Intramolecular cyclization of diarylmethanols to \( \alpha,\beta \)-unsaturated amides promoted by KOt-Bu/DMF: a metal-free approach towards 3,4-disubstituted quinolinones

Jia-hua Chen, Zi-cong Chen, Hong Zhao, Yong Zou, Xue-jing Zhang* and Ming Yan*

Institute of Drug Synthesis and Pharmaceutical Process, School of Pharmaceutical Sciences, Sun Yat-sen University, Guangzhou 510006, China
E-mail: zhangxj33@mail.sysu.edu.cn; Fax/Tel: + 86-20-39943051

Supporting Information

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**General Information**

$^1$H NMR and $^{13}$C NMR spectra were recorded on Bruker AVANCE 400 spectrometer. Melting points were measured on a WRS-2A melting point apparatus and are uncorrected. Chemical shifts of protons are reported in parts per million downfield from tetramethylsilane and are referenced to residual protium in the NMR solvent (CDCl$_3$: $\delta$ 7.26; DMSO: $\delta$ 2.50). Chemical shifts of carbon are referenced to the carbon resonances of the solvent (CDCl$_3$: $\delta$ 77.0; DMSO: $\delta$ 39.5). Peaks are labeled as singlet (s), broad singlet (br), doublet (d), triplet (t), double doublet (dd), multiplet (m). Infrared spectra were recorded on a Nicolet 6700-Contium spectrometer. All products were further characterized by HRMS (high resolution mass spectra). Copies of their $^1$H NMR and $^{13}$C NMR spectra were provided. GC spectra were taken on an Agilent-6890A instrument. The normal and 99.99% purity of KO$_t$-Bu were purchased from J&K chemical company and Sigma-Aldrich chemical company respectively and used without further purification. DMF ($N,N$-dimethylformamide) and THF (tetrahydrofuran) were dried and redistilled according to standard methods. DMSO (dimethyl sulfoxide) was dried over 4Å molecular sieves.

**General procedures**

### 2.1 Synthesis of the starting materials$^{[1]}$

![Reagents and Step 1a](image)

To a solution of (2-aminophenyl)(phenyl)methanol **S1** (398.5 mg, 2.0 mmol) and sodium bicarbonate (504.0 mg, 6.0 mmol) in dry DCM (20 mL) was added a solution of acryloyl chloride (199.1 mg, 2.2 mmol) in dry DCM (2 mL) dropwise at 0°C. The mixture was allowed to stir at this temperature for 2 hours. Upon completion (judged by TLC), water (20 mL) was added to quench the reaction. The organic phase was separated and evaporated to dryness. The residue was purified by column chromatography (petroleum ether/ethyl acetate = 5:2) to give **1a** (480.0 mg, 95% yield).

The diphenylmethanol **S1** was synthesized according to known literatures.$^{[2]}$

### 2.2 Representative experiment procedure

![Reagents and Step 1b](image)

To a 25 mL dried reaction tube was added **1a** (63.4 mg, 0.25 mmol), KO$_t$-Bu (112.3 mg, 1.0 mmol) and dry DMF (2.5 mL). The mixture was stirred at 100°C for 2 hours under argon atmosphere. After cooled down to room temperature, the reaction mixture was quenched with saturated NH$_4$Cl solution (1 mL) and was extracted with ethyl acetate (10 mL × 3). The combined organic layers was then washed with brine (5 mL) and dried over anhydrous MgSO$_4$. The residue was purified by column chromatography (petroleum ether/ethyl acetate = 10:3) to give **2a** as a white solid (53.1 mg, 90% yield).

### 2.3 Gram-scale experiment
To a 100 mL dried flask was added the mixture of 1a (2.351 g, 10.0 mol), KOt-Bu (4.492 g, 40.0 mmol) and dry DMF (100.0 mL). The mixture was stirred at 100°C for 2 hours under argon atmosphere. After cooled down to room temperature, the mixture was poured into 500 mL ice water and stirred for 5 minutes. The precipitated solid was collected by filtration and further purified by recrystallization (petroleum ether/ethyl acetate) to afford 2a as a pale solid (2.139 g, 91% yield).

