Supporting information for:

Synthesis of (±)-Brazilin

Yaodong Huang, Jinsong Zhang, Thomas R. R. Pettus*

Department of Chemistry and Biochemistry
University of California at Santa Barbara

General procedure of IBX oxidation (compound 15-20):

IBX (1.2 equiv) was added to the solution of phenol (1.0 equiv) in d₇-DMF (0.06 M) in an NMR tube. CH₂Cl₂ (1.0 equiv) was added as standard. The reaction mixture was shaken periodically and ¹H NMR was taken every 15 min. The yield and the regioselectivity were recorded. After 75 min-90 min, Na₂S₂O₄ (3.0 equiv) was added and the reaction mixture was stirred at room temperature for another 3 hours. The reaction was quenched with water, extracted with ethyl acetate. The combined organic layers were washed with brine, water, dried over MgSO₄, filtered and condensed to give the crude product. The crude product was subjected to preparative TLC with ethyl acetate/Hexane (3:7) to give the desired product.

Compound 21: To a stirring solution of **11** (1.000 g, 1.00 equiv) in 80 mL CH₂Cl₂ and 8 mL THF at room temperature, oxylyl chloride (0.816 mL, 1.05 equiv) was added. One drop of DMF was added every 30-60 minutes, and the total amount of DMF equal to 20 μ L (0.03 equiv). The acid **11** was transferred to corresponding acetyl chloride completely after 4 hours, which was confirmed by FT-IR (1720 cm⁻¹ to 1780 cm⁻¹). Then the reaction was concentrated to about 10 mL solution, which was then diluted by 80 mL CH₂Cl₂ and cooled to 0°C. 44 mL CH₂N₂ (0.33 M¹ in Et₂O, 2.2 equiv) was added to the reaction mixture and the solution was allowed to warm to room temperature overnight and then was condensed. Flash chromatography (1:4 EtOAc:Hexane) yielded 786 mg (68%).
¹H NMR [CDCl₃, 400 MHz] δ 7.98 (br.s, 1H), 7.15(t, J=7.8H, 1H), 6.79 (ddd, J₁=8.1 Hz, J₂=2.5 Hz, J₃=0.9 Hz, 1H), 6.74(s, 1H), 6.72(d, J=7.4Hz, 1H), 5.23(s, 4H), 3.53 (s, 2H);
¹³C NMR [CDCl₃, 100MHz] δ 194.96, 156.85, 135.76, 130.17, 121.22, 116.48, 114.79, 56.24, 47.77; IR [CH₂Cl₂ solution, $\nu_{\max}$ cm⁻¹] 2958, 2110, 1732, 1639, 1600, 1456, 1360, 1246, 1153, 1045.

¹ Titration of diazomethane: Arndt, F. *Organic Synthesis* collective volume 2, 165-167.

Compound 24: To a stirring solution of **21** (760 mg, 1.00 equiv) in 50 mL acetone, potassium carbonate (2.98 g, 5.0 equiv) was added at room temperature. After 4.5 hours, MOMCl (1.64 mL, 5.0 equiv.) and *n*-Bu₄NI (80 mg, 0.05 equiv) was added. The reaction was stirred for an additional 24 hours at room temperature. The reaction was quenched by H₂O, extracted by Et₂O, washed with brine, H₂O, dried with MgSO₄. Flash chromatography (1:6 EtOAc:Hexane) yielded 630 mg (76%).

¹H NMR [CDCl₃, 400 MHz] δ 7.26(t, J=8.0 Hz, 1H), 6.79 (dd, J₁=8.2 Hz, J₂=2.4 Hz, 1H), 6.91(d, J=2.3Hz, 1H), 6.88(dd, J₁=7.5 Hz, J₂=0.5 Hz, 1H), 5.17(s, 3H), 3.58 (s, 2H), 3.48 (s, 3H); ¹³C NMR [CDCl₃, 100MHz] δ 192.90, 157.78, 136.23, 130.09, 123.09, 117.52, 115.19, 94.56, 56.26, 55.13, 48.23; IR [CH₂Cl₂ solution, ν_{max} cm⁻¹] 2108, 1639, 1358, 1151, 1016.

