

A Facile, Environmentally Benign Sulfonamide Synthesis in Water

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Supporting information

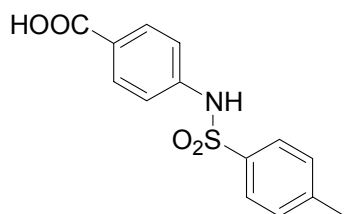
- 1) General Experimental Methods
- 2) Experimental procedures
- 3) Proton and carbon NMR spectra of sulfonamide compounds (Table 1, entries 1-13; Table 2, entries 1-8)

General Experimental Methods:

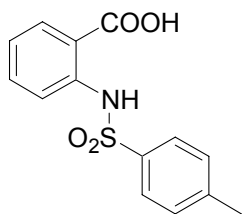
^1H and ^{13}C NMR spectra were recorded at Bruker 500 NMR spectrometer (^1H , 500 MHz; ^{13}C , 125 MHz) and internally referenced to TMS. Infrared spectroscopy was performed on a Nicolet Avatar 360 FT-IR. HPLC analysis was performed on a Hewlett Packard 1100 (Agilent ZORBAX® Eclipse XDB-C8, 5 μm , 4.6x150 mm, Flow rate 1 mL/min, Gradient (acetonitrile/water with 0.05% trifluoroacetic acid): 1% acetonitrile/99% water to 99% acetonitrile/1% water ramp over 8 min, then hold at 99% acetonitrile/1% water). HRMS (ESI) was performed on Bruker μTof . The pH control was achieved using a J-Kem syringe pump (model 1250) coupled with a J-Kem Infinity controller.

All the reagents were purchased from commercial sources and used without further purification.

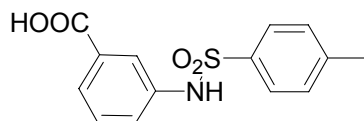
General procedure. The solid mixture of 4-aminobenzoic acid (1.0 g, 7.3 mmol, 1.0 equiv.) and p-toluenesulfonyl chloride (1.4 g, 7.3 mmol, 1.0 equiv.) was suspended in 30 mL water. The pH of the suspension was adjusted and was maintained at 8.0 by adding 1 mol/L Na_2CO_3 aqueous solution at room temperature using a syringe pump equipped with a pH controller. It took 2 hours for the reaction to complete. Concentrated HCl was added slowly to adjust pH = 2.0. The precipitate was collected by filtration, washed with water and dried to afford the title compound as a white solid (2.08 g, 7.1 mmol, 98%). No further purification was needed.



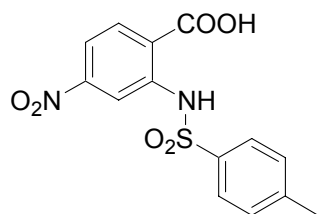
4-(Toluene-4-sulfonylamino)-benzoic acid: HPLC retention time: 7.93 min. ^1H NMR (500 MHz, DMSO- d_6 , δ): 12.72 (br. s, 1H), 10.71 (s, 1H), 7.79 (d, $J = 8.7$ Hz, 2H), 7.70 (d, $J = 8.3$ Hz, 2H), 7.36 (d, $J = 8.3$ Hz, 2H), 7.18 (d, $J = 8.6$ Hz, 2H), 2.33 (s, 3H). ^{13}C NMR (125.7 MHz, DMSO- d_6 , δ): 165.1, 142.0, 140.5, 134.9, 129.1, 128.2, 125.1, 124.0, 116.5, 19.3. IR (dry film, cm^{-1}): 3212 (m), 3004 (w), 1707 (s), 1336 (s), 1275 (s), 1150 (s). HRMS-ESI (m/z): $[\text{M}-\text{H}]^-$ calcd for $\text{C}_{14}\text{H}_{13}\text{NO}_4\text{S}$ 290.0482; found, 290.0473.



