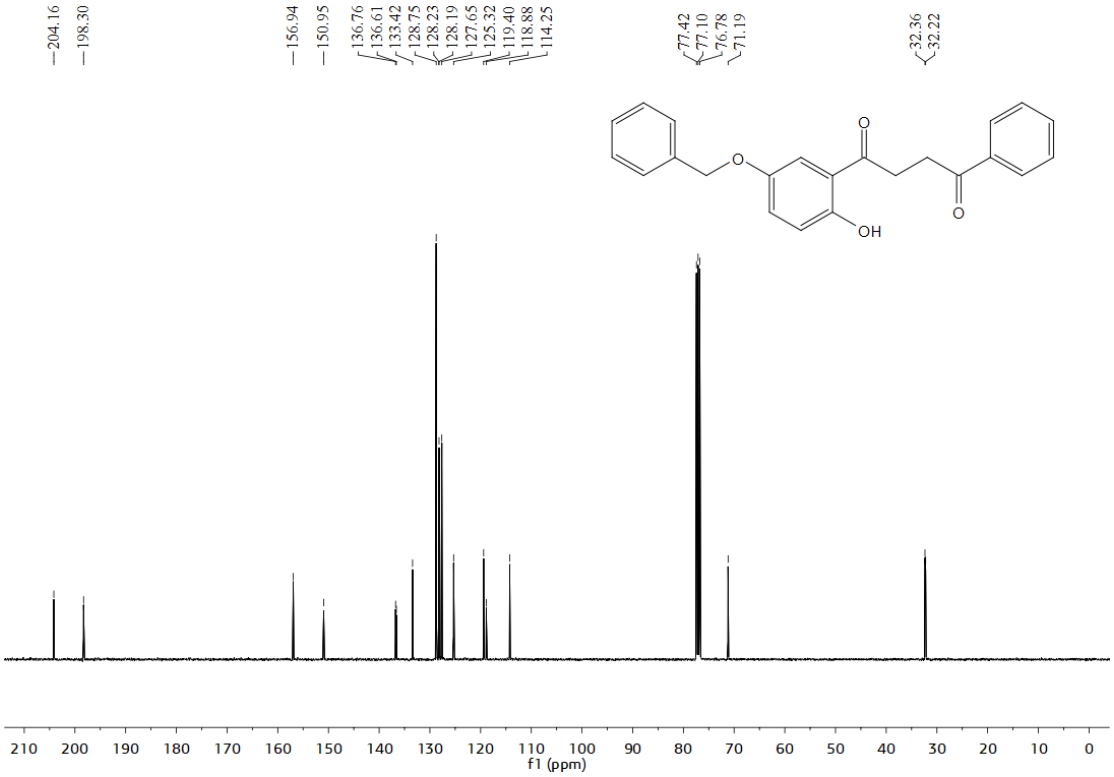


^{13}C NMR (100 MHz, $CDCl_3$) of **3fa**

1-(5-(benzyloxy)-2-hydroxyphenyl)-4-phenylbutane-1,4-dione (3ga):