2.4 Synthesis of 5a

Under argon atmosphere, NaH (60.0 mg, 1.5 mmol, 60% in mineral) was added to a mixture of 2a (235.3 mg, 1.0 mmol) in dry DMF (10 mL) at 0°C in portions and stirred at this temperature for 2 hours. BnBr (205.3 mg, 1.2 mmol) was added to the mixture slowly and then warm up to room temperature. After 10 hours, the mixture was quenched with saturated NH₄Cl solution (5 mL) and was extracted with ethyl acetate (10 mL × 3). The combined organic layers was then washed with brine (5 mL) and dried over anhydrous MgSO₄. The residue was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to give 5a as a white solid (289.2 mg, 89% yield).

2.5 Synthesis of 5b

A mixture of 2a (235.3 mg, 1.0 mmol) and 5 mL freshly distilled POCl₃ was refluxed for 5 hours. The mixture was poured into ice water (50 mL). Saturated Na₂CO₃ was added to adjust pH=7. The mixture was extracted with ethyl acetate (10 mL × 3). The combined organic layers was then washed with brine (5 mL) and dried over anhydrous MgSO₄. The residue was purified by column chromatography (petroleum ether/ethyl acetate = 100:1) to give 5b as a white solid (233.4 mg, 92% yield).

2.6 Synthesis of 5c

A mixture of 2a (235.3 mg, 1.0 mmol), N-bromosuccinimide (267.0 mg, 1.5 mmol), azodiisobutyronitrile (33.0 mg, 0.2 mmol) in CCl₄ (10 mL) was heated at 90°C for 7 hours. The mixture was washed with water (20 mL) and brine (5 mL). The organic layer was dried over anhydrous MgSO₄. The residue was purified by column chromatography (petroleum ether/EtOAc = 10:1) to give 5c as a white solid (265.8 mg, 85% yield).
3. Characterization data

*N-(2-(hydroxy(phenyl)methyl)phenyl)acrylamide (1a)*

![Chemical structure of 1a]

White solid. M.p.: 110.1–113.5°C. $^1$H NMR (400 MHz, CDCl$_3$) δ 9.06 (br, 1H), 8.03 (d, $J = 8.2$ Hz, 1H), 7.33–7.17 (m, 6H), 7.06–6.98 (m, 2H), 6.10 (d, $J = 17.0$ Hz, 1H), 5.97 (dd, $J = 17.0$, 10.1 Hz, 1H), 5.80 (s, 1H), 5.55 (d, $J = 10.1$ Hz, 1H), 4.49 (br, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 164.0, 141.5, 136.5, 132.5, 131.6, 128.9, 128.7, 128.5, 127.6, 127.1, 126.2, 124.4, 122.9, 75.3. IR (thin film): $\nu$(cm$^{-1}$) = 3344, 3265, 3054, 1662, 1627, 1583, 1533, 1444, 1400, 1018(s), 758. HRMS (ESI) calculated for C$_{16}$H$_{15}$NO$_2$Na [M+Na]$^+$: 276.0995, found: 276.0984.

*N-(2-(hydroxy(o-tolyl)methyl)phenyl)acrylamide (1b)*

![Chemical structure of 1b]

White solid. M.p.: 136.2–138.1°C. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.92 (br, 1H), 8.03 (d, $J = 8.2$ Hz, 1H), 7.44 (dd, $J = 5.4$, 4.0 Hz, 1H), 7.31–7.21 (m, 3H), 7.19–7.15 (m, 1H), 7.01 (t, $J = 7.7$ Hz, 1H), 6.80 (d, $J = 7.8$ Hz, 1H), 6.31 (d, $J = 16.9$ Hz, 1H), 6.18 (dd, $J = 16.9$, 10.0 Hz, 1H), 6.02 (d, $J = 2.1$ Hz, 1H), 5.70 (d, $J = 10.2$ Hz, 1H), 3.54 (br, 1H), 2.13 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 164.2, 139.1, 136.7, 135.8, 132.0, 131.6, 130.7, 128.8, 128.3, 128.0, 127.5, 126.4, 126.3, 124.8, 123.2, 71.7, 19.4. IR (thin film): $\nu$(cm$^{-1}$) = 3465, 3237, 3018, 1643, 1591, 1517, 144, 1400, 1022, 767, 744. HRMS (ESI) calculated for C$_{17}$H$_{17}$NO$_2$Na [M+Na]$^+$: 290.1152, found: 290.1149.