INDEX	FREQUENCY	PPM	HEIGHT
1	2910.344	7.277	99.7
2	2907.166	7.257	43.6
3	2907.166	7.257	43.6
4	2902.560	7.257	100.6
5	2894.728	7.238	70.8
6	2789.084	6.974	59.7
7	2789.084	6.974	59.7
8	2783.249	6.959	45.0
9	2780.346	6.953	59.3
10	2778.531	6.948	74.8
11	2762.827	6.908	74.8
12	2756.531	6.892	74.2
13	2756.071	6.891	74.9
14	2748.547	6.872	63.9
15	2748.547	6.872	63.9
16	2070.153	5.176	613.4
17	2066.928	5.168	89.1
18	1844.293	3.586	122.6
19	1844.293	3.586	122.6
20	1402.201	3.506	27.9
21	1393.448	3.484	90.2
22	30.211	0.076	47.8

Compound 25: To a stirring solution of **24** (180 mg, 0.818 mmol) in 70 mL dry CH_2Cl_2 at room temperature, $\text{Rh}_2(\text{OAc})_4 \cdot 2\text{H}_2\text{O}$ (4 mg, 0.01 equiv) was added. After 5 hours, the reaction was filtered through celite and condensed. Flash chromatography (1:8 EtOAc:Hexane) yielded 112 mg (74%).

^1H NMR [CDCl_3 , 400 MHz] δ 7.20(d, $J=8.3$ Hz, 1H), 7.00 (s, 1H), 6.94(d, $J_1=8.3\text{Hz}$, $J_2=2.3$ Hz, $J_3=0.7$ Hz, 1H), 5.17(d, $J=0.7$ Hz, 1H), 3.49 (s, 2H), 3.48 (s, 2H), 3.48 (s, 3H); ^{13}C NMR [CDCl_3 , 100MHz] δ 215.45, 156.79, 139.14, 131.09, 125.97, 116.10, 112.88, 94.66, 56.11, 44.57, 43.58.

INDEX	FREQUENCY	PPM	HEIGHT
1	21667.220	215.450	19.3
2	13991.341	139.144	23.9
3	13183.437	131.095	23.5
4	12668.437	125.974	84.2
5	1197.220	111.144	85.8
6	11311.944	112.109	85.8
7	9519.327	94.656	85.7
8	9507.120	94.535	-18.8
9	9505.594	94.520	20.2
10	7766.802	77.230	32.8
11	7734.757	76.911	31.5
12	5642.714	56.109	70.2
13	4382.289	43.576	87.0
14	4382.289	43.576	87.0

Compound 28: To a stirring solution of **27** (63.0 mg, 1.2 equiv) in 2.0 mL THF (clear solution) at -78°C , *n*-BuLi (50.0 μL , 2.5 M in Hexane, 1.2 equiv) was added, which gave yellow solution. After 5 minutes, the reaction was further cooled to -100°C , and the compound **25** (20 mg, 1.0 equiv) was added. The reaction was allowed to warm up to room temperature over night. The reaction was quenched by 1 M NH_4Cl , extracted by Et_2O , washed with brine, H_2O , dried with MgSO_4 . Flash chromatography (1:4 EtOAc:Hexane) yielded 25.6 mg (61%).

^1H NMR [CDCl_3 , 400 MHz] δ 7.45-7.34(m, 5H), 7.20 (t, $J=8.1\text{Hz}$, 1H), 7.13(d, $J=8.2\text{Hz}$, 1H), 6.93(d, $J=2.3\text{Hz}$, 1H), 6.87(dd, $J_1=8.2\text{Hz}$, $J_2=2.4\text{Hz}$, 1H), 6.63-6.59(m, 2H), 6.55(d, $J=8.3\text{Hz}$, 1H), 5.16(d, $J=0.8\text{Hz}$, 2H), 5.06(s, 2H), 4.02(s, 2H), 3.49 (s, 3H), 3.19(AB, $J=16.2\text{Hz}$, 2H), 3.07(AB, $J=15.5\text{Hz}$, 2H).

Compound 8: To a stirring solution of **28** (24.0 mg) in MeOH (4.0 mL), concentrated HCl (0.16 mL) was added. The reaction mixture was warmed to 60 °C and stirred for 2 hours, and then was cooled to room temperature, quenched by cold water, extracted by Et₂O, washed with brine, H₂O, dried with MgSO₄. No further purification was required.