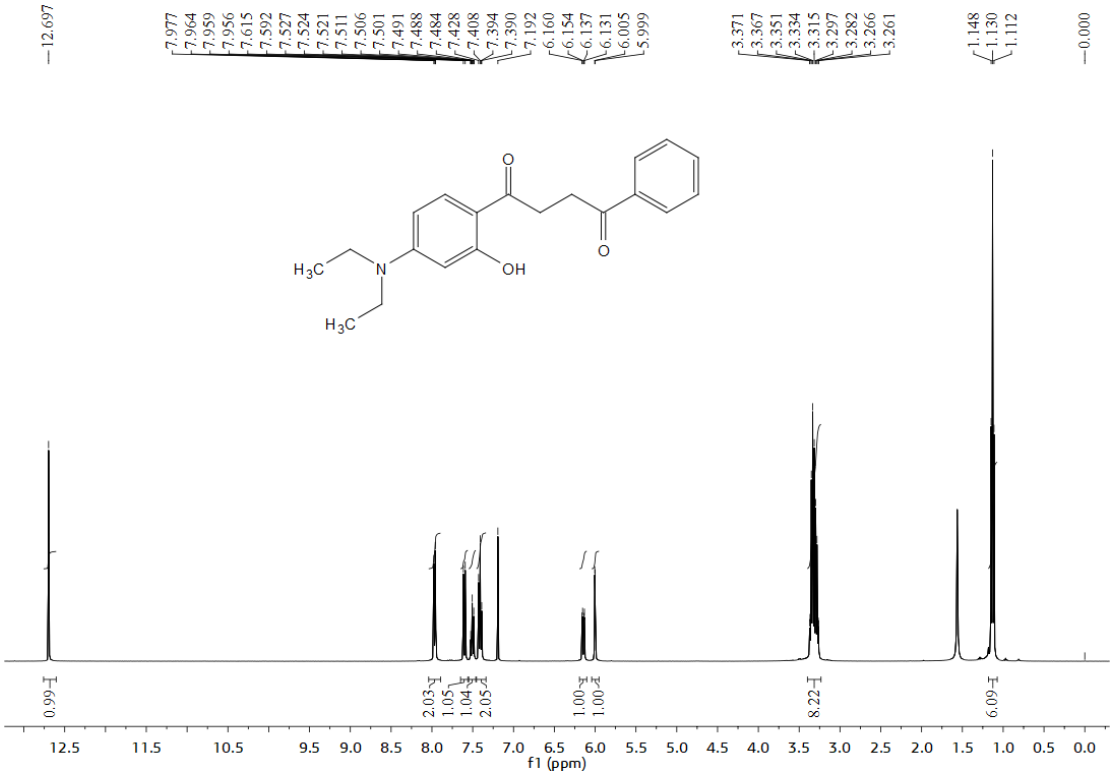


^{1}H NMR (400 MHz, $CDCl_3$) of **3ga**

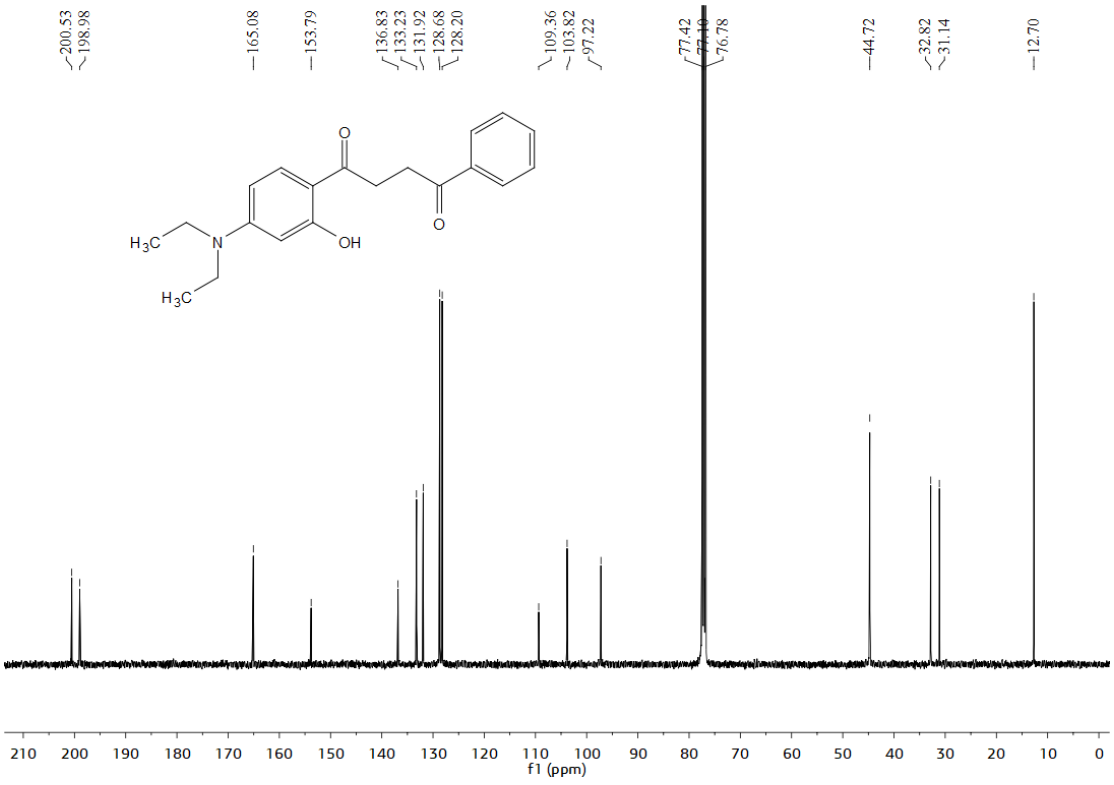


^{13}C NMR (100 MHz, $CDCl_3$) of **3ga**

1-(4-(diethylamino)-2-hydroxyphenyl)-4-phenylbutane-1,4-dione (3ha):

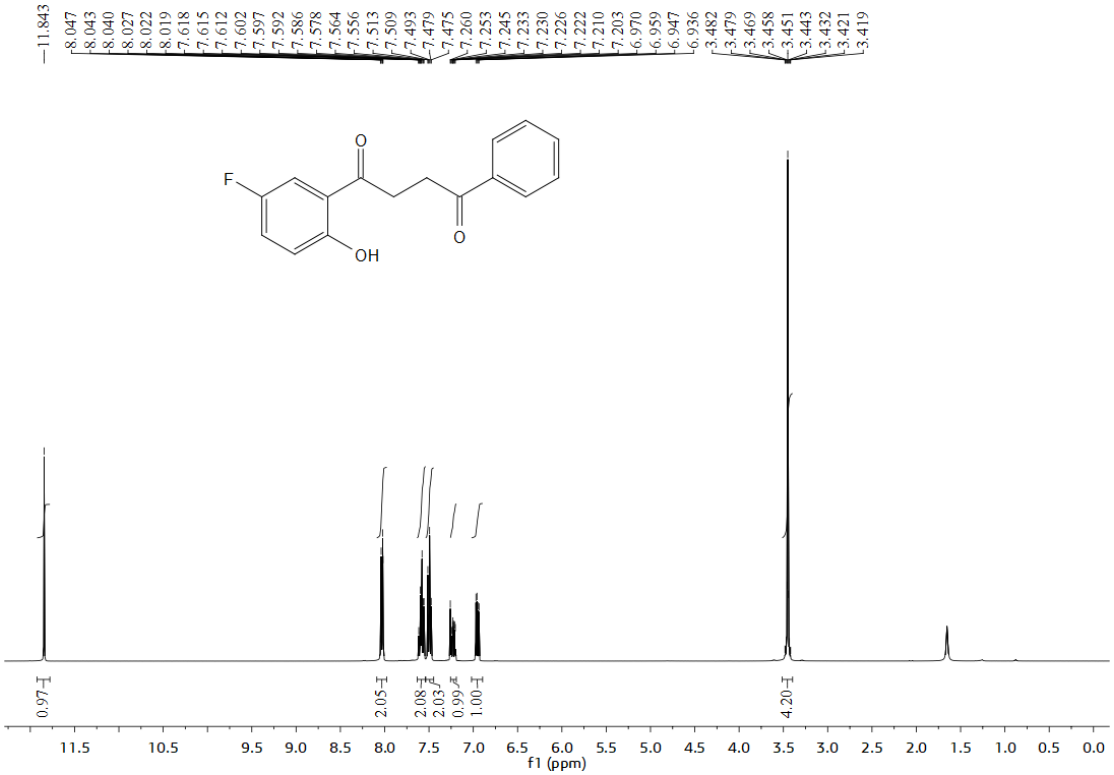


^{1}H NMR (400 MHz, $CDCl_3$) of **3ha**

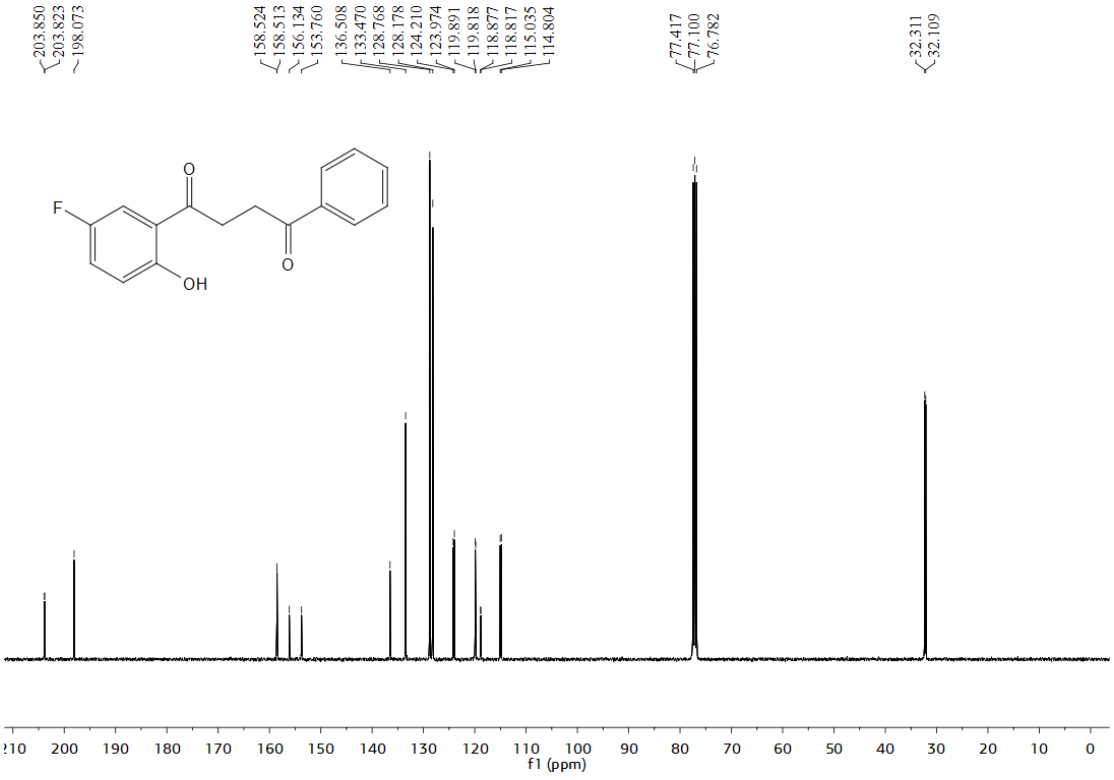


^{13}C NMR (100 MHz, $CDCl_3$) of **3ha**

1-(5-fluoro-2-hydroxyphenyl)-4-phenylbutane-1,4-dione (3ia):