*N-(2-(hydroxy(m-tolyl)methyl)phenyl)acrylamide (1c)*

![Chemical structure of 1c]

White solid. M.p.: 97.8–98.4°C. $^1$H NMR (400 MHz, CDCl$_3$) δ 8.84 (br, 1H), 8.13 (d, $J = 8.3$ Hz, 1H), 7.33–7.27 (m, 1H), 7.20 (t, $J = 7.7$ Hz, 1H), 7.15–7.03 (m, 5H), 6.18 (dd, $J = 17.0$, 0.8 Hz, 1H), 6.05 (dd, $J = 17.0$, 10.2 Hz, 1H), 5.85 (d, $J = 3.1$ Hz, 1H), 5.62 (dd, $J = 10.2$, 1.2 Hz, 1H), 3.50 (br, 1H), 2.30 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 163.8, 141.2, 138.2, 136.6, 132.1, 131.8, 128.8, 128.6, 128.5(0), 128.4(5), 126.9, 126.8, 124.3, 123.3, 122.9, 75.6, 21.5. IR (thin film): $\nu$(cm$^{-1}$) = 3347, 3052 , 2873, 1664, 1604, 1591, 1517, 144, 1400, 1270, 755. HRMS (ESI) calculated for C$_{17}$H$_{17}$NO$_2$Na [M+Na]$^+$: 290.1152, found: 290.1146.

*N-(2-(hydroxy(mesityl)methyl)phenyl)acrylamide (1d)*

![Chemical structure of 1d]

Vicious oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 9.55 (br, 1H), 8.33 (d, $J = 8.3$ Hz, 1H), 7.30 (d, $J = 7.8$ Hz, 1H), 6.97–6.90
(m, 3H), 6.71 (d, J = 7.9 Hz, 1H), 6.43–6.37 (m, 2H), 6.26 (dd, J = 17.0, 10.2 Hz, 1H), 5.75 (d, J = 10.2 Hz, 1H), 2.41 (br, 1H), 2.32 (s, 3H), 2.25 (s, 6H). $^1$C NMR (100 MHz, CDCl$_3$) $\delta$ 164.0, 137.6, 137.4, 137.3, 134.3, 132.2, 130.2, 130.0, 128.1, 127.4, 126.7, 124.0, 122.1, 71.1, 20.9, 20.8. IR (thin film): $\nu$(cm$^{-1}$) 3380, 3237, 3025, 2925, 2848, 1672, 1585, 1521, 1444, 1317, 1039, 755. HRMS (ESI) calculated for C$_{19}$H$_{21}$NO$_2$Na $[M+Na]^+$: 318.1465, found: 318.1461.

N-(2-((4-(tert-butyl)phenyl)(hydroxy)methyl)phenyl)acrylamide (1e)

White solid. M.p.: 156.2–159.3°C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.87 (s, 1H), 8.20 (d, $J = 8.3$ Hz, 1H), 7.36 – 7.29 (m, 3H), 7.23 (d, $J = 8.3$ Hz, 2H), 7.12 – 7.03 (m, 2H), 6.18 (d, $J = 16.8$ Hz, 1H), 6.05 (dd, $J = 17.0$, 10.1 Hz, 1H), 5.62 (d, $J = 10.2$ Hz, 1H), 3.21 (br, 1H), 1.29 (s, 9H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 163.7, 150.9, 138.1, 136.7, 131.9, 131.8, 128.8(4), 128.7(7), 126.8, 126.0, 125.5, 124.2, 122.8, 75.7, 34.6, 31.3. IR (thin film): $\nu$(cm$^{-1}$) 3334, 3230, 3072, 2960, 2942, 1662, 1610, 1585, 1403, 1332, 1288, 757. HRMS (ESI) calculated for C$_{20}$H$_{23}$NO$_2$Na $[M+Na]^+$: 332.1621, found: 332.1614.