¹H NMR [CDCl₃, 400 MHz] δ 7.45-7.34(m, 5H), 7.20 (t, J=8.2Hz, 1H), 7.07(d, J=8.2Hz, 1H), 6.71(s, 1H), 6.66(dd, J₁=8.0Hz, J₂=2.6Hz, 1H), 6.62-6.59(m, 2H), 6.55(ddd, J₁=8.2Hz, J₂=2.3Hz, J₃=0.8H), 5.06(s, 2H), 4.68(br.s, 1H), 4.02(s, 2H), 3.19(AB, J=16.2Hz, 2H), 3.07(AB, J=16.2Hz, 2H).

Compound 29: A stirring solution of **8** (20.0 mg, 1.00 equiv) in DMF (2.0 mL) was stirred with IBX (16.6 mg, 1.05 equiv) at room temperature. After 2 hours, Na₂S₂O₄ (31.0 mg, 3.20 equiv) was added as solid. The reaction stirred for an additional 2 hours, then quenched by 1 M NH₄Cl, extracted by Et₂O, washed with brine, H₂O, dried with MgSO₄. Flash chromatography (1:2 EtOAc:Hexane) yielded 12.0 mg (58%).

¹H NMR [(CD₃)₂CO, 400 MHz] δ 7.63(s, 2H), 7.47(d, J=7.3Hz, 2H), 7.39(t, J=7.8Hz, 2H), 7.34-7.30(m, 1H), 7.17(t, J=8.2Hz, 1H), 6.67(s, 2H), 6.63(t, J=2.3Hz, 1H), 6.59(dd, J₁=8.2Hz, J₂=2.2Hz, 1H), 6.56(dd, J₁=8.2Hz, J₂=2.2Hz, 1H), 5.09(s, 2H), 4.19(s, 1H), 4.00(s, 2H), 3.12(d, J=15.9Hz, 2H), 2.86(d, J=15.9Hz, 2H); ¹³C NMR [(CD₃)₂CO, 100MHz] δ 161.54, 161.07, 144.84, 138.46, 133.11, 130.77, 129.32, 128.64, 128.44, 112.43, 108.15, 107.96, 102.58, 81.67, 74.58, 70.41, 44.42. HRMS (EI) *m/z* calcd. for C₂₃H₂₂O₅ (M⁺) = 378.1467, found 378.1480.

Compound 7: A stirring solution of **29** (5 mg) in THF (0.5 mL) was stirred with PIFA (5.4 mg, 1.00 equiv) at room temperature. After 10 minutes, the reaction was condensed by rotary evaporator. No further purification was necessary.

^1H NMR [CDCl_3 , 400 MHz] δ 7.45-7.33(m, 5H), 7.23(t, $J=8.2\text{Hz}$, 1H), 6.67(ddd, $J_1=8.2\text{Hz}$, $J_2=2.2\text{Hz}$, $J_3=0.6\text{Hz}$, 1H), 6.57(t, $J=2.2\text{Hz}$, 1H), 6.54(ddd, $J_1=8.2\text{Hz}$, $J_2=2.4\text{Hz}$, $J_3=0.6\text{Hz}$, 1H), 6.41(s, 2H), 5.07(s, 2H), 4.04(s, 2H), 3.00(AB, $J=16.5\text{Hz}$, 4H).

Compound 31: To a stirring solution of **7** (5 mg, 1.0 equiv) in THF (0.5 mL) at room temperature, PhSH (1.4 μ L, 1.05 equiv) was added. The reaction stirred for an additional 1 hour, then quenched by 1 M NH_4Cl , extracted by Et_2O , washed with brine, H_2O , dried with MgSO_4 . Preparative TLC (1:20 $\text{MeOH}:\text{CH}_2\text{Cl}_2$) yielded 5.9 mg (91%).

^1H NMR [CDCl_3 , 400 MHz] δ 7.44-7.33(m, 5H), 7.24-7.12(m, 5H), 7.05(dd, $J_1=8.5\text{Hz}$, $J_2=1.2\text{Hz}$, 2H), 6.93(s, 1H), 6.61(dd, $J_1=7.4\text{Hz}$, $J_2=2.0\text{Hz}$, 1H), 6.55(t, $J=2.4\text{Hz}$, 1H), 6.50(md, $J=8.3\text{Hz}$, 1H), 6.35(s, 1H, ArOH), 5.36(s, 1H, ArOH), 5.04(s, 2H), 3.96(AB, $J=2\text{H}$), 3.15(AB, $J=16.6\text{Hz}$, 2H), 3.05(AB, $J=16.6\text{Hz}$, 2H).