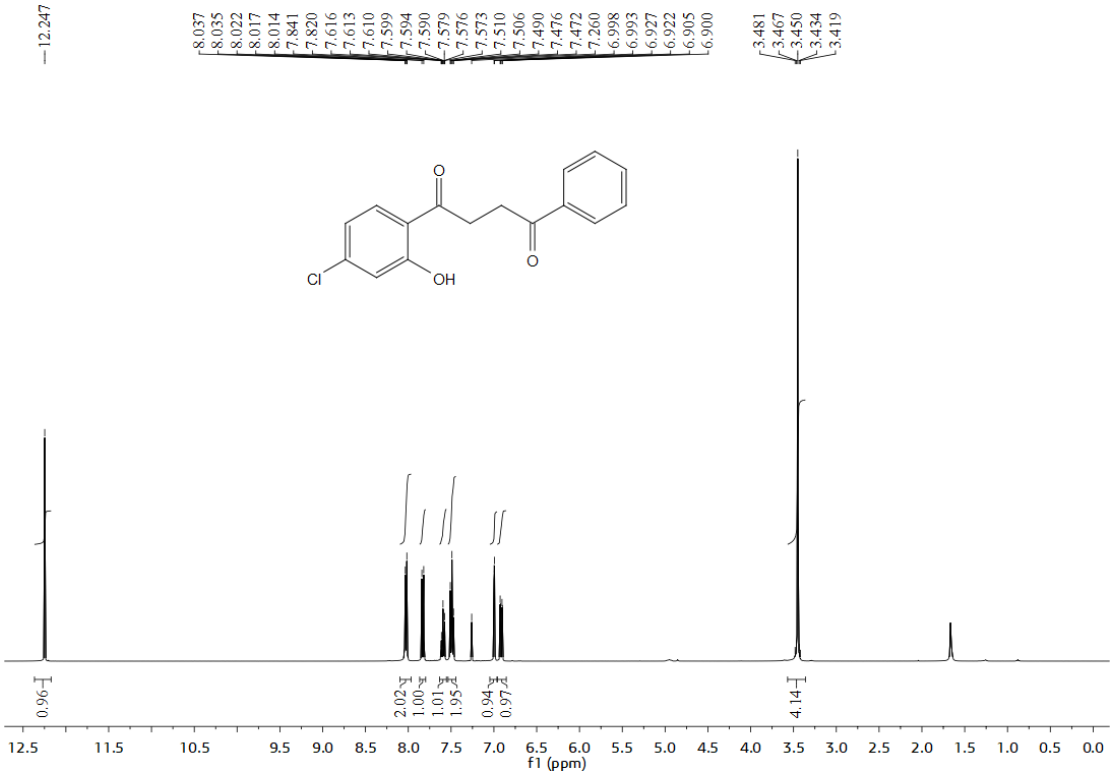


^{1}H NMR (400 MHz, $CDCl_3$) of **3ia**

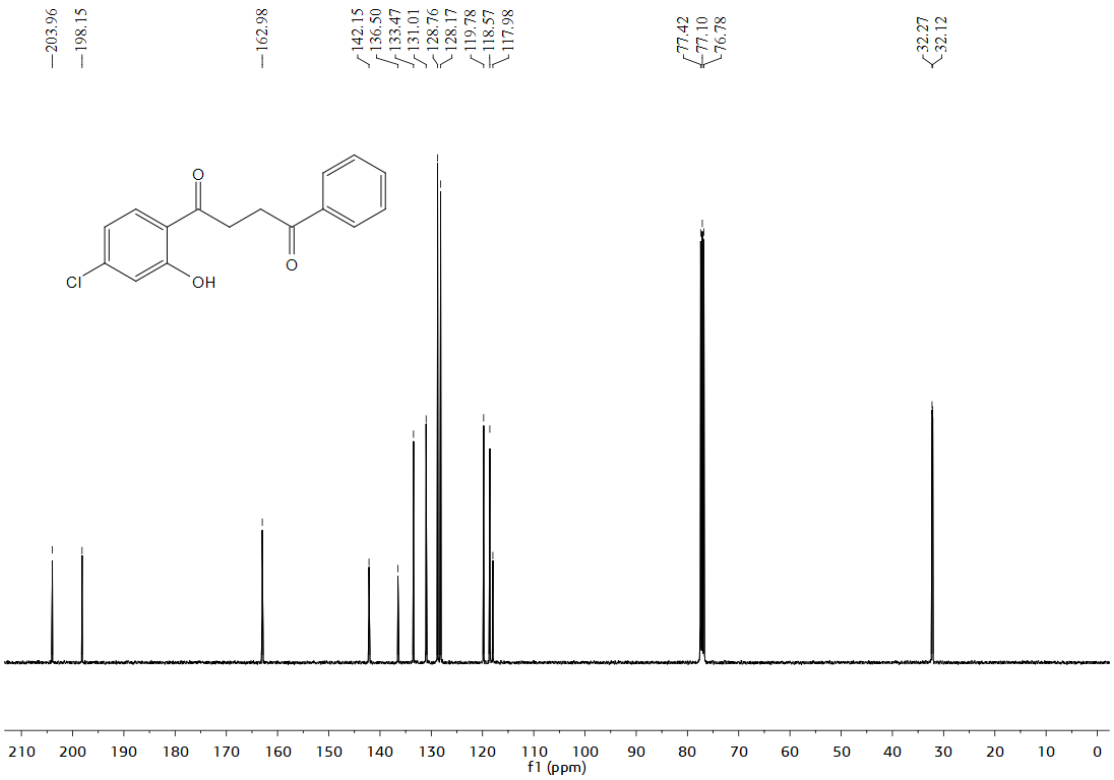


^{13}C NMR (100 MHz, $CDCl_3$) of **3ia**

1-(4-chloro-2-hydroxyphenyl)-4-phenylbutane-1,4-dione (3ja):