N-(2-(hydroxy(2-methoxyphenyl)methyl)phenyl)acrylamide (1f)

White solid. M.p.: 146.3–149.2°C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 9.34 (br, 1H), 8.29 (d, $J = 8.3$ Hz, 1H), 7.31 (m, 2H), 7.07–6.89 (m, 5H), 6.27 (d, $J = 16.9$ Hz, 1H), 6.21–6.08 (m, 2H), 5.65 (d, $J = 10.0$ Hz, 1H), 4.05 (br, 1H), 3.87 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 163.7, 156.9, 137.3, 132.2, 130.2, 129.4, 128.9, 128.6, 128.3, 128.2, 126.6, 124.0, 122.5, 121.3, 110.8, 72.8, 55.6. IR (thin film): 3407(s), 3367, 2991, 2908, 2832, 1672, 1631, 1585, 1452, 1405, 1284, 1045, 754. HRMS (ESI) calculated for C$_{17}$H$_{17}$NO$_3$Na $[M+Na]^+$: 306.1101, found: 306.1095.

N-(2-((4-fluorophenyl)(hydroxy)methyl)phenyl)acrylamide (1g)

White solid. M.p.: 96.0–100.3°C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.97 (s, 1H), 8.02 (d, $J = 8.3$ Hz, 1H), 7.29–7.18 (m, 3H), 7.09–7.01 (m, 2H), 6.95 (d, $J = 8.3$ Hz, 2H), 6.13 (d, $J = 17.0$ Hz, 1H), 5.99 (dd, $J = 16.9$, 10.3 Hz, 1H), 5.81 (s, 1H), 5.59 (d, $J = 10.2$ Hz, 1H), 4.31 (s, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 164.0, 162.1 ($^{19}$C$_F$ = 244.2 Hz), 137.2 ($^{19}$C$_F$ = 3.0 Hz), 136.4, 132.3, 131.5, 128.9, 127.9 ($^{19}$C$_F$ = 8.0 Hz), 127.1, 124.6, 123.2, 115.3 ($^{19}$C$_F$ = 21.2 Hz), 74.8. $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -114.87. IR (thin film): $\nu$(cm$^{-1}$) 3365, 3293, 3072, 2875, 2832, 1668, 1608, 1572, 1452, 1405, 1222, 1060, 767. HRMS (ESI) calculated for C$_{16}$H$_{14}$NO$_2$FNa $[M+Na]^+$: 294.0901, found: 294.0893.

N-(2-((3,5-bis(trifluoromethyl)phenyl)(hydroxy)methyl)phenyl)acrylamide (1h)

White solid. M.p.: 96.0–100.3°C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.97 (s, 1H), 8.02 (d, $J = 8.3$ Hz, 1H), 7.29–7.18 (m, 3H), 7.09–7.01 (m, 2H), 6.95 (d, $J = 8.3$ Hz, 2H), 6.13 (d, $J = 17.0$ Hz, 1H), 5.99 (dd, $J = 16.9$, 10.3 Hz, 1H), 5.81 (s, 1H), 5.59 (d, $J = 10.2$ Hz, 1H), 4.31 (s, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 164.0, 162.1 ($^{19}$C$_F$ = 244.2 Hz), 137.2 ($^{19}$C$_F$ = 3.0 Hz), 136.4, 132.3, 131.5, 128.9, 127.9 ($^{19}$C$_F$ = 8.0 Hz), 127.1, 124.6, 123.2, 115.3 ($^{19}$C$_F$ = 21.2 Hz), 74.8. $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -114.87. IR (thin film): $\nu$(cm$^{-1}$) 3365, 3293, 3072, 2875, 2832, 1668, 1608, 1572, 1452, 1405, 1222, 1060, 767. HRMS (ESI) calculated for C$_{18}$H$_{14}$NO$_2$FNa $[M+Na]^+$: 294.0901, found: 294.0893.
White solid. M.p.: 94.6–100.5°C. \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) 8.49 (br, 1H), 7.84 (d, \(J = 8.2\) Hz, 1H), 7.77 (s, 3H), 7.33 (dd, \(J = 9.6, 1.8\) Hz, 1H), 7.22–7.11 (m, 2H), 6.21 (dd, \(J = 2.4\) Hz, 1H), 5.68 (dd, \(J = 10.2, 0.8\) Hz, 1H), 4.35 (br, 1H).

\textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta\) 164.4, 144.4, 135.8, 132.7, 131.6 (q, \(J = 34.0\) Hz), 130.7, 129.7, 129.2, 127.9, 126.1, 125.6, 124.6, 124.4, 121.9, 121.4 74.0.