Compound 32: To a stirring solution of **7** (5 mg, 1.0 equiv) in THF (0.5 mL) at room temperature, Hünig's base (2.3 μ L, 1.0 equiv) was added. After an additional 1 hour, PhSH (1.4 μ L, 1.05 equiv) was added and stirring was continued for 30 more minutes. The reaction was quenched by 1 M NH_4Cl , extracted by Et_2O , washed with brine, H_2O , dried with MgSO_4 . Preparative TLC (1:20 $\text{MeOH}:\text{CH}_2\text{Cl}_2$) yielded 5.2 mg (81%).

^1H NMR [CDCl_3 , 400 MHz] δ 7.49(dd, $J_1=7.9\text{Hz}$, $J_2=1.6\text{Hz}$, 2H), 7.44-7.32(m, 5H), 7.23-7.17(m, 3H), 7.13(t, $J=8.2\text{Hz}$, 1H), 6.90(s, 1H), 6.70(s, 1H), 6.58(dd, $J_1=7.8\text{Hz}$, $J_2=2.1\text{Hz}$, 1H), 6.43(t, $J=2.2\text{Hz}$, 1H), 6.50(dd, $J_1=8.3\text{Hz}$, $J_2=2.0\text{Hz}$, 1H), 5.48(br.s, 1H, ArOH), 5.34(br.s, 1H, ArOH), 5.02(s, 2H), 3.86(AB, $J=9.5\text{Hz}$, 2H), 3.38(s, 1H), 3.05(AB, $J=16.1\text{Hz}$, 2H). HRMS (ESI+/TOF) m/z calcd for $\text{C}_{29}\text{H}_{26}\text{O}_5\text{S}$ ($\text{M}+\text{Na}$) $^+$ = 509.1393, found 509.1395.

Compound 6: To a stirring solution of **7** (5 mg, 1.0 equiv) in THF (0.5 mL) at room temperature, Hünig's base (2.3 μ L, 1.0 equiv) and LiI (1.06 mg, 1.0 equiv) was added at room temperature. After 12 hours, the reaction was quenched by 1 M NH_4Cl , extracted by Et_2O , washed with brine, H_2O , dried with MgSO_4 . Preparative TLC (1:20 $\text{MeOH}:\text{CH}_2\text{Cl}_2$) yielded 4.1 mg (82%) **6** and **29** in 1:1 ratio.

^1H NMR [$(\text{CD}_3)_2\text{CO}$, 400 MHz] δ 7.74(s, 1H, ArOH), 7.66(s, 1H, ArOH), 7.46(d, 7.5Hz, 1H), 7.38(t, $J=7.0\text{Hz}$, 1H), 7.33-7.29(m, 2H), 6.76(d, $J=0.7\text{Hz}$, 1H), 6.66(dd, $J_1=8.4\text{Hz}$, $J_2=2.7\text{Hz}$, 1H), 6.65(s, 1H), 6.46(d, $J=2.7\text{Hz}$, 1H), 5.08(s, 2H), 4.51(s, 1H, ROH), 4.00(s, 1H), 3.96(dd, $J_1=11.2\text{Hz}$, $J_2=1.3\text{Hz}$, 1H), 3.72(d, $J=11.2\text{Hz}$, 1H), 3.02(d, $J=15.6\text{Hz}$, 1H), 2.82(d, $J=15.8\text{Hz}$, 1H); ^{13}C NMR [$(\text{CD}_3)_2\text{CO}$, 100MHz] δ 159.25, 155.60, 145.27, 144.91, 138.60, 137.18, 132.09, 131.49, 129.34, 128.62, 128.38, 117.00, 112.72, 112.33, 109.39, 103.42, 77.70, 70.94, 70.42, 51.14, 42.95. HRMS (ESI+/TOF) m/z calcd for $\text{C}_{23}\text{H}_{20}\text{O}_5$ ($\text{M}+\text{Na}$) $^+$ = 399.1202, found 399.1203.