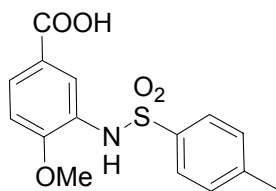
2-(Toluene-4-sulfonylamino)-benzoic acid: Following the general procedure, the title compound was prepared in 94% yield. HPLC retention time: 8.84 min. ^1H NMR (500 MHz, DMSO- d_6 , δ): 11.22 (br. s, 1H), 7.89 (dd, $J = 7.9, 1.5$ Hz, 1H), 7.69 (d, $J = 8.3$ Hz, 2H), 7.60-7.46 (m, 2H), 7.35 (dd, $J = 8.3$ Hz, 2H), 7.10 (td, $J = 7.4, 1.5$ Hz, 1H), 2.33 (s, 3H). ^{13}C NMR (125.7 MHz, DMSO- d_6 , δ): 168.1, 142.4, 138.4, 134.2, 132.8, 129.9, 128.3, 125.3, 121.6, 116.7, 115.1, 19.4. IR (dry film, cm^{-1}): 3193 (br. m), 2924 (w), 1669 (s), 1341 (m), 1225 (s), 1154 (s). HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{14}\text{H}_{13}\text{NO}_4\text{S}$ 292.038; found, 292.0639.



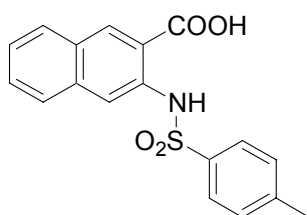
3-(Toluene-4-sulfonylamino)-benzoic acid: Following the general procedure, the title compound was prepared in 93% yield. HPLC retention time: 8.01 min. ^1H NMR (500 MHz, DMSO- d_6 , δ): 10.42 (br. s, 1H), 7.68 (s, 1H), 7.64 (d, $J = 8.5$ Hz, 1H), 7.60-7.54 (m, 1H), 7.33 (t, $J = 8.1$ Hz, 4H), 2.32 (s, 3H). ^{13}C NMR (125.7 MHz, DMSO- d_6 , δ): 165.3, 141.8, 136.5, 135.0, 130.8, 128.1, 127.7, 125.1, 123.1, 122.1, 118.9, 19.3. IR (dry film, cm^{-1}): 3258 (br. w), 3005 (w), 2988 (s), 1679 (m), 1336 (m), 1275 (s), 1261 (s), 1157 (s). HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{14}\text{H}_{13}\text{NO}_4\text{S}$ 292.0638; found, 292.0630.



4-Nitro-2-(toluene-4-sulfonylamino)-benzoic acid: Following the general procedure, the title compound was prepared in 83% yield. HPLC retention time: 9.01 min. ^1H NMR (500 MHz, DMSO- d_6 , δ): 11.52 (br. s, 1H), 8.25 (d, $J = 2.2$ Hz, 1H), 8.12 (d, $J = 8.6$ Hz, 1H), 7.94-7.88 (m, 1H), 7.75 (d, $J = 8.3$ Hz, 2H), 7.39 (d, $J = 8.1$ Hz, 2H), 2.34 (s, 3H). ^{13}C NMR (125.7 MHz, DMSO- d_6 , δ): 150.5, 144.8, 141.0, 135.9, 133.6, 130.5, 127.2, 117.8, 113.2, 110.9, 108.3, 21.3. IR (dry film, cm^{-1}): 3208 (br. w), 3006 (w), 1702 (s), 1530 (s), 1350 (s), 1275 (s), 1261 (s), 1155 (s). HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_6\text{S}$ 337.0489; found, 337.0488.

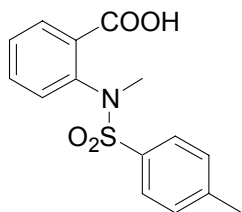


4-Methoxy-3-(toluene-4-sulfonylamino)benzoic acid: Following the general procedure, the title compound was prepared in 91% yield. HPLC retention time: 7.97 min. ^1H NMR (500 MHz, DMSO- d_6 , δ): 12.71 (br. s, 1H), 9.57 (s, 1H), 7.81 (d, $J = 2.1$ Hz, 1H), 7.70 (dd, $J = 8.6, 2.1$ Hz, 1H), 7.59 (d, $J = 8.3$ Hz, 2H), 7.33 (d, $J = 8.1$ Hz, 2H), 6.99 (d, $J = 8.6$ Hz, 1H), 3.59 (s, 3H), 2.34 (s, 3H). ^{13}C NMR (125.7 MHz, DMSO- d_6 , δ): 165.0, 154.0, 146.8, 141.4, 135.9, 127.7, 126.6, 125.1, 123.9, 121.3, 109.8, 54.2, 19.3. IR (dry film, cm^{-1}): 3271 (m), 3001 (w), 1672 (m), 1607 (m), 1337 (s), 1272 (s), 1162 (s). HRMS-ESI (m/z): $[\text{M}-\text{H}]^-$ calcd for $\text{C}_{15}\text{H}_{15}\text{NO}_5\text{S}$ 320.0587; found, 320.0576.