\textsuperscript{19}F NMR (376 MHz, CDCl\textsubscript{3}) \(\delta\) -62.9.

IR (thin film): \(\nu(\text{cm}^{-1})\) 3344, 3072, 3089, 3060, 1673, 1612, 1519, 1450, 1278, 1128, 754.

HRMS (ESI) calculated for C\textsubscript{18}H\textsubscript{13}NO\textsubscript{2}F\textsubscript{6}Na \([\text{M+Na}]^+\): 412.0743, found: 412.0735.

\textit{N-(2-((hydroxy(naphthalen-2-yl)methyl)phenyl)acrylamide (1i)}

White solid. M.p.: 169.9–172.7°C. \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) 8.90 (br, 1H), 8.12 (d, \(J = 8.4\) Hz, 1H), 7.89 (d, \(J = 8.0\) Hz, 1H), 7.85 (d, \(J = 8.3\) Hz, 2H), 7.57 (d, \(J = 7.1\) Hz, 1H), 7.48 (t, \(J = 7.3\) Hz, 2H), 7.42 (t, \(J = 7.2\) Hz, 1H), 7.31 (t, \(J = 7.7\) Hz, 1H), 6.97 (t, \(J = 7.7\) Hz, 1H), 6.61 (s, 1H), 6.33 (d, \(J = 17.0\) Hz, 1H), 6.17 (dd, \(J = 16.9, 10.3\) Hz, 1H), 5.69 (d, \(J = 10.3\) Hz, 1H), 3.34 (br, 1H).

\textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta\) 164.2, 136.6, 136.3, 133.9, 132.1, 131.6, 130.7, 129.0, 128.9(4), 128.8(9), 128.74, 127.4, 126.4, 125.9, 125.4, 124.9, 123.9, 123.3, 72.3. (One carbon was missing due to overlapping) IR (thin film): \(\nu(\text{cm}^{-1})\) 3492, 3363, 3054, 2863, 1673, 1585, 1510, 1446, 1405, 1287, 975, 794. HRMS (ESI) calculated for C\textsubscript{20}H\textsubscript{17}NO\textsubscript{2}Na \([\text{M+Na}]^+\): 326.1152, found: 326.1143.

\textit{N-(2-(cyclohexyl((hydroxy)methyl)phenyl)acrylamide (1j)}

White solid. M.p.: 120.8–123.1°C. \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) 9.43 (br, 1H), 8.33 (d, \(J = 8.3\) Hz, 1H), 8.12 (d, \(J = 8.4\) Hz, 1H), 7.89 (d, \(J = 8.0\) Hz, 1H), 7.85 (d, \(J = 8.3\) Hz, 2H), 7.29 (ddd, \(J = 8.6, 2.2\) Hz, 1H), 7.08–6.97 (m, 2H), 6.36 (dd, \(J = 17.0, 1.3\) Hz, 1H), 6.24 (dd, \(J = 17.0, 10.0\) Hz, 1H), 5.24 (dd, \(J = 10.1, 1.4\) Hz, 1H), 4.43 (dd, \(J = 8.7, 3.3\) Hz, 1H), 2.55 (d, \(J = 2.7\) Hz, 1H), 2.17–2.08 (m, 1H), 1.82–1.72 (m, 2H), 1.67–1.59 (m, 2H), 1.27–1.17 (m, 2H), 1.15–1.07 (m, 2H), 1.06–0.98 (m, 1H), 0.91–0.82 (m, 1H).

\textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta\) 163.7, 136.8, 132.2, 130.8, 128.9, 128.1, 126.5, 123.6, 122.2, 81.3, 43.0, 29.8, 29.6, 26.2, 25.8(7), 25.8(5). IR (thin film): \(\nu(\text{cm}^{-1})\) 3398, 3288, 3048, 2919, 2850, 1662, 1612, 1589, 1533, 1446, 140, 1193, 1014, 748. HRMS (ESI) calculated for C\textsubscript{16}H\textsubscript{17}NO\textsubscript{2}Na \([\text{M+Na}]^+\): 282.1465, found: 282.1452.