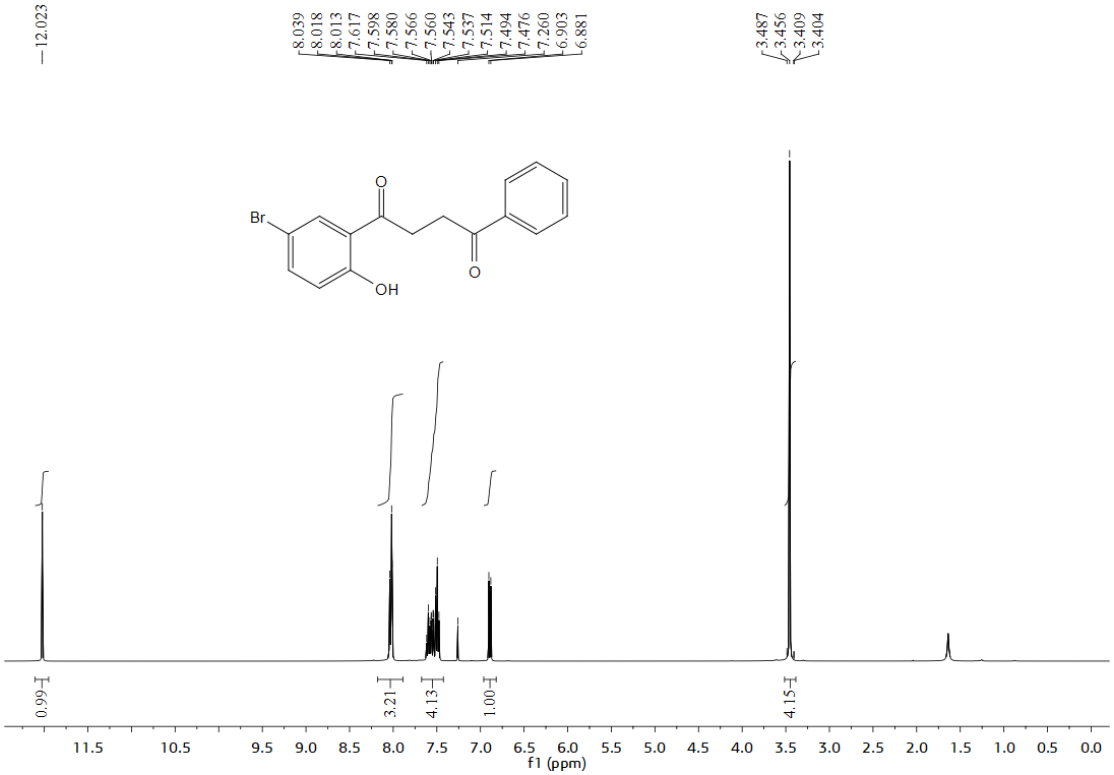


^{1}H NMR (400 MHz, $CDCl_3$) of **3ja**

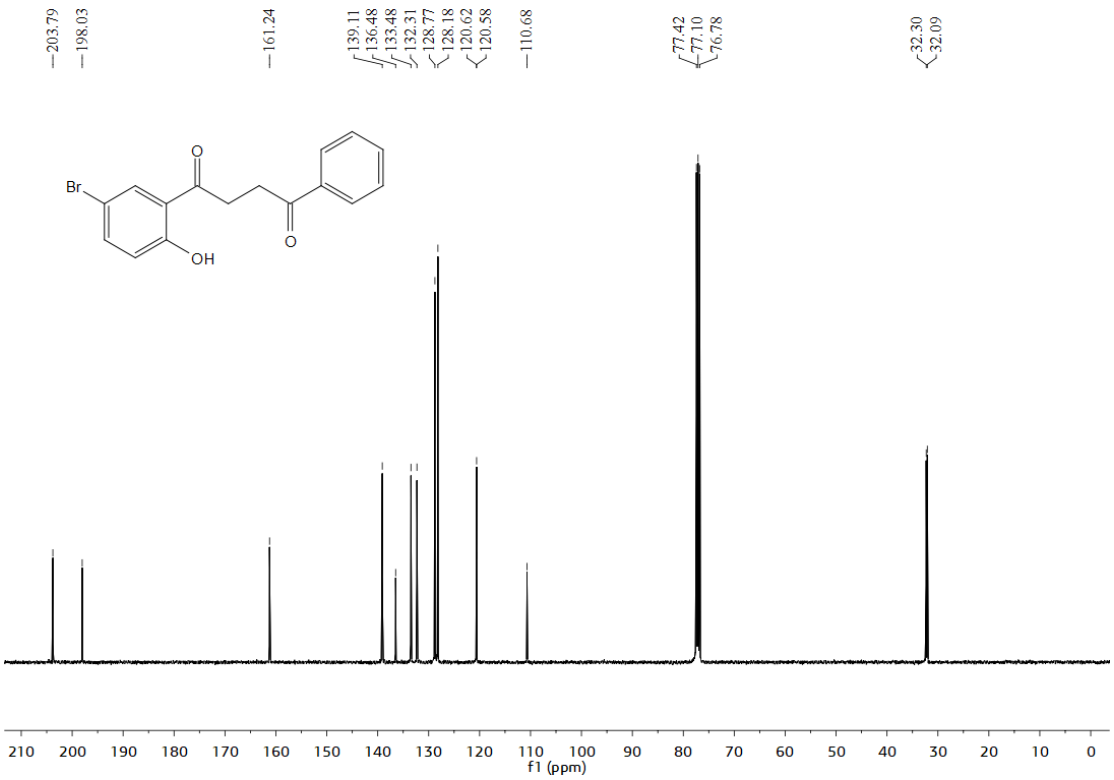


^{13}C NMR (100 MHz, $CDCl_3$) of **3ja**

1-(5-bromo-2-hydroxyphenyl)-4-phenylbutane-1,4-dione (3ka):

