

Supporting Information

Cascade Synthesis of 2,4-Disulfonylpyrroles by Sulfonation/[2+3]-Cycloaddition Reactions of *gem*-Dibromoalkenes with Arylsulfonyl Methyl Isocyanides

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Table of Contents

General information-----	S02
General procedures for the synthesis of 3-aryl-2,4-disulfonyl-1H-pyrroles -----	S02
Characterization of products-----	S02
NMR Spectra-----	S10

General Information

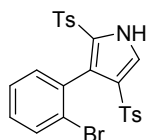
The solvents and chemicals were purchased from Merck and Aldrich chemical companies. Unless otherwise mentioned they were used without further purification. The 1,1-dibromoalkenes were prepared according to the reported procedures.¹ Melting points are taken on an Electrothermal 9100 apparatus and are uncorrected. FT-IR spectra were recorded on a Shimadzu Infra-Red Spectroscopy IR-435. Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker AVANCE Spectrometer 400 MHz for ¹H, 100 MHz for ¹³C in DMSO-*d*₆ as solvent. Mass spectra recorded on Agilent Technology (HP) 5973 Network Mass Selective Detector operating at an ionization potential of 70 eV and a Leco CHNS, model 932 was used for elemental analysis.

General Procedure for the Synthesis of 2,4-Disulfonylpyrroles by Reaction of *gem*-Dibromoalkenes with Arylmethyl Sulfonyl Isocyanides

To a mixture of respective *gem*-dibromoalkene **1** (1.0 mmol) and Cs₂CO₃ (652 mg, 2.0 mmol) in DMSO (4.0 mL) was added the corresponding arylmethyl sulfonyl isocyanide (2.0 mmol). Then the mixture was stirred at 100 °C for 3-6 h. Upon completion of the reaction, H₂O (20 mL) was added and the whole was extracted with CH₂Cl₂ (20 mL). The organic layer was washed with brine and dried (MgSO₄). The solvent was removed and the residue was purified by column chromatography using *n*-hexane/EtOAc (6:4) to obtain **3** in pure form.

Characterization of Products

3-(2-Bromophenyl)-2,4-ditosyl-1H-pyrrole (3a)

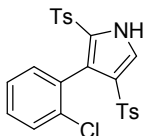


Yield: 412.6 mg (78%). White solid. M.p. = 105-107 °C. **FT-IR** (KBr): 3415, 3060, 2023, 2854, 1645, 1595, 1380, 1146 cm⁻¹.

¹H NMR (400 MHz, DMSO-*d*₆) δ = 13.48 (s, 1H), 7.90 (s, 1H), 7.56-7.54 (m, 1H), 7.47 (d, J = 8.0 Hz, 2H), 7.35 (d, J = 8.0 Hz, 4H), 7.26 (q, 4H), 6.82 (t, J = 4.4 Hz, 1H), 2.36 (s, 3H), 2.34 (s, 3H) ppm. **¹³C NMR** (100 MHz, DMSO-*d*₆) δ = 145.0, 144.1, 139.3, 138.3, 132.7,

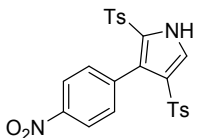
132.1, 131.5, 130.6, 130.3, 129.9, 128.5, 127.7, 127.5, 126.8, 126.2, 126.0, 125.3, 21.5, 21.4 ppm. **MS (EI)** m/z: calcd for $C_{24}H_{20}BrNO_4S_2$; 529.0, found, 530.45. **Anal. calcd.** For $C_{24}H_{20}BrNO_4S_2$; C, 54.34; H, 3.80; N, 2.64; S, 12.09%; Found; C, 54.43; H, 3.75; N, 2.60; S, 12.00%.

3-(2-Chlorophenyl)-2,4-ditosyl-1H-pyrrole (3b)