\textit{N-(2-((hydroxy(phenyl)methyl)-4-methylphenyl)acrylamide (1k)}
White solid. M.p.: 130.1–132.9°C. ¹H NMR (400 MHz, CDCl₃) δ 8.82 (br, 1H), 7.89 (d, J = 8.3 Hz, 1H), 7.32–7.17 (m, 5H), 7.05 (d, J = 8.3 Hz, 1H), 6.86 (s, 1H), 6.10 (d, J = 17.0 Hz, 1H), 5.97 (dd, J = 16.8, 10.1 Hz, 1H), 5.77 (s, 1H), 5.54 (d, J = 10.2 Hz, 1H), 4.20 (br, 1H), 2.25 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 164.0, 141.6, 134.2, 133.8, 132.7, 131.6, 129.5, 129.2, 128.4, 127.6, 126.7, 126.2, 123.2, 75.3, 21.0. IR (thin film): ν (cm⁻¹) 3365, 3309, 3027, 2879, 2730, 1666, 1619, 1519, 1452, 1446, 1282, 1037, 719. HRMS (ESI) calculated for C₁₇H₁₇NO₂Na [M+Na]⁺: 290.1152, found: 290.1146.

N-(2-(hydroxy(phenyl)methyl)-4-nitrophenyl)acrylamide (I)

Yellow solid. M.p.: 120.9–124.1°C. ¹H NMR (400 MHz, CDCl₃) δ 9.36 (br, 1H), 8.54 (d, J = 9.0 Hz, 1H), 8.17 (d, J = 9.1, 2.6 Hz, 1H), 8.00 (d, J = 2.6 Hz, 1H), 7.42–7.29 (m, 5H), 6.25 (dd, J = 17.0, 0.7 Hz, 1H), 6.08 (dd, J = 17.0, 10.3 Hz, 1H), 6.01 (s, 1H), 5.74 (dd, J = 10.3, 0.7 Hz, 1H), 3.68 (br, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 163.8, 143.0, 142.9, 139.7, 131.4, 131.3, 129.0, 128.6, 128.3, 126.0, 124.6, 124.2, 121.8, 75.7. IR (thin film): ν (cm⁻¹) 3369, 3264, 3058, 2852, 2730, 1679, 1618, 1585, 1513, 1446, 1336, 1215, 975, 698. HRMS (ESI) calculated for C₁₆H₁₄N₂O₄Na [M+Na]⁺: 321.0846, found: 321.0835.

N-(2-(hydroxy(phenyl)methyl)phenyl)but-2-enamide (3a)

White solid. M.p.: 144.7–148.7°C. ¹H NMR (400 MHz, CDCl₃) δ 8.63 (br, 1H), 8.09 (s, 1H), 7.32–7.24 (m, 6H), 7.08–7.02 (m, 2H), 6.77–6.68 (m, 1H), 5.88 (s, 1H), 5.73 (dq, J = 15.2, 1.5 Hz, 1H), 3.63 (br, 1H), 1.80 (dd, J = 6.9, 1.6 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 164.3, 141.4, 140.8, 136.8, 128.8, 128.5, 127.7, 126.3, 125.8, 124.1, 123.1, 75.4, 17.7. (Some carbons were missing due to overlapping) IR (thin film): ν (cm⁻¹) 3322, 3201, 2910, 2723, 2642, 1670, 1633, 1585, 1452, 1533, 1456, 1290, 1027, 755. HRMS (ESI) calculated for C₁₇H₁₇NO₂Na [M+Na]⁺: 290.1152, found: 290.1141.

N-(2-(hydroxy(phenyl)methyl)phenyl)cinnamamide (3b)
White solid. M.p.: 145.6–148.3°C. \(^1\)H NMR (400 MHz, DMSO) \(\delta\) 9.68 (br, 1H), 7.78 (d, \(J = 8.0\) Hz, 1H), 7.66 (d, \(J = 7.5\) Hz, 2H), 7.52 (d, \(J = 15.7\) Hz, 1H), 7.47–7.40 (m, 4H), 7.35 (d, \(J = 7.5\) Hz, 2H), 7.27 (m, 3H), 7.20–7.14 (m, 2H), 6.88 (d, \(J = 15.7\) Hz, 1H), 6.29 (d, \(J = 3.3\) Hz, 1H), 6.03 (d, \(J = 3.1\) Hz, 1H). \(^1\)C NMR (100 MHz, DMSO) \(\delta\) 163.5, 143.9, 140.2, 136.5, 134.7, 129.7, 128.9, 127.9, 127.1, 126.7, 126.2, 124.6, 124.0, 122.2, 71.3. IR (thin film): \(\nu(\text{cm}^{-1})\) 3322, 3216, 3020, 1656, 1680, 1581, 1531, 1456, 985, 763. HRMS (ESI) calculated for \(\text{C}_{22}\text{H}_{19}\text{NO}_2\ [M+Na]^+: 352.1308\), found: 352.1297.