INDEX	PPM	PPM	PPM	PPM
1	16015.208	159.248	159.248	45.9
2	15648.223	155.599	155.599	37.0
3	14609.831	145.274	145.274	30.8
4	13757.507	134.210	134.210	34.0
5	13978.429	133.555	133.555	38.4
6	13795.750	137.179	137.179	39.1
7	13283.802	132.088	132.088	87.2
8	13224.291	131.497	131.497	45.4
9	13067.409	129.342	129.342	202.7
10	12987.111	128.522	128.522	208.8
11	12950.713	128.375	128.375	238.5
12	11766.269	116.999	116.999	38.4
13	11335.958	112.720	112.720	55.8
14	11286.284	112.325	112.325	62.8
15	11046.147	107.469	107.469	38.5
16	10400.166	103.419	103.419	95.7
17	7854.132	77.700	77.700	47.5
18	7134.322	70.941	70.941	45.9
19	7081.678	70.417	70.417	98.8
20	644.569	61.445	61.445	75.5
21	4324.990	43.946	43.946	53.5

Compound 1: A solution of **6** (3.0 mg) in EtOAc (1.0 mL) was stirred under N₂. 10% Pd/C (0.5 mg) was added to the reaction and the N₂ line was replaced with a H₂ balloon. After 12 hours, the reaction was filtered through celite, which was rinsed with EtOAc. The organic layer was condensed under reduced pressure. No further purification was required.

¹H NMR [CD₃OD, 400 MHz] δ 7.18 (d, J=8.2Hz, 1H), 6.70(s, 1H), 6.60(s, 1H), 6.46(dd, J₁=8.3Hz, J₂=2.5Hz, 1H), 6.28(d, J=2.4Hz, 1H), 3.96(s, 1H), 3.92(dd, J₁=11.3Hz, J₂=1.3Hz, 1H), 3.68(d, J=11.2Hz, 1H), 3.01(d, J=15.4Hz, 1H), 2.77(d, J=15.7Hz, 1H).

Compound 30: A solution of **29** (5.0 mg) in EtOH (2.5 mL) with EtOAc (2.5 mL) was stirred under N₂. 10% Pd/C (0.5 mg) was added to the reaction and the N₂ line was replaced with a H₂ balloon. After 24 hours, the reaction was filtered through celite, which was rinsed with EtOAc. The organic layer was condensed under reduced pressure. The crude ¹H NMR showed **30** (85%) and **1** (8%).

¹H NMR [(CD₃)₂CO, 400 MHz] δ 8.31 (s, 1H, ArOH), 7.59(s, 1H, ArOH), 7.58(s, 1H, ArOH), 7.06(t, J=7.9Hz, 1H), 6.67(s, 2H), 6.45-6.41(m, 3H), 3.96(s, 2H), 3.11(d, J=15.8Hz, 2H), 2.85(d, J=15.8Hz, 2H).

Compound 26: 26 was obtained from *p*-hydroxyphenylacetic acid (500 mg), following a similar procedure used in preparation of **21**. Flash chromatography (1:4 EtOAc:Hexane) yielded 411mg (71%).

^1H NMR [CDCl_3 , 400 MHz] δ 7.07 (dd, $J_1=6.4\text{Hz}$, $J_2=1.9\text{Hz}$, 2H), 6.80 (dd, $J_1=6.5\text{ Hz}$, $J_2=2.0\text{ Hz}$, 2H), 6.32(br. s, 1H), 5.18(s, 1H), 3.55 (s, 2H); HRMS (HREI) m/z calcd. for $\text{C}_9\text{H}_8\text{N}_2\text{O}_2(\text{M})^+ = 176.0586$, found 176.0593.

Compound 9: 9 was obtained from **26** (100 mg), following a similar procedure used in preparation of **25**. Flash chromatography (1:4 EtOAc:Hexane) yielded 24 mg (28%).

^1H NMR $[(\text{CD}_3)_2\text{CO}, 400 \text{ MHz}]$ δ 8.34 (s, 1H), 7.12(dd, $J_1=8.2\text{Hz}$, $J_2=0.5\text{Hz}$, 1H), 6.79(m, 1H), 6.73(d, $J=8.2\text{Hz}$, 1H), 3.44(s, 2H), 3.43(s, 2H); HRMS (HREI) m/z calcd. for $\text{C}_9\text{H}_8\text{O}_5 (\text{M})^+ = 148.0524$, found 148.0531.

Compound 23: **23** was obtained from **21** (80 mg), following a similar procedure used in preparation of **25**. Flash chromatography (1:4 EtOAc:Hexane) yielded 35 mg (53%).