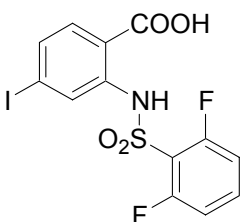


3-(Toluene-4-sulfonylamino)naphthalene-2-carboxylic acid: Following the general procedure, the title compound was prepared in 84% yield. HPLC retention time: 9.50 min. ^1H NMR (500 MHz, DMSO- d_6 , δ): 11.38 (br. s, 1H), 8.60 (s, 1H), 7.97 (d, $J = 8.1$ Hz, 1H), 7.91 (s, 1H), 7.88 (d, $J = 8.3$ Hz, 1H), 7.72 (d, $J = 8.3$ Hz, 2H), 7.60 (t, $J = 7.8$ Hz, 1H), 7.47 (t, $J = 7.8$ Hz, 1H), 7.30 (d, $J = 8.2$ Hz, 2H), 2.28 (s, 3H). ^{13}C NMR (125.7 MHz, DMSO- d_6 , δ): 170.0, 144.2, 136.2, 135.6, 135.6, 133.9, 130.1, 129.8, 129.4, 128.8, 127.3, 127.3, 126.1, 118.0, 115.8, 21.2. IR (dry film, cm^{-1}): 3209 (br, w), 3006 (w), 167

(s), 1350 (m), 1277 (s), 1157 (s). HRMS-ESI (m/z): $[M+H]^+$ calcd for $C_{18}H_{15}NO_4S$ 342.0795; found, 342.0800.



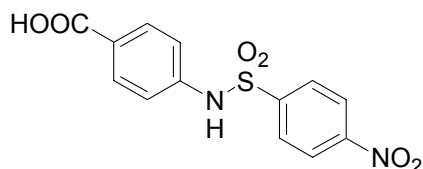
2-[Methyl-(toluene-4-sulfonyl)-amino]-benzoic acid: Following the general procedure, the title compound was prepared in 85% yield. HPLC retention time: 8.39 min. 1H NMR (500 MHz, DMSO- d_6 , δ): 12.91 (br. s, 1H), 7.75 (dd, $J = 7.3, 1.8$ Hz, 1H), 7.55-7.33 (m, 6H), 6.83 (dd, $J = 7.6, 1.4$ Hz, 1H), 3.16 (s, 3H), 2.41 (s, 3H). ^{13}C NMR (125.7 MHz, DMSO- d_6 , δ): 165.7, 141.7, 137.9, 133.6, 131.9, 130.3, 128.7, 128.1, 126.5, 126.5, 125.7, 37.1, 19.4. IR (dry film, cm^{-1}): 3313 (br. m), 1717 (s), 1330 (s), 1148 (s). HRMS-ESI (m/z): $[M+H]^+$ calcd for $C_{15}H_{15}NO_4S$, 306.0795; found, 306.0797.



2-(2,6-Difluoro-benzenesulfonylamino)-4-iodo-benzoic acid: Following the general procedure, the title compound was prepared in 95% yield. HPLC retention time: 9.55 min. 1H NMR (500 MHz, DMSO- d_6 , δ): 11.95 (br. s, 1H), 7.88 (d, $J = 1.35$ Hz, 1H), 7.82-7.70 (m, 1H), 7.67 (d, $J = 8.3$ Hz, 1H), 7.53 (d, $J = 8.3$ Hz, 1H), 7.33 (t, $J = 9.2$ Hz, 2H). ^{13}C NMR (125.7 MHz, DMSO- d_6 , δ): 169.7, 160.4, 158.3, 140.0, 137.5, 137.4, 137.3, 133.3, 133.0, 132.9, 126.2, 124.8, 123.5, 116.5, 115.8, 114.1, 113.9, 102.6. IR

(dry film, cm^{-1}): 3104 (br. w), 3005 (m), 1672 (s), 1354 (m), 1276 (s), 1259 (s), 1171 (s).

HRMS-ESI (m/z): $[M-H]^-$ calcd for $\text{C}_{13}\text{H}_8\text{F}_2\text{INO}_4\text{S}$ 437.9103; found, 437.9112.