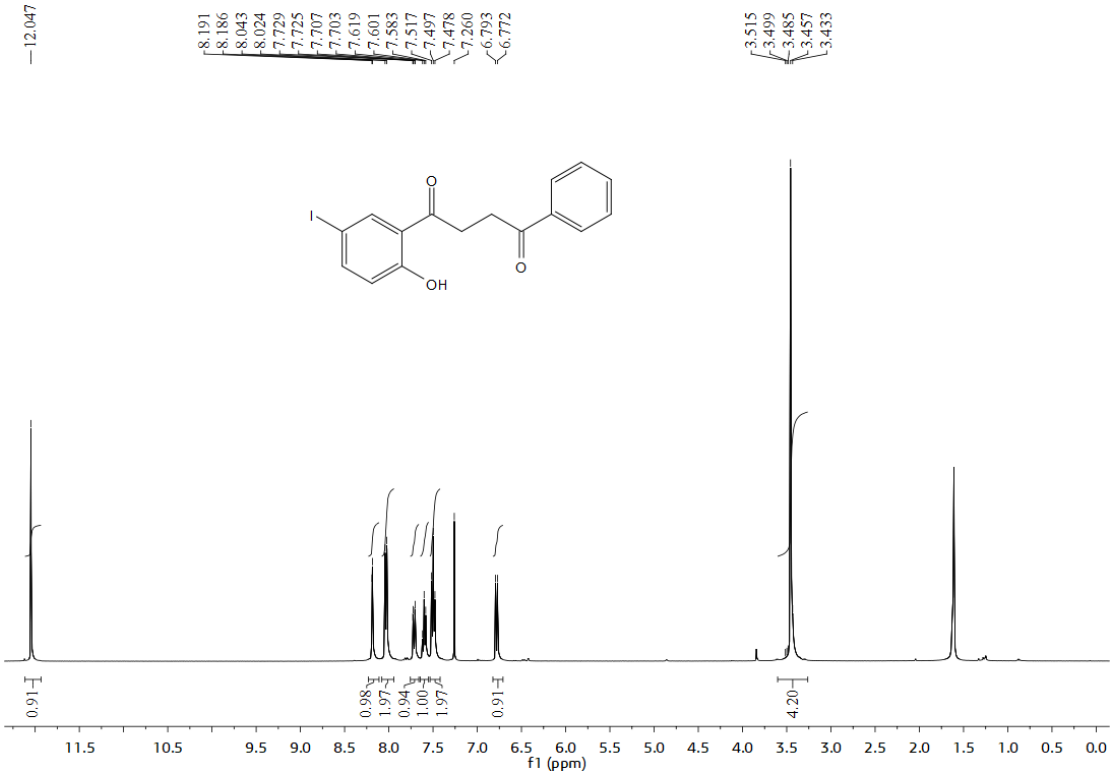


^{1}H NMR (400 MHz, $CDCl_3$) of **3ka**

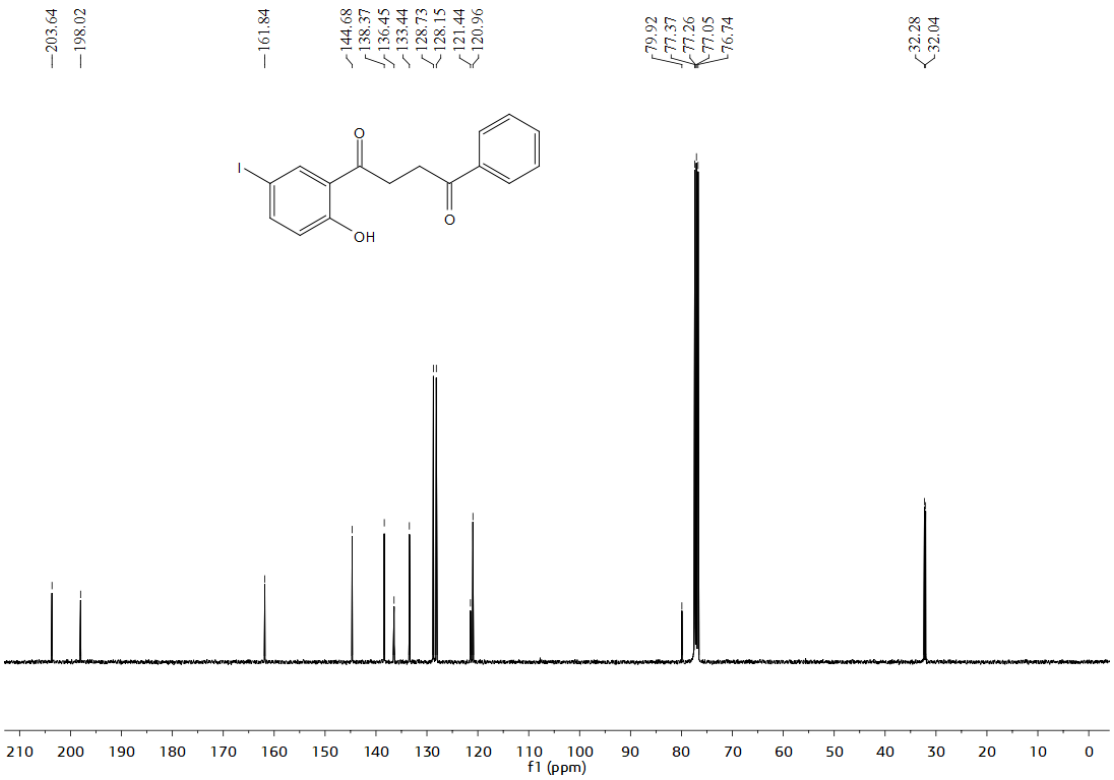


^{13}C NMR (100 MHz, $CDCl_3$) of **3ka**

1-(2-hydroxy-5-iodophenyl)-4-phenylbutane-1,4-dione (3la):