^1H NMR $[(\text{CD}_3)_2\text{CO}, 400 \text{ MHz}]$ δ 8.48 (s, 1H), 7.09(t, $J=7.8\text{Hz}$, 1H), 6.80(d, $J=7.5\text{Hz}$, 1H), 6.75(dd, $J_1=8.2\text{Hz}$, $J_2=0.7\text{Hz}$, 1H), 3.47(d, $J=0.7\text{Hz}$, 2H), 3.39(s, 2H); ^{13}C NMR $[(\text{CD}_3)_2\text{CO}, 100\text{MHz}]$ δ 214.45, 154.56, 140.81, 129.11, 125.44, 116.91, 114.28, 44.59, 41.34.

INDEX	FREQUENCY	PPM	HEIGHT
1	3391.889	8.481	34.5
2	2844.068	7.111	13.7
3	2836.110	7.091	24.8
4	2828.471	7.072	17.4
5	2728.132	6.816	20.9
6	2719.129	6.799	19.2
7	2718.652	6.797	19.5
8	2704.168	6.761	23.7
9	2703.373	6.759	24.1
10	2696.051	6.741	21.9
11	2695.255	6.739	22.0
12	1389.366	3.474	90.6
13	1388.728	3.472	80.3
14	1352.282	3.381	95.7
15	1188.349	2.971	43.8
16	1181.506	2.954	21.7
17	1177.527	2.944	82.7
18	1176.572	2.942	70.5
19	1164.635	2.912	11.0
20	824.355	2.061	20.2
21	822.127	2.056	37.0
22	819.899	2.050	54.4
23	817.671	2.044	37.5
24	815.443	2.039	19.3

INDEX	FREQUENCY (PPM)	HEIGHT
1	21640.821	215.207
2	21506.525	214.448
3	20970.027	207.423
4	20755.495	206.384
5	20244.311	201.301
6	20018.474	199.055
7	17777.752	165.499
8	17077.752	165.499
9	16040.223	159.696
10	15540.697	154.560
11	15400.786	153.149
12	14161.209	140.113
13	13784.720	137.114
14	12984.720	129.114
15	12835.343	127.430
16	12634.683	125.435
17	12250.328	121.802
18	11720.884	116.627
19	11450.138	114.283
20	9590.299	95.362
21	8850.722	88.099
22	4138.004	41.345
23		
24		
25

Compound 27: To a stirring solution of 3-(benzyloxy)phenol (500 mg, 1.00 equiv) in 50 ml acetone, potassium carbonate (1.725 g, 5.0 equiv) was added at room temperature. After 1 hour, *n*-Bu₃SnCH₂Br (1.150 g, 1.2 equiv) and *n*-Bu₄NI (92 mg, 0.1 equiv) was added. The reaction was allowed to warm up to reflux for an additional 24 hours. And then the reaction was cooled to room temperature and quenched by H₂O, extracted by Et₂O, washed with brine, H₂O, dried with MgSO₄. Flash chromatography (1:15 EtOAc:Hexane) yielded 855 mg (68%).

¹H NMR [CDCl₃, 200 MHz] δ 7.48-7.30(m, 5H), 7.18(t, J=8.2Hz, 1H), 6.63-6.53(m, 3H), 5.06(s, 2H), 4.15(s, 2H), 1.58-1.41(m, 6H), 1.37-1.23(m, 7H), 1.01-0.87(m, 14H).
HRMS (HREI) *m/z* calcd. for C₂₆H₄₀O₂Sn (M-C₄H₉)⁺ = 443.1342, found 443.1352.

Compound 22: A solution of trifluoroacetic acid (1.00 mL) and CH_2Cl_2 (0.10 mL) was set stirring at -25°C under N_2 . **21** (7 mg, in 0.35 mL CH_2Cl_2) was added slowly. After stirring for an additional 30 minutes, the reaction was allowed to warm to room temperature, condensed and then diluted by Et_2O , washed by brine, H_2O , dried by MgSO_4 . Flash chromatography (1:6 EtOAc:Hexane) yielded 6.3 mg (90%).

^1H NMR [CDCl_3 , 400 MHz] δ 7.24 (t, $J=7.8\text{Hz}$, 1H), 6.79 (dd, $J_1=7.8\text{ Hz}$, $J_2=1.8\text{ Hz}$, 2H), 6.70(d, $J=1.8\text{Hz}$, 1H), 5.32(br. s, 1H), 4.94(s, 2H), 3.74 (s, 2H).