4-(4-Nitro-benzenesulfonylamino)-benzoic acid: Following the general procedure, the

title compound was prepared in 92% yield. HPLC retention time: 7.95 min. ^1H NMR

(500 MHz, DMSO-d_6 , δ): 12.81 (br. s, 1H), 11.11 (br. s, 1H), 8.38 (dt, $J = 8.9, 2.0$ Hz,

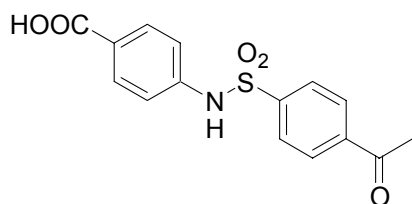
2H), 8.06 (dt, $J = 8.9, 2.0$ Hz, 2H), 7.83 (dt, $J = 8.9, 2.0$ Hz, 2H), 7.22 (dt, $J = 8.8, 1.9$

Hz, 2H). ^{13}C NMR (125.7 MHz, DMSO-d_6 , δ): 166.5, 149.9, 144.5, 141.1, 130.8, 128.2,

126.2, 124.7, 118.7. IR (dry film, cm^{-1}): 3256 (m), 1675 (s), 1607 (m), 1528 (s), 1300

(s), 1160 (s). HRMS-ESI (m/z): $[M-H]^-$ calcd for $\text{C}_{13}\text{H}_{10}\text{N}_2\text{O}_6\text{S}$ 321.0181; found,

321.0183.



4-(4-Acetyl-benzenesulfonylamino)-benzoic acid: Following the general procedure, the

title compound was prepared in 93% yield. HPLC retention time: 7.57 min. ^1H NMR

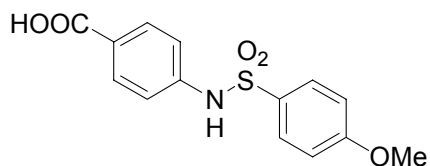
(500 MHz, DMSO-d_6 , δ): 12.77 (br. s, 1H), 10.97 (s, 1H), 8.10 (dt, $J = 8.5, 1.8$ Hz, 2H),

7.95 (dt, $J = 8.5, 1.8$ Hz, 2H), 7.81 (dt, $J = 8.8, 1.9$ Hz, 2H) 8.10 (dt, $J = 8.8, 1.9$ Hz, 2H),

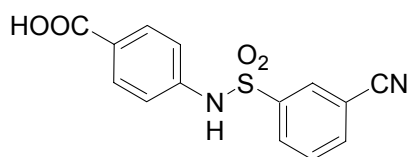
2.51 (s, 3H). ^{13}C NMR (125.7 MHz, DMSO-d_6 , δ): 197.1, 166.6, 142.7, 141.4, 139.9,

130.7, 129.1, 127.0, 125.9, 118.3, 26.9. IR (dry film, cm^{-1}): 3257 (m), 3004 (w), 2987

(w), 1676 (s), 1607 (m), 1275 (s), 1260 (s), 1160 (s). HRMS-ESI (m/z): [M-H]⁻ calcd for C₁₅H₁₃NO₅S 318.0436; found, 318.0438.

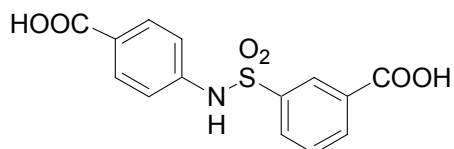


4-(4-Methoxy-benzenesulfonylamino)-benzoic acid: Following the general procedure, the title compound was prepared in 84% yield. HPLC retention time: 7.69 min. ¹H NMR (500 MHz, DMSO-d₆, δ): 12.71 (br. s, 1), 10.67 (br. s, 1H), 7.80 (dt, *J* = 8.8, 1.9 Hz, 2H), 7.76 (dt, *J* = 9.0, 2.1 Hz, 2H), 7.19 (dt, *J* = 8.8, 1.9 Hz, 2H), 7.08 (dt, *J* = 9.0, 2.1 Hz, 2H), 3.80 (s, 3H). ¹³C NMR (125.7 MHz, DMSO-d₆, δ): 166.6, 162.5, 142.1, 130.8, 130.6, 128.8, 125.3, 117.9, 114.4, 55.6. IR (dry film, cm⁻¹): 3261 (m), 3000 (w), 2985 (w), 1697 (s), 1607 (s), 1275 (s), 1260 (s), 1147 (s). HRMS-ESI (m/z): [M-H]⁻ calcd for C₁₄H₁₃NO₅S 306.0436; found, 306.0443.