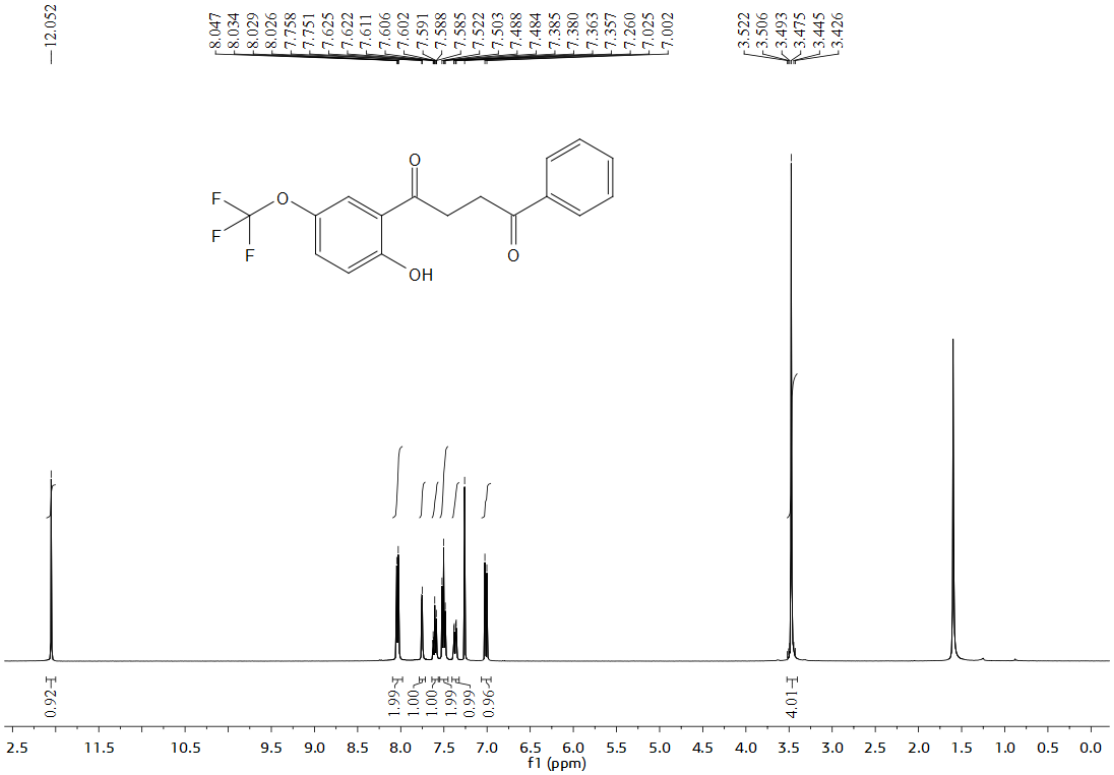


^{1}H NMR (400 MHz, $CDCl_3$) of **3la**

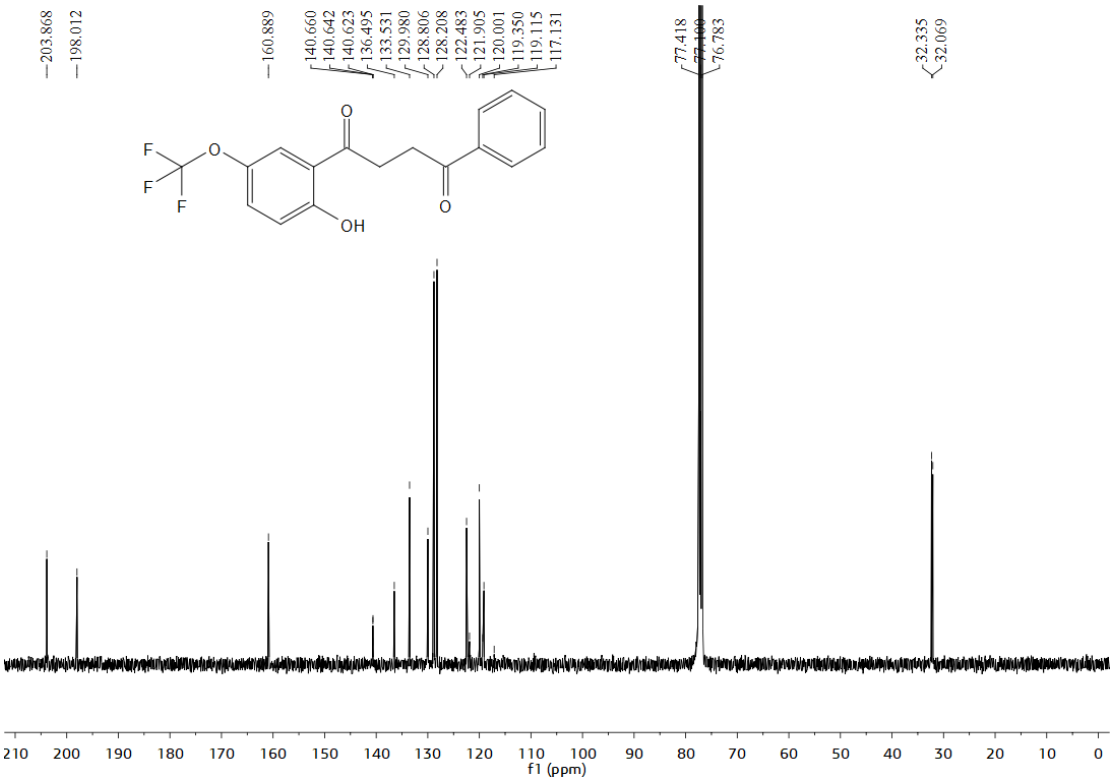


^{13}C NMR (100 MHz, $CDCl_3$) of **3la**

1-(2-hydroxy-5-(trifluoromethoxy)phenyl)-4-phenylbutane-1,4-dione (3ma):