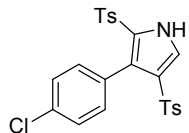
Yield: 392.8 mg (81%). White solid. M.p. = 131-134 °C. **FT-IR** (KBr): 3298, 2956, 1654, 1517, 1343, 1143 cm^{-1} . **1H NMR** (400 MHz, $DMSO-d_6$) δ = 13.46 (s, 1H), 7.87 (s, 1H), 7.44 (q, 3H), 7.35 (t, J = 7.0 Hz, 3H), 7.31-7.22 (m, 5H), 6.84 (d, J = 7.6 Hz, 1H), 2.36 (s, 3H), 2.34 (s, 3H) ppm. **^{13}C NMR** (100 MHz, $DMSO-d_6$) δ = 144.8, 144.0, 139.4, 138.5, 135.1, 132.9, 130.4, 130.3, 129.9, 129.7, 129.0, 128.2, 127.5, 127.4, 126.2, 125.3, 124.4, 21.5, 21.4 ppm. **MS (EI)** m/z: calcd for $C_{24}H_{20}ClNO_4S_2$; 485.05, found, 486.00. **Anal. calcd.** For $C_{24}H_{20}ClNO_4S_2$; C, 59.31; H, 4.15; N, 2.88; S, 13.19%; Found; C, 59.48; H, 4.25; N, 2.86; S, 13.00%.

3-(4-Nitrophenyl)-2,4-ditosyl-1H-pyrrole (3c)



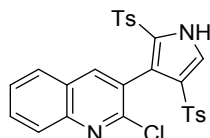
Yield 396.8 mg (80%). White solid. M.p. = 185-187 °C. **FT-IR** (KBr): 3444, 3063, 2923, 2854, 1645, 1596, 1383, 1147 cm^{-1} . **1H NMR** (400 MHz, $DMSO-d_6$) δ = 13.68 (s, 1H), 8.18 (d, J = 8.4 Hz, 2H), 7.96 (s, 1H), 7.46 (d, J = 8.0 Hz, 2H), 7.36 (d, J = 8.0 Hz, 2H), 7.29 (d, J = 8.9 Hz, 2H), 7.24 (d, J = 8.0 Hz, 2H), 7.10 (d, J = 8.4 Hz, 2H), 2.36 (s, 3H), 2.33 (s, 3H) ppm. **^{13}C NMR** (100 MHz, $DMSO-d_6$) δ = 147.7, 145.3, 144.4, 139.2, 138.3, 137.4, 132.5, 130.6, 130.2, 129.1, 128.1, 127.3, 125.9, 125.3, 122.7, 21.5, 21.4 ppm. **MS (EI)** m/z: calcd for $C_{24}H_{20}N_2O_6S_2$; 496.08, found, 496.55. **Anal. calcd.** For $C_{24}H_{20}N_2O_6S_2$; C, 58.05; H, 4.06; N, 5.64; S, 12.91%; Found; C, 58.13; H, 4.00; N, 5.60; S, 13.00%.

3-(4-Chlorophenyl)-2,4-ditosyl-1H-pyrrole (3d)



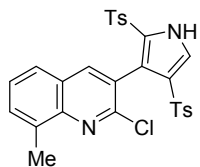
Yield: 412 mg (85%). White solid. M.p. = 150-153 °C. **FT-IR** (KBr): 3328, 2950, 1554, 1517, 1243, 1143, 870 cm^{-1} . **^1H NMR** (300 MHz, $\text{DMSO-}d_6$) δ = 13.61 (s, 1H), 8.20 (d, J = 8.7 Hz, 2H), 7.90 (d, J = 8.4 Hz, 2H), 7.49 (q, 4H), 7.38 (t, J = 7.05 Hz, 3H), 7.28 (d, J = 8.4 Hz, 2H), 2.45 (s, 3H), 2.35 (s, 3H) ppm. **^{13}C NMR** (75 MHz, $\text{DMSO-}d_6$) δ = 147.3, 145.1, 144.5, 139.9, 139.8, 138.4, 132.1, 131.2, 130.2, 130.0, 128.5, 127.2, 126.4, 124.5, 123.90, 123.1, 21.6, 21.5 ppm. **MS (EI)** m/z : calcd for $\text{C}_{24}\text{H}_{20}\text{ClNO}_4\text{S}_2$; 485.05, found, 486.00. **Anal. calcd.** For $\text{C}_{24}\text{H}_{20}\text{ClNO}_4\text{S}_2$; C, 59.31; H, 4.15; N, 2.88; S, 13.19%; Found; C, 59.53; H, 4.19; N, 2.80; S, 12.99%.