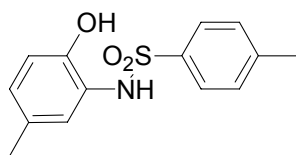


4-(3-Cyano-benzenesulfonylamino)-benzoic acid: Following the general procedure, the title compound was prepared in 65% yield. HPLC retention time: 7.66 min. ¹H NMR (500 MHz, DMSO-d₆, δ): 12.75 (br. s, 1H), 10.98 (br. s, 1H), 8.26 (t, *J* = 1.4 Hz, 1H), 8.19-8.06 (m, 2H), 7.91-7.76 (m, 3H), 7.22 (dt, *J* = 8.7, 1.8 Hz, 2H). ¹³C NMR (125.7 MHz, DMSO-d₆, δ): 166.5, 141.1, 140.4, 136.8, 130.9, 130.9, 130.7, 130.2, 126.1, 118.6, 117.2, 112.6. IR (dry film, cm⁻¹): 3252 (w), 3189 (w), 3005 (w), 2979 (w), 1674 (s),

1607 (m), 1338 (s), 1276 (s), 1156 (s). HRMS-ESI (m/z): [M-H]⁻ calcd for C₁₄H₁₀N₂O₄S 301.0283; found, 301.0286.



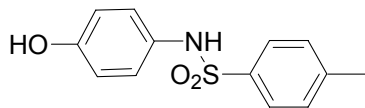
3-[[[(4-carboxyphenyl)amino]sulfonyl]-benzoic acid: Following the general procedure, the title compound was prepared in 96% yield. HPLC retention time: 7.11 min. ¹H NMR (500 MHz, DMSO-d₆, δ): 13.11 (br. s, 2H), 10.91 (s, 1H), 8.36 (t, *J* = 1.7 Hz, 1H), 8.16 (dt, *J* = 7.8, 1.3 Hz, 1H), 8.05 (ddd, *J* = 7.9, 1.7, 1.1 Hz, 1H), 7.83 (dt, *J* = 8.7, 1.8 Hz, 2H), 7.71 (t, *J* = 7.8 Hz, 1H), 7.22 (dt, *J* = 8.7, 1.8 Hz, 2H). ¹³C NMR (125.7 MHz, DMSO-d₆, δ): 166.6, 165.7, 141.5, 139.7, 133.6, 131.9, 130.7, 130.6, 130.0, 127.1, 125.9, 118.4. IR (dry film, cm⁻¹): 3265 (m), 3001 (w), 2990 (w), 1674 (s), 1607 (m), 1299 (s), 1275 (s), 1171 (s). HRMS-ESI (m/z): [M-H]⁻ calcd for C₁₄H₁₁NO₆S 320.0229; found, 320.0231.



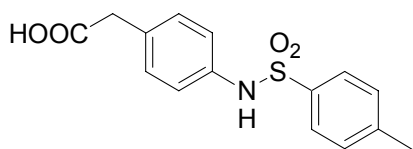
N-(2-Hydroxy-5-methyl-phenyl)-4-methyl-benzenesulfonamide: Following the general procedure, the title compound was prepared in 92% yield. HPLC retention time: 8.77 min. ¹H NMR (500 MHz, DMSO-d₆, δ): 9.26 (s, 1H), 9.03 (s, 1H), 7.62 (d, *J* = 8.3 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 6.96 (d, *J* = 1.9 Hz, 1H), 6.72 (dd, *J* = 8.6, 2.6 Hz, 1H), 6.59 (d, *J* = 8.1 Hz, 1H), 2.34 (s, 3H), 2.12 (s, 3H). ¹³C NMR (125.7 MHz, DMSO-d₆, δ): 147.6, 142.6, 137.7, 129.2, 127.3, 126.6, 126.3, 124.8, 123.7, 115.2, 20.8, 20.1. IR

(dry film, cm^{-1}): 3367 (m), 3246 (m), 2920 (w), 1320 (m), 1160 (s). HRMS-ESI (m/z):

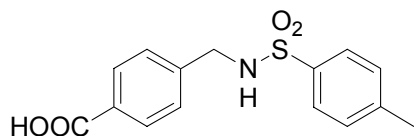
$[\text{M}+\text{H}]^+$ calcd for $\text{C}_{14}\text{H}_{15}\text{NO}_3\text{S}$ 278.0845; found, 278.0843.