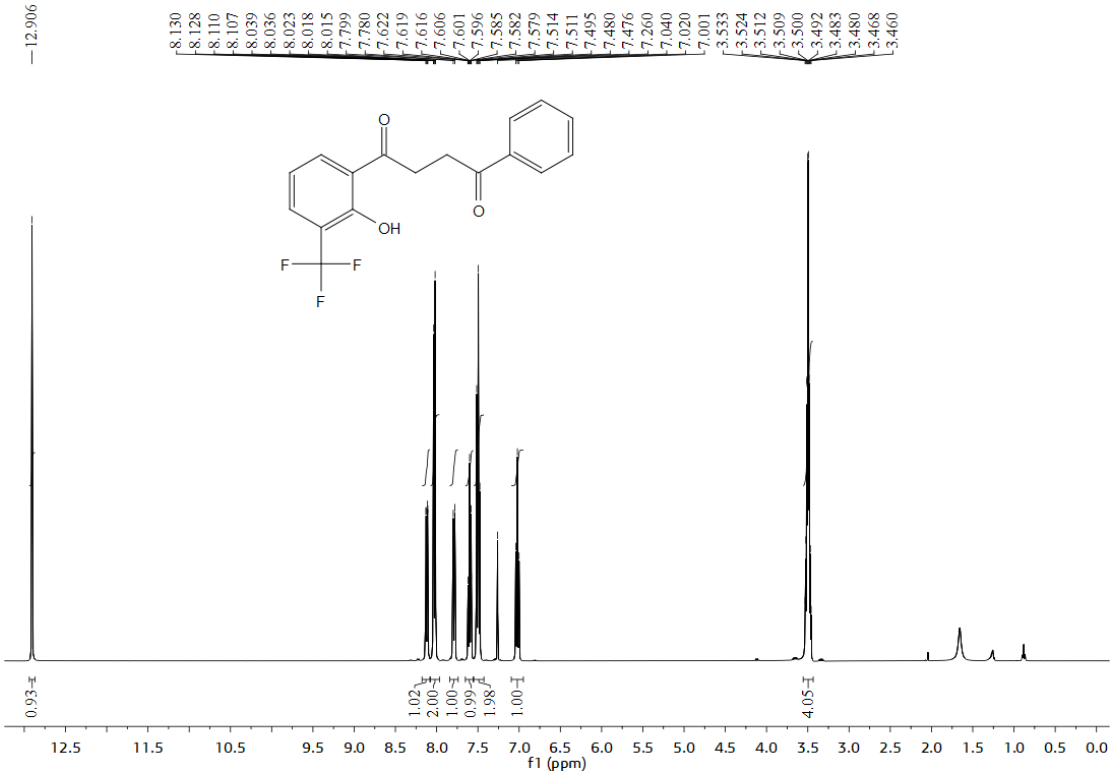


^{1}H NMR (400 MHz, $CDCl_3$) of **3ma**

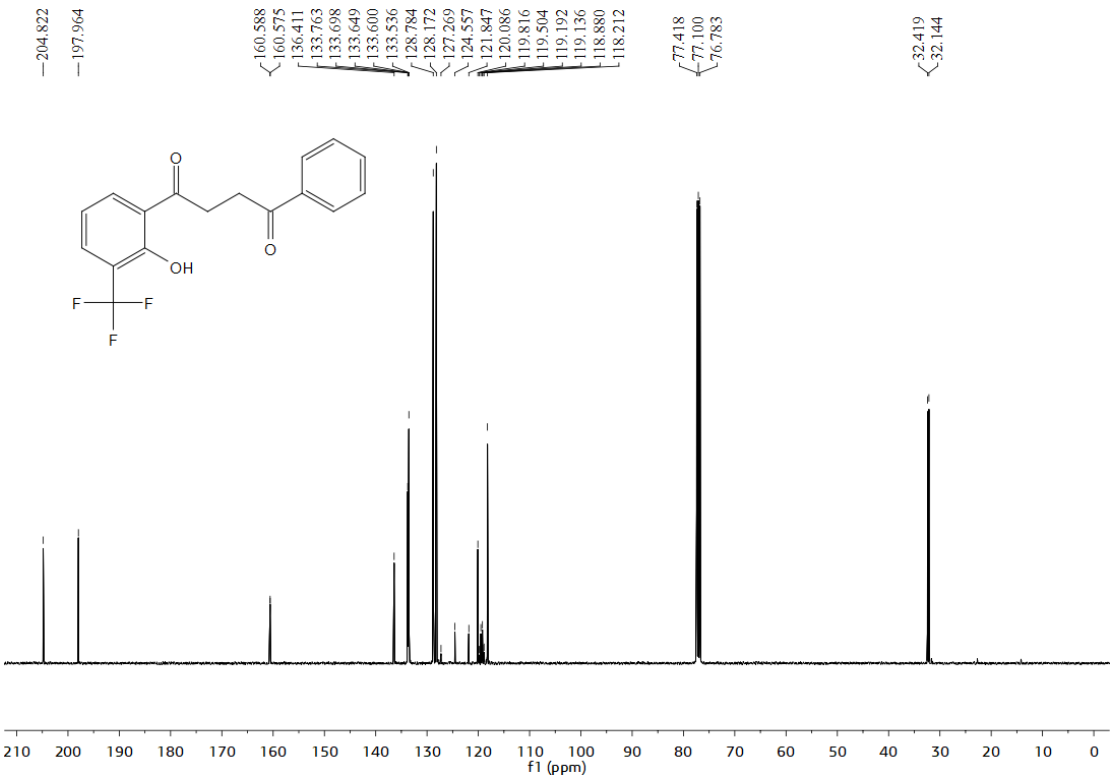


^{13}C NMR (100 MHz, $CDCl_3$) of **3ma**

1-(2-hydroxy-3-(trifluoromethyl)phenyl)-4-phenylbutane-1,4-dione (3na):

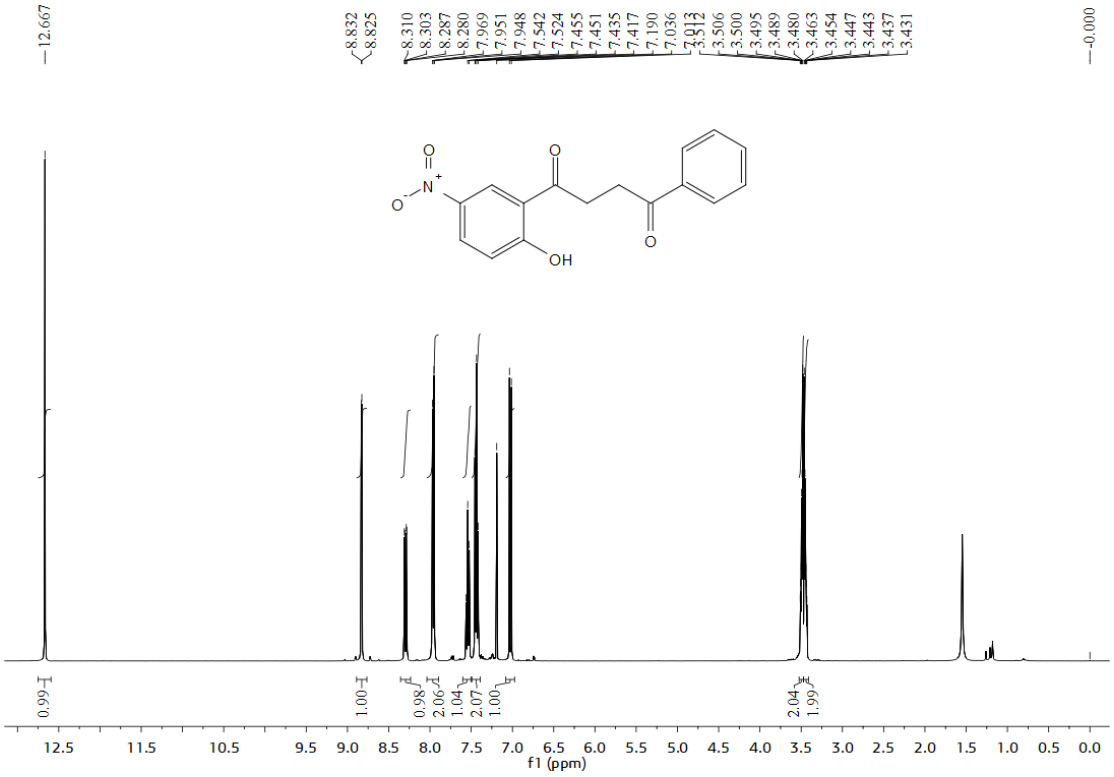


^{1}H NMR (400 MHz, $CDCl_3$) of **3na**

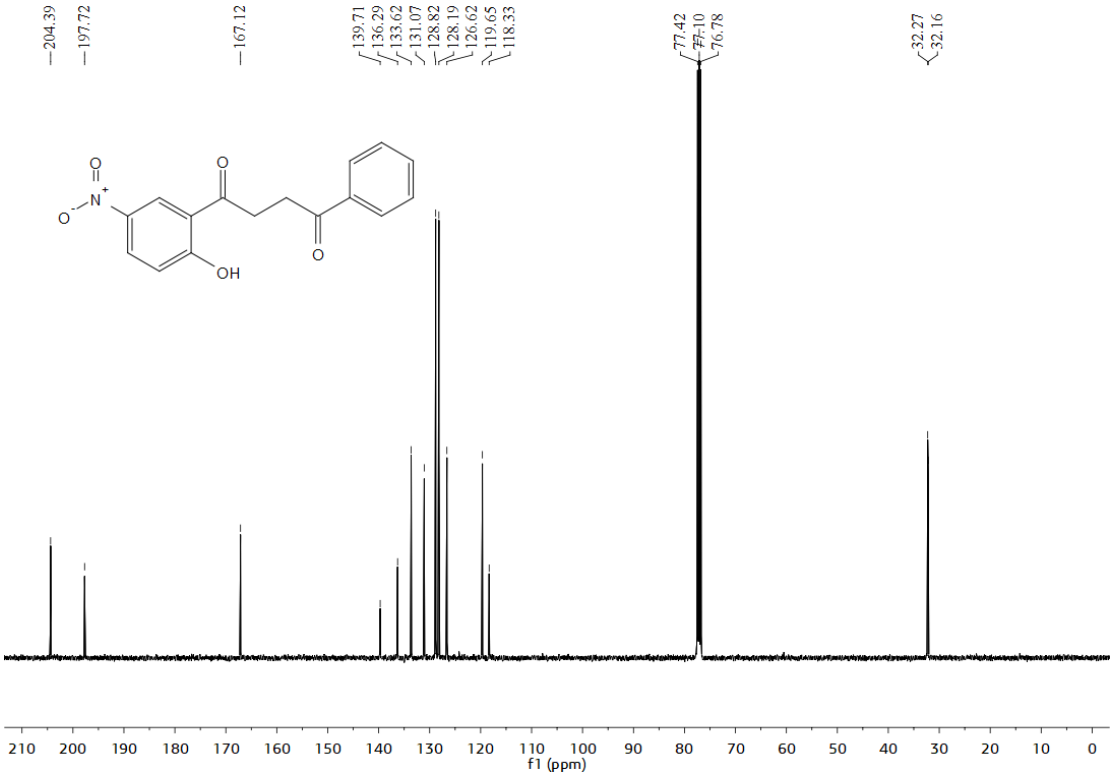


^{13}C NMR (100 MHz, $CDCl_3$) of **3na**

1-(2-hydroxy-5-nitrophenyl)-4-phenylbutane-1,4-dione (3oa):