\(\text{N-(2-(hydroxy(phenyl)methyl)phenyl)-3-(\(\rho\)-tolyl)acrylamide (3c)}\)

White solid. M.p.: 181.6–185.2°C. \(^1\)H NMR (400 MHz, DMSO) \(\delta\) 9.64 (br, 1H), 7.78 (d, \(J = 8.4\) Hz, 1H), 7.54 (d, \(J = 8.0\) Hz, 2H), 7.48 (d, \(J = 15.7\) Hz, 1H), 7.40 (d, \(J = 7.9\) Hz, 1H), 7.34 (d, \(J = 7.5\) Hz, 2H), 7.31–7.21 (m, 5H), 7.20–7.13 (m, 2H), 6.81 (d, \(J = 15.7\) Hz, 1H), 6.28 (br, 1H), 6.02 (s, 1H), 2.34 (s, 3H). \(^1\)C NMR (100 MHz, DMSO) \(\delta\) 163.6, 143.9, 140.1, 139.5, 136.4, 131.9, 129.5, 127.9, 127.8, 127.6, 126.7, 126.2, 124.5, 123.9, 121.2, 71.3, 20.9. IR (thin film): \(\nu(\text{cm}^{-1})\) 3334, 3290, 3021, 2861, 2736, 1658, 1602, 1528, 1448, 1336, 975, 744. HRMS (ESI) calculated for \(\text{C}_{23}\text{H}_{21}\text{NO}_2\ [M+Na]^+: 366.1465\), found: 366.1453.

\(\text{N-(2-(hydroxy(phenyl)methyl)phenyl)-3-(4-methoxyphenyl)acrylamide (3d)}\)

White solid. M.p.: 180.8–184.2°C. \(^1\)H NMR (400 MHz, DMSO) \(\delta\) 9.65 (br, 1H), 7.79 (d, \(J = 8.0\) Hz, 1H), 7.61 (d, \(J = 8.7\) Hz, 2H), 7.47 (d, \(J = 15.7\) Hz, 1H), 7.31–7.22 (m, 3H), 7.21–7.11 (m, 2H), 7.00 (d, \(J = 8.7\) Hz, 2H), 6.76 (d, \(J = 15.7\) Hz, 1H), 6.31 (br, 1H), 6.03 (s, 1H), 3.80 (s, 3H). \(^1\)C NMR (100 MHz, DMSO) \(\delta\) 163.8, 160.6, 143.9, 139.9, 136.4, 135.8, 129.4, 127.9(2), 127.8(6), 127.7, 127.3, 126.7, 126.2, 124.3, 123.8, 119.7, 114.4, 71.2, 55.3. IR (thin film): \(\nu(\text{cm}^{-1})\) 3322, 3249, 2998, 2829, 1658, 1602, 1528, 1448, 1336, 975, 744. HRMS (ESI) calculated for \(\text{C}_{23}\text{H}_{21}\text{NO}_3\ [M+Na]^+: 382.1414\), found: 382.1406.