2-Chloro-3-(2,4-ditosyl-1H-pyrrol-3-yl)quinolone (3e)



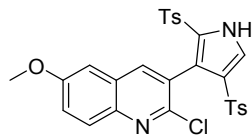
Yield: 483.3 mg (90%). White solid. M.p. = 106-109 °C. **FT-IR** (KBr): 3383, 3062, 2924, 1322, 1146 cm^{-1} . **^1H NMR** (500 MHz, CDCl_3) δ = 11.00 (s, 1H), 8.10 (t, J = 9.75 Hz, 1H), 7.85-7.72 (m, 4H), 7.64 (q, 1H), 7.32 (d, J = 8.0 Hz, 2H), 7.26 (t, J = 8.0 Hz, 2H), 7.07 (d, J = 8.0 Hz, 2H), 7.03 (d, J = 7.5 Hz, 2H), 2.32 (s, 6H) ppm. **^{13}C NMR** (125 MHz, CDCl_3) δ = 150.7, 147.4, 145.2, 144.3, 143.9, 141.8, 141.0, 138.0, 136.8, 131.2, 129.8, 129.5, 128.3, 128.0, 127.8, 127.7, 127.6, 127.5, 127.3, 126.1, 125.4, 122.6, 106.9, 106.8, 106.7, 21.6, 21.5 ppm. **MS (EI)** m/z : calcd for $\text{C}_{27}\text{H}_{21}\text{ClN}_2\text{O}_4\text{S}_2$; 536.06, found, 537.05. **Anal. calcd.** For $\text{C}_{27}\text{H}_{21}\text{ClN}_2\text{O}_4\text{S}_2$; C, 60.39; H, 3.94; N, 5.22; S, 11.94%; Found; C, 60.51; H, 4.15; N, 5.28; S, 12.09%.

2-Chloro-3-(2,4-ditosyl-1H-pyrrol-3-yl)-8-methylquinoline (3f)



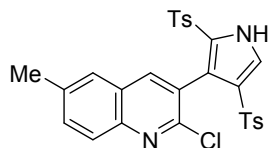
Yield: 458.2 mg (83%). White solid. M.p. = 107-110 °C. **FT-IR** (KBr): 3274, 3062, 2922, 1311, 1144 cm^{-1} . **$^1\text{H NMR}$** (400 MHz, $\text{DMSO-}d_6$) δ = 13.62 (s, 1H), 8.17(s, 1H), 7.89 (d, J = 8.4 Hz, 2H), 7.81 (d, J = 8 Hz, 1H), 7.73 (d, J = 6.8 Hz, 1H), 7.62-7.56 (m, 2H), 7.50 (d, J = 8.4 Hz, 2H), 7.44 (s, 1H), 7.38 (d, J = 8.4 Hz, 2H), 7.27 (d, J = 8 Hz, 2H), 2.70 (s, 3H), 2.45 (s, 3H), 2.36 (s, 3H) ppm. **$^{13}\text{C NMR}$** (100 MHz, $\text{DMSO-}d_6$) δ = 150.2, 146.0, 145.2, 144.5, 141.8, 139.7, 138.4, 135.9, 131.5, 131.1, 130.3, 129.8, 128.3, 127.7, 127.5, 126.7, 126.4, 126.1, 125.0, 124.9, 123.5, 21.6, 21.5, 17.9 ppm. **MS (EI)** m/z : calcd for $\text{C}_{28}\text{H}_{23}\text{ClN}_2\text{O}_4\text{S}_2$; 551.07, found, 552.08. **Anal. calcd.** For $\text{C}_{28}\text{H}_{23}\text{ClN}_2\text{O}_4\text{S}_2$; C, 61.03; H, 4.21; N, 5.08; S, 11.64%; Found; C, 60.99; H, 4.15; N, 5.13; S, 11.70%.

2-Chloro-3-(2,4-ditosyl-1H-pyrrol-3-yl)-6-methoxyquinoline (3g)