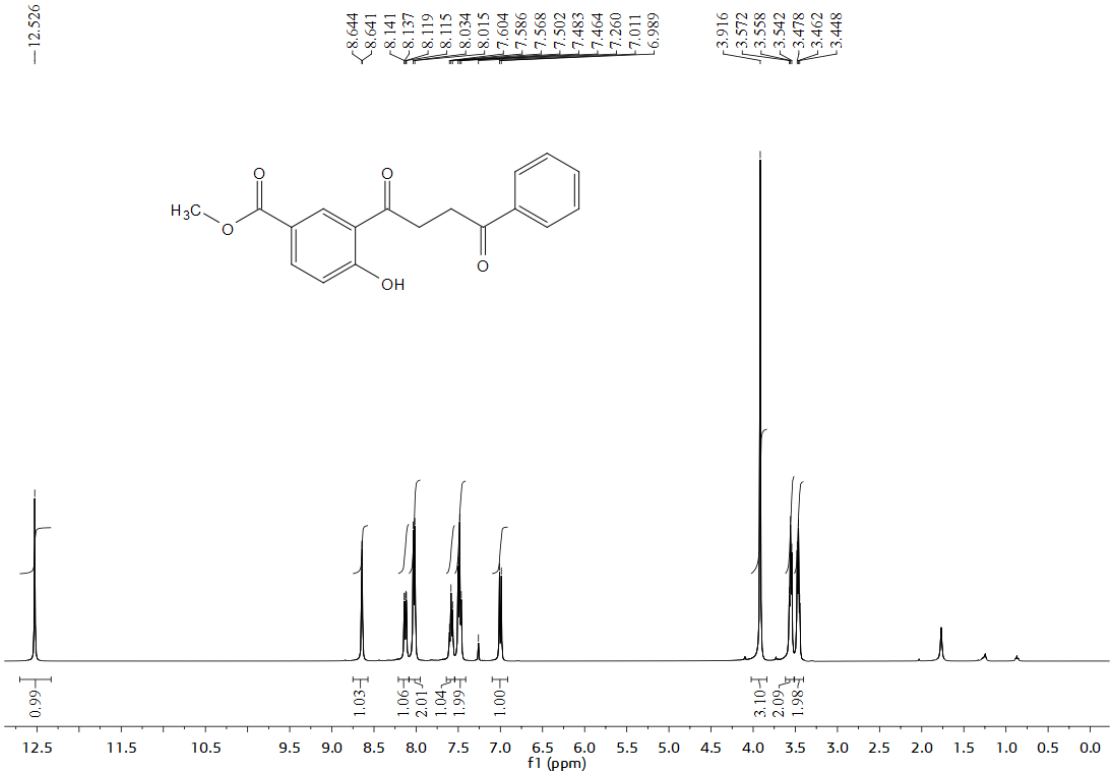


^{1}H NMR (400 MHz, $CDCl_3$) of **3oa**

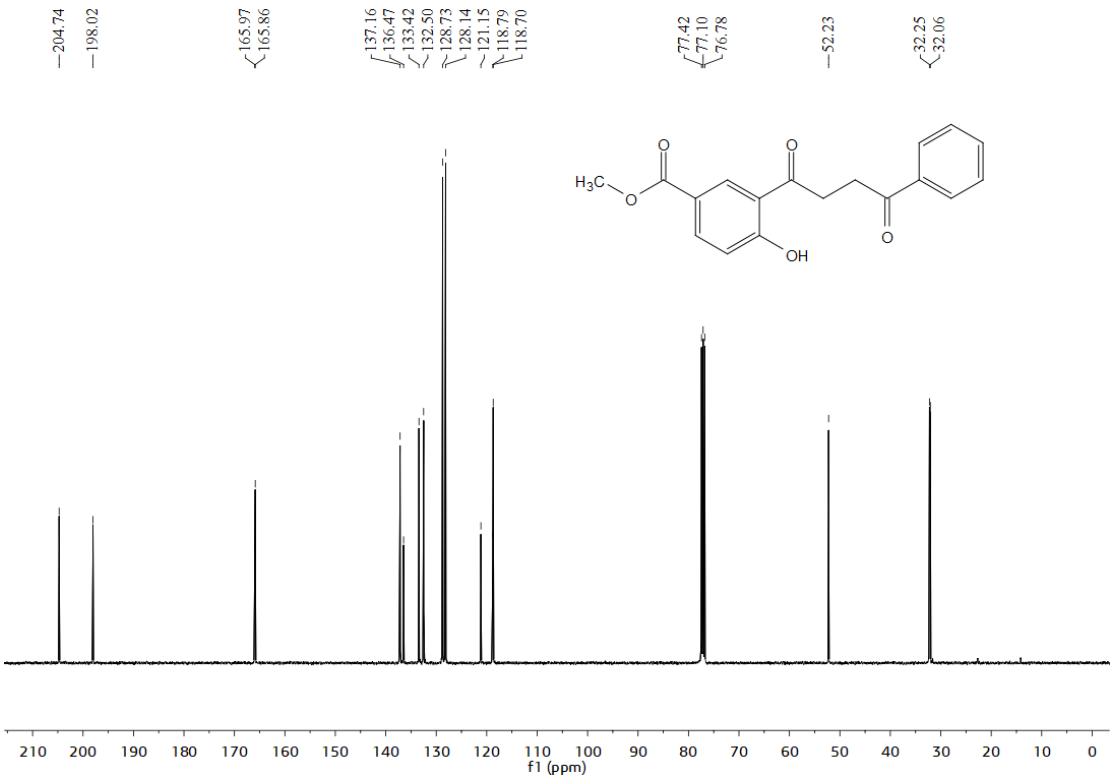


^{13}C NMR (100 MHz, $CDCl_3$) of **3oa**

methyl 4-hydroxy-3-(4-oxo-4-phenylbutanoyl)benzoate (3pa):



^{1}H NMR (400 MHz, $CDCl_3$) of **3pa**



^{13}C NMR (100 MHz, $CDCl_3$) of **3pa**