\(\text{N-(2-(hydroxy(phenyl)methyl)phenyl)-3-(3,4,5-trimethoxyphenyl)acrylamide (3e)}\)

White solid. M.p.: 116.3–119.7°C. \(^1\)H NMR (400 MHz, DMSO) \(\delta\) 9.64 (br, 1H), 7.78 (d, \(J = 8.0\) Hz, 1H), 7.46 (d, \(J = 15.6\) Hz, 1H), 7.39–7.34 (m, 3H), 7.30–7.24 (m, 3H), 7.21–7.17 (m, 1H), 7.14 (t, \(J = 7.5\) Hz, 1H), 6.99 (s, 2H), 6.83 (d, \(J = 15.7\) Hz, 1H), 6.31 (d, \(J = 4.4\) Hz, 1H), 6.02 (s, 1H), 3.85 (s, 6H), 3.71 (s, 3H). \(^1\)C NMR (100 MHz, DMSO) \(\delta\) 163.6, 153.1, 143.9, 140.5, 139.1, 136.4, 135.8, 130.3, 127.9, 127.8, 127.3, 126.7, 126.2, 124.5, 124.0, 121.4, 105.5, 71.4, 60.1, 56.0. IR (thin film): \(\nu(\text{cm}^{-1})\) 3378, 3300, 2927, 2836, 1664, 1583, 1519, 1448, 1319, 1121, 755. HRMS (ESI) calculated for \(\text{C}_{25}\text{H}_{25}\text{NO}_5\ [M+Na]^+: 442.1625\), found: 442.1611.
**N-(2-(hydroxy(phenyl)methyl)phenyl)-3-(naphthalen-2-yl)acrylamide (3f)**

White solid. M.p.: 166.5–170.3°C. \(^1\text{H NMR}\) (400 MHz, DMSO) \(\delta\) 9.80 (br, 1H), 8.28 (d, \(J = 15.6\) Hz, 1H), 8.20 (d, \(J = 8.6\) Hz, 1H), 8.06–7.97 (m, 2H), 7.90 (d, \(J = 6.6\) Hz, 1H), 7.80 (d, \(J = 6.4\) Hz, 1H), 7.68–7.57 (m, 3H), 7.46 (d, \(J = 8.4\) Hz, 1H), 7.39–7.33 (m, 2H), 7.31–7.25 (m, 3H), 7.21–7.16 (m, 2H), 6.94 (d, \(J = 15.7\) Hz, 1H), 6.30 (s, 1H), 6.06 (s, 1H).

\(^{13}\text{C NMR}\) (100 MHz, DMSO) \(\delta\) 163.9, 144.5, 137.3, 137.0, 136.0, 133.8, 132.2, 131.3, 130.4, 129.2, 128.4, 128.1, 127.8, 127.5, 127.3, 126.8, 126.3, 125.7, 125.3, 125.2, 124.7, 123.6, 71.7. (One carbon was missing due to overlapping)

IR (thin film): \(\nu(\text{cm}^{-1})\) 2996, 2871, 1639, 1592, 1548, 1417, 1380, 755.

HRMS (ESI) calculated for C\(_{26}\)H\(_{21}\)NO\(_2\)Na \([\text{M+H}]^+\): 402.1465, found: 402.1457.

**3-(4-chlorophenyl)-N-(2-(hydroxy(phenyl)methyl)phenyl)acrylamide (3g)**

White solid. M.p.: 201.8–205.8°C. \(^1\text{H NMR}\) (400 MHz, DMSO) \(\delta\) 9.71 (br, 1H), 7.79 (d, \(J = 8.0\) Hz, 1H), 7.70 (d, \(J = 8.2\) Hz, 2H), 7.57–7.47 (m, 3H), 7.43 (d, \(J = 7.7\) Hz, 1H), 7.36 (d, \(J = 7.8\) Hz, 2H), 7.28 (d, \(J = 7.5\) Hz, 3H), 7.21–7.14 (m, 2H), 6.92 (d, \(J = 15.8\) Hz, 1H), 6.31 (br, 1H), 6.03 (s, 1H). \(^{13}\text{C NMR}\) (100 MHz, DMSO) \(\delta\) 163.3, 143.9, 138.8, 136.5, 135.6, 134.2, 133.7, 129.5, 128.9, 127.9, 127.8, 127.7, 127.3, 126.7, 126.2, 124.6, 124.0, 123.1, 71.3. IR (thin film): \(\nu(\text{cm}^{-1})\) 3293, 3031, 1656, 1618, 1527, 1444, 1336, 1089, 754. HRMS (ESI) calculated for C\(_{22}\)H\(_{18}\)NO\(_2\)ClNa \([\text{M+H}]^+\): 386.0918, found: 386.0902.

4. References

5. NMR Spectra

- 1a

- 1a
$\text{2e}$

$\text{2e}$
2i