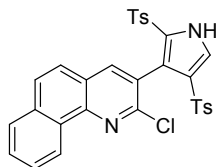
Yield: 481 mg (85%). White solid. M.p. = 121-124 °C. **FT-IR** (KBr): 3422, 3061, 2924, 1313, 1140 cm^{-1} . **$^1\text{H NMR}$** (400 MHz, $\text{DMSO-}d_6$) δ = 13.59 (s, 1H), 8.04 (d, J = 16.4 Hz, 1H), 7.94-7.88 (m, 3H), 7.50 (d, J = 8.4 Hz, 3H), 7.43 (d, J = 4.4 Hz, 1H), 7.35 (q, 3H), 7.27 (d, J = 7.2 Hz, 1H), 3.92 (s, 3H), 2.46 (s, 3H), 2.36 (s, 3H) ppm. **$^{13}\text{C NMR}$** (100 MHz, $\text{DMSO-}d_6$) δ = 158.4, 158.3, 148.4, 145.1, 144.4, 143.5, 142.8, 140.4, 139.7, 138.5, 130.3, 129.9, 129.6, 128.3, 127.9, 127.6, 127.5, 125.0, 123.8, 123.5, 106.4, 56.2, 21.6, 21.5 ppm. **MS (EI)** m/z : calcd for $\text{C}_{28}\text{H}_{23}\text{ClN}_2\text{O}_5\text{S}_2$; 566.07, found, 567.07. **Anal. calcd.** For $\text{C}_{28}\text{H}_{23}\text{ClN}_2\text{O}_5\text{S}_2$; C, 59.31; H, 4.09; N, 4.94; S, 11.31%; Found; C, 60.01; H, 4.15; N, 4.95; S, 11.25%.

2-Chloro-3-(2,4-ditosyl-1H-pyrrol-3-yl)-6-methylquinoline (3h)



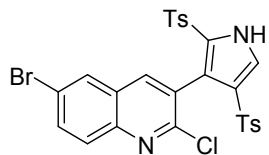
Yield: 446.3 mg (81%). White solid. M.p. = 103-106 °C. **FT-IR** (KBr): 3446, 3060, 1329, 1145 cm^{-1} . **$^1\text{H NMR}$** (400 MHz, $\text{DMSO-}d_6$) δ = 13.70 (s, 1H), 8.03 (d, J = 4.8 Hz, 1H), 7.95 (q, 1H), 7.81 (s, 1H), 7.74 (t, J = 7.0 Hz, 2H), 7.51 (q, 3H), 7.34 (d, J = 8.0 Hz, 2H), 7.28 (d, J = 8.0 Hz, 2H), 7.20 (d, J = 8.0 Hz, 2H), 2.57 (s, 3H), 2.34 (s, 3H), 2.32 (s, 3H) ppm. **$^{13}\text{C NMR}$** (100 MHz, $\text{DMSO-}d_6$) δ = 149.7, 145.2, 144.3, 140.6, 139.3, 138.1, 137.7, 133.8, 130.5, 130.1, 129.2, 128.2, 127.9, 127.6, 127.5, 127.4, 127.30, 126.4, 125.7, 123.9, 122.7, 21.6, 21.5, 21.4 ppm. **MS (EI)** m/z : calcd for $\text{C}_{28}\text{H}_{23}\text{ClN}_2\text{O}_4\text{S}_2$; 550.08, found, 551.07. **Anal. calcd.** For $\text{C}_{28}\text{H}_{23}\text{ClN}_2\text{O}_4\text{S}_2$; C, 61.03; H, 4.21; N, 5.08; S, 11.64%; Found; C, 60.98; H, 4.25; N, 5.10; S, 11.70%.

2-Chloro-3-(2,4-ditosyl-1H-pyrrol-3-yl)benzo[h]quinolone (3i)



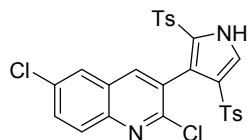
Yield: 487 mg (83%). White solid. M.p. = 161-164 °C. **FT-IR** (KBr): 3436, 3055, 2922, 1321, 1146 cm^{-1} . **$^1\text{H NMR}$** (400 MHz, $\text{DMSO-}d_6$) δ = 13.65 (s, 1H), 9.06-9.03 (m, 1H), 8.28 (s, 1H), 8.12 (q, 1H), 8.07 (d, J = 8.8 Hz, 1H), 7.90 (t, J = 8.6 Hz, 3H), 7.84 (q, 2H), 7.51 (d, J = 10.0 Hz, 3H), 7.38 (d, J = 8.0 Hz, 2H), 7.26 (d, J = 8.0 Hz, 2H), 2.46 (s, 3H), 2.35 (s, 3H) ppm. **$^{13}\text{C NMR}$** (100 MHz, $\text{DMSO-}d_6$) δ = 162.9, 150.3, 145.3, 145.2, 144.5, 141.8, 139.6, 138.4, 134.1, 130.3, 129.9, 129.8, 129.6, 128.8, 128.7, 128.3, 128.2, 127.5, 127.0, 125.3, 125.0, 124.9, 124.8, 124.2, 123.4, 21.6, 21.5 ppm. **MS (EI)** m/z : calcd for $\text{C}_{31}\text{H}_{23}\text{ClN}_2\text{O}_4\text{S}_2$; 586.08, found, 587.11. **Anal. calcd.** For $\text{C}_{31}\text{H}_{23}\text{ClN}_2\text{O}_4\text{S}_2$; C, 63.42; H, 3.95; N, 4.77; S, 10.92%; Found; C, 63.49; H, 4.02; N, 4.56; S, 10.88%.