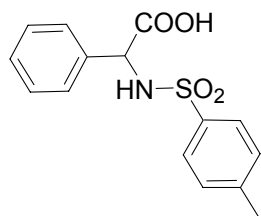
N-(4-Hydroxy-phenyl)-4-methyl-benzenesulfonamide: Following the general procedure, the title compound was prepared in 91% yield. HPLC retention time: 7.93 min. ^1H NMR (500 MHz, DMSO-d_6 , δ): 9.63 (s, 1H), 9.26 (s, 1H), 7.53 (d, $J = 8.3$ Hz, 2H), 7.31 (d, $J = 8.1$ Hz, 2H), 6.83 (dt, $J = 8.8, 2.2$ Hz, 2H), 6.58 (dt, $J = 8.8, 2.2$ Hz, 2H), 2.33 (s, 3H). ^{13}C NMR (125.7 MHz, DMSO-d_6 , δ): 153.2, 141.2, 135.2, 127.8, 127.0, 125.1, 122.3, 113.9, 19.2. IR (dry film, cm^{-1}): 3239 (m), 3005 (m), 2988 (m), 1331 (m), 1275 (s), 1261 (s), 1154 (m). HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{13}\text{H}_{13}\text{NO}_3\text{S}$, 264.0689; found, 264.0683.



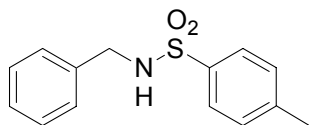
[4-(Toluene-4-sulfonylamino)-phenyl]-acetic acid: Following the general procedure, the title compound was prepared in 92% yield. HPLC retention time: 7.81 min. ^1H NMR (500 MHz, DMSO-d_6 , δ): 12.24 (br. s, 1H), 10.15 (s, 1H), 7.64 (d, $J = 8.3$ Hz, 2H), 7.33 (d, $J = 8.1$ Hz, 2H), 7.09 (d, $J = 8.5$ Hz, 2H), 7.01 (d, $J = 8.5$ Hz, 2H) 3.44 (s, 2H), 2.33 (s, 3H). ^{13}C NMR (125.7 MHz, DMSO-d_6 , δ): 172.4, 143.1, 136.7, 136.2, 130.5, 129.9, 129.6, 126.6, 119.8, 39.8, 20.8. IR (dry film, cm^{-1}): 3204 (br. m), 3005 (w), 1700 (s), 1330 (s), 1276 (s), 1146 (s). HRMS-ESI (m/z): $[\text{M}-\text{H}]^-$ calcd for $\text{C}_{15}\text{H}_{15}\text{NO}_4\text{S}$ 304.0638; found, 304.0651.



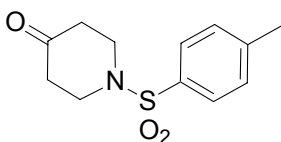
4-[(Toluene-4-sulfonylamino)-methyl]-benzoic acid: Following the general procedure, the title compound was prepared in 81% yield. HPLC retention time: 8.05 min. ^1H NMR (500 MHz, DMSO- d_6 , δ): 12.88 (br. s, 1H), 8.16 (t, $J = 6.4$ Hz, 1H), 7.84 (d, $J = 8.2$ Hz, 2H), 7.68 (d, $J = 8.2$ Hz, 2H), 7.36 (t, $J = 8.6$ Hz, 4H), 4.02 (d, $J = 6.4$ Hz, 2H), 2.38 (s, 3H). ^{13}C NMR (125.7 MHz, DMSO- d_6 , δ): 165.5, 141.3, 141.1, 136.2, 128.0, 127.7, 126.0, 124.9, 44.2, 19.3. IR (dry film, cm^{-1}): 3238 (w), 3005 (m), 2998 (m), 1679 (m), 1320 (m), 1275 (s), 1261 (s), 1158 (m). HRMS-ESI (m/z): $[\text{M}-\text{H}]^-$ calcd for $\text{C}_{15}\text{H}_{15}\text{NO}_4\text{S}$ 304.0638; found, 304.0637.