6-Bromo-2-chloro-3-(2,4-ditosyl-1H-pyrrol-3-yl)quinolone (3j)



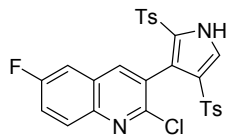
Yield 503 mg (82%). White solid. M.p. = 116-119 °C. **FT-IR** (KBr): 3423, 3106, 2924, 1332, 1154 cm^{-1} . **$^1\text{H NMR}$** (400 MHz, $\text{DMSO-}d_6$) δ = 13.77 (s, 1H), 8.16 (q, 1H), 8.11-8.06 (m, 2H), 7.97-7.89 (m, 2H), 7.52 (q, 2H), 7.35 (d, J = 8.4 Hz, 2H), 7.30 (t, J = 4.0 Hz, 2H), 7.21 (d, J = 8.0 Hz, 2H), 2.35 (s, 3H), 2.33 (s, 3H) ppm. **$^{13}\text{C NMR}$** (100 MHz, $\text{DMSO-}d_6$) δ = 151.2, 145.9, 145.5, 145.3, 145.0, 144.4, 140.5, 139.5, 139.2, 137.9, 132.4, 132.3, 130.6, 130.2, 129.3, 128.4, 128.3, 127.6, 127.5, 127.4, 127.3, 125.7, 125.3, 123.6, 122.1, 21.5, 21.4 ppm. **MS (EI)** m/z : calcd for $\text{C}_{27}\text{H}_{20}\text{BrClN}_2\text{O}_4\text{S}_2$; 613.97, found, 615.94. **Anal. calcd.** For $\text{C}_{27}\text{H}_{20}\text{ClN}_2\text{O}_4\text{S}_2$; C, 52.65; H, 3.27; N, 4.55; S, 10.41%; Found; C, 52.55; H, 3.34; N, 4.51; S, 10.12%.

2,6-Dichloro-3-(2,4-ditosyl-1H-pyrrol-3-yl)quinolone (3k)



Yield: 485.7 mg (85%). White solid. M.p. = 134-137 °C. **FT-IR** (KBr): 3435, 3135, 3064, 2926, 1319, 1170 cm^{-1} . **$^1\text{H NMR}$** (400 MHz, $\text{DMSO-}d_6$) δ = 13.65 (s, 1H), 8.25 (s, 1H), 8.14 (t, J = 2.2 Hz, 1H), 8.05 (q, 1H), 7.91-7.87 (m, 4H), 7.50 (d, J = 8.0 Hz, 2H), 7.46 (d, J = 3.2 Hz, 1H), 7.37 (q, 2H), 7.27 (d, J = 7.6 Hz, 1H), 2.45 (s, 3H), 2.36 (s, 3H) ppm. **$^{13}\text{C NMR}$** (100 MHz, $\text{DMSO-}d_6$) δ = 151.8, 145.8, 145.7, 145.3, 145.2, 144.5, 140.7, 139.6, 138.4, 132.4, 132.3, 132.1, 130.3, 129.9, 128.3, 127.6, 127.5, 127.1, 125.1, 124.9, 123.0, 21.6, 21.5 ppm. **MS (EI)** m/z : calcd for $\text{C}_{27}\text{H}_{20}\text{Cl}_2\text{N}_2\text{O}_4\text{S}_2$; 570.02, found, 571.49. **Anal. calcd.** For $\text{C}_{27}\text{H}_{20}\text{Cl}_2\text{N}_2\text{O}_4\text{S}_2$; C, 56.75; H, 3.53; N, 4.90; S, 11.22%; Found; C, 57.01; H, 3.45; N, 4.85; S, 11.18%.

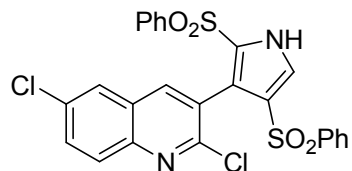
2-Chloro-3-(2,4-ditosyl-1H-pyrrol-3-yl)-6-fluoroquinoline (3l)



Yield: 459.8 mg (83%). White solid. M.p. = 144-147 °C. **FT-IR** (KBr): 3345, 3125, 3046, 2962, 1319, 1170 cm^{-1} . **$^1\text{H NMR}$** (300 MHz, $\text{DMSO-}d_6$) δ = 13.74 (s, 1H), 8.06 (s, 1H), 8.94 (s, 1H), 7.85 (d, J = 9 Hz, 2H), 7.51 (d, J = 7.8 Hz, 2H), 7.50 (d, J = 8.0 Hz, 2H), 7.33 (q, J = 7.6 Hz, 5H), 7.22 (d, J = 7.8 Hz, 2H), 2.35 (s, 3H), 2.33 (s, 3H) ppm. **$^{13}\text{C NMR}$** (75 MHz, $\text{DMSO-}d_6$) δ = 150.2, 145.3, 144.4, 144.2, 139.2,

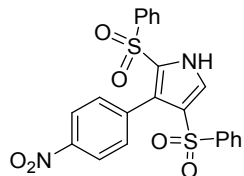
138.0, 131.1, 131.0, 130.5, 130.2, 129.3, 128.3, 127.5, 127.3, 125.7, 125.1, 122.2, 121.9, 121.5, 21.5, 21.4 ppm. **MS (EI)** m/z: calcd for $C_{27}H_{20}ClFN_2O_4S_2$; 554.05, found, 555.04. **Anal. calcd.** For $C_{27}H_{20}ClFN_2O_4S_2$; C, 58.43; H, 3.63; N, 5.05; S, 11.55%; Found; C, 58.21; H, 3.57; N, 4.89; S, 11.38%.

3-(2,5-Bis(phenylsulfonyl)-1H-pyrrol-3-yl)-2,6-dichloroquinoline (3m)



Yield: 434.7 mg (80%). White solid. M.p. = 148-151 °C. **FT-IR** (KBr): 3335, 3085, 2915, 1328, 1172 cm^{-1} . **1H NMR** (300 MHz, DMSO- d_6) δ = 13.75 (s, 1H), 8.28 (d, J = 13.5 Hz, 1H), 8.15 (s, 1H), 8.08-8.00 (m, 3H), 7.90 (dd, J = 2.1 Hz, J = 8.85 Hz, 1H), 7.82-7.63 (m, 4H), 7.5 (q, 5H) ppm. **^{13}C NMR** (75 MHz, DMSO- d_6) δ = 151.7, 145.9, 145.4, 142.4, 141.3, 140.8, 140.1, 134.5, 134.0, 132.3, 132.1, 130.3, 129.9, 129.5, 128.2, 127.4, 127.3, 127.2, 125.4, 124.7, 123.2 ppm. **MS (EI)** m/z: calcd for $C_{25}H_{16}Cl_2N_2O_4S_2$; 541.99, found, 543.43. **Anal. calcd.** For $C_{25}H_{16}Cl_2N_2O_4S_2$; C, 55.26; H, 2.97; N, 5.16; S, 11.80%; Found; C, 55.38; H, 3.13; N, 5.04; S, 11.77%.

3-(4-Nitrophenyl)-2,4-bis(phenylsulfonyl)-1H-pyrrole (3n)



Yield: 408 mg (87%). White solid. M.p. = 183-186 °C. **FT-IR** (KBr): 3446, 3053, 2922, 2855, 1650, 1586, 1373, 1174 cm^{-1} . **1H NMR** (300 MHz, DMSO- d_6) δ = 13.76 (s, 1H), 8.16 (d, J = 8.7 Hz, 2H), 8.01 (s, 1H), 7.57 (q, 4H), 7.48-7.40 (m, 4H), 7.09 (d, J = 8.7 Hz, 2H) ppm. **^{13}C NMR** (75 MHz, DMSO- d_6) δ = 147.8, 141.9, 141.2, 137.2, 134.5, 133.9, 132.5, 130.2, 129.7, 128.9, 128.4, 127.2, 125.7, 125.6, 122.7

ppm. **MS (EI)** m/z: calcd for C₂₂H₁₆N₂O₆S₂; 468.04, found, 468.50. **Anal. calcd.** For C₂₂H₁₆N₂O₆S₂; C, 56.40; H, 3.44; N, 5.98; S, 13.69%; Found; 56.38; H, 3.52; N, 5.84; S, 13.74%.

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