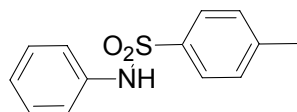
Phenyl-(toluene-4-sulfonylamino)-acetic acid: Following the general procedure, the title compound was prepared in 55% yield. HPLC retention time: 8.21 min. ^1H NMR (500 MHz, DMSO- d_6 , δ): 12.94 (br. s, 1H), 8.59 (d, $J = 8.7$ Hz, 1H), 7.61 (d, $J = 8.3$ Hz, 2H), 7.30-7.22 (m, 7H), 4.84 (d, $J = 8.5$ Hz, 1H), 2.33 (s, 3H). ^{13}C NMR (125.7 MHz, DMSO- d_6 , δ): 170.8, 142.3, 138.1, 136.7, 129.1, 128.2, 127.7, 127.2, 126.4, 59.5, 20.8. IR (dry film, cm^{-1}): 3005 (m), 2988 (m), 1723 (m), 1423 (m), 1275 (s), 1261 (s), 1162 (m). HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{15}\text{H}_{15}\text{NO}_4\text{S}$, 306.0795; found, 306.0793.



N-Benzyl-4-methyl-benzenesulfonamide: Following the general procedure, the title compound was prepared in 85% yield. HPLC retention time: 9.16 min. ^1H NMR (500 MHz, DMSO- d_6 , δ): 8.04 (t, $J = 6.3$ Hz, 1H), 7.69 (dt, $J = 8.2, 1.6$ Hz, 2H), 7.38 (d, $J = 8.1$ Hz, 2H), 7.32-7.18 (m, 5H), 3.95 (d, $J = 6.3$ Hz, 2H), 2.38 (s, 3H). ^{13}C NMR (125.7 MHz, DMSO- d_6 , δ): 142.4, 137.8, 137.6, 129.5, 128.1, 127.5, 127.0, 126.4, 46.0, 20.8. IR (dry film, cm^{-1}): 3267 (m), 3006 (9m), 2981 (m), 1322 (m), 1275 (s), 1160 (s). HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{14}\text{H}_{15}\text{NO}_2\text{S}$ 262.0896; found, 262.0904.



1-(Toluene-4-sulfonyl)-piperidin-4-one: Following the general procedure, the title compound was prepared in 90% yield. HPLC retention time: 8.02 min. ^1H NMR (500 MHz, DMSO- d_6 , δ): 7.68 (dt, $J = 8.3, 1.6$ Hz, 2H), 7.45 (dt, $J = 8.3, 1.6$ Hz, 2H), 3.29 (t, $J = 6.2$ Hz, 4H), 2.41 (t, $J = 6.2$ Hz, 4H), 2.41 (s, 3H). ^{13}C NMR (125.7 MHz, DMSO- d_6 , δ): 205.3, 143.6, 133.3, 129.9, 127.2, 44.9, 39.7, 20.9. IR (dry film, cm^{-1}): 3006 (w), 2988 (w), 1717 (s), 1361 (m), 1275 (s), 1161 (m). HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{12}\text{H}_{15}\text{NO}_3\text{S}$, 254.0845; found, 254.0855.



4-Methyl-N-phenyl-benzenesulfonamide: Following the general procedure while 5% $\text{Bu}_4\text{N}^+\text{Br}^-$ was added as the phase transfer reagent, the title compound was prepared in 94% yield. HPLC retention time: 9.01 min. ^1H NMR (500 MHz, DMSO- d_6 , δ): 10.19 (s, 1H), 7.63 (d, $J = 8.2$ Hz, 2H), 7.33 (d, $J = 8.0$ Hz, 2H), 7.21 (td, $J = 8.3, 1.0$ Hz, 2H),

7.08 (dd, $J = 7.5, 1.0$ Hz, 2H), 7.00 (td, $J = 7.5, 1.0$ Hz, 1H) 2.32 (s, 3H). ^{13}C NMR (125.7 MHz, DMSO- d_6 , δ): 143.1, 137.7, 136.7, 129.5, 129.0, 126.6, 123.8, 119.8, 20.8. IR (dry film, cm^{-1}): 3236 (s), 1597 (s), 1482 (s), 1335 (s), 1153 (s). HRMS-ESI (m/z): $[\text{M-H}]^-$ calcd for $\text{C}_{13}\text{H}_{13}\text{NO}_2\text{S}$ 246.0583; found, 246.0594.

