Supporting Information

A simple and Efficient Method for Constructing Azepino[4,5-b]indole Derivatives via Acid Catalysis

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General information:

All chemicals were purchased form best grade in commercially available and are used without further purification. All solvents were purchased as HPLC grade and used without any purification or distillation. Analytical thin layer chromatography was performed on aluminium plate coated with silica gel (Merck). Gravity column chromatography was performed using 100-200 mesh silica gel and mixtures of hexane-ethyl acetate were used for elution. Visualization was accomplished using ultraviolet light (254 nm) and chemical staining with acidic potassium permanganate solution and Iodine.

Melting points was determined using “Mel-Temp” melting point apparatus and were uncorrected. Proton nuclear magnetic resonance (\(^1\)H NMR) and Carbon nuclear magnetic resonance (\(^{13}\)C NMR) spectra’s were recorded using a Varian (400 MHz) and Oxford (400 MHz) spectrometer. All spectra were recorded at ambient temperature (298 K). Chemical shifts (δ) are quoted in ppm relative to residual solvent (CDCl\(_3\): δ = 7.26 ppm for \(^1\)H and δ = 77.0 for 13C). Coupling constants (\(J\)) are corrected and quoted to the nearest 0.1 Hz. The following abbreviations are used to indicate the multiplicity of the signals: s = singlet; d = doublet; t = triplet; q = quartet; m = multiplet; bs = broad singlet; dt = doublet of triplet; td = triplet of doublet; ddd = doublet of doublet of doublet. High resolution mass spectra (HRMS) were measured on a Micromass Q-TOF spectrometer using EI (electron impact, 70 eV) at the Joint Center for High valued Instruments, National Sun-Yat Sen University, Kaohsiung, Taiwan.

General experiment procedure for synthesis of starting materials: (1a-s)\(^{1-3}\)
To a solution of tryptamine (S1, 5 mmol) in DCM (50 mL) was added with triethyl amine (5 equiv) followed by tosyl chloride (1.5 equiv) at 0°C. Then the reaction mixture was stirred at RT for 8 hr and reaction monitored by TLC. After reaction completed, reaction mass portioned between water and dichloromethane. The combined organic layer was washed with ammonium chloride solution and dried on sodium sulphate and evaporated under vacuum to obtain crude. The crude was purified through column chromatography (100-200 silica gel; EA/Hexane).

Then the obtained compound (S2; 2 mmol) in DMF (10 ml) was added to K2CO3 (1.5 equiv) at 0°C and stirred at same temperature for 30 min. Then the respective 2-bromoacetophenones (S3, 1.2 equiv) were added and slowly heated to RT and stirred till the completion of starting materials (~30 min to 2 hr). After reaction completed (monitored by TLC), reaction mass quenched with ice-cold water and extracted into EtOAc. The combined organic layer was washed with ice-cold water and sat. NH4Cl. Then it was dried and evaporated to give crude residue. The obtained crude material passed through flash column chromatography to give pure product in high yields (1a-s).

**General Experiment Procedure for Compound 2a-s:**

An oven-dried 50 mL RB flask charged with 1 (0.5 mmol) in 1,2-Dichloroethane (0.15 M) was added with TfOH (0.2 equiv), and stirred at respective temperature for given time. After reaction completed (monitored by TLC), reaction mass was partitioned between water and Dichloromethane, then the combined organic phases washed with brine solution, dried on sodium sulphate and evaporated under vacuum. The resulting crude material was purified by flash column chromatography on silica gel (100-200 mesh) with suitable ratio of hexane and ethyl acetate to afford the desired product (3). The identity and purity of the compounds were determined by 1HNMR, 13CNMR, and HRMS data.
Spectral Characterization Data for Final Compounds (2a-v):

5-Phenyl-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-b]indole (2a): Yield: 96%; yellow solid; m.p. 192-194 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.73 (d, \(J = 8.4\) Hz, 2H), 7.54 (s, 1H), 7.49-7.40 (m, 6H), 7.29 (d, \(J = 8.4\) Hz, 2H), 7.16-7.03 (m, 3H), 6.88 (s, 1H), 3.88 (t, \(J = 4.4\) Hz, 2H), 3.04 (t, \(J = 4.4\) Hz, 2H), 2.39 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 144.0, 138.6, 136.1, 134.6, 130.6, 129.9, 129.7, 128.9, 128.4, 128.0, 126.9, 126.6, 122.3, 119.6, 118.1, 117.9, 113.6, 110.6, 46.3, 26.4, 21.5; HRMS-ESI (m/z): calcd for C\(_{25}\)H\(_{23}\)N\(_2\)O\(_2\)S [M+H]^+ : 415.1402 found 415.1474.

5-(4-Methoxyphenyl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-b]indole (2b): Yield: 92%; brown solid; m.p. 184-186 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.73 (d, \(J = 8.4\) Hz, 2H), 7.54 (s, 1H), 7.43 (d, \(J = 7.6\) Hz, 1H), 7.34 (d, \(J = 8.4\) Hz, 2H), 7.29 (d, \(J = 8.4\) Hz, 2H), 7.16-6.97 (m, 5H), 6.84 (s, 1H), 3.88 (t, \(J = 3.2\) Hz, 5H), 3.04 (t, \(J = 5.2\) Hz, 2H), 2.36 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 159.5, 144.0, 136.3, 134.7, 131.14, 131.01, 130.86, 130.5, 128.59, 127.06, 126.41, 124.45, 119.7, 118.0, 117.9, 114.4, 113.6, 110.7, 55.5, 46.4, 29.7, 26.5, 21.6; HRMS-ESI (m/z): calcd for C\(_{26}\)H\(_{25}\)N\(_2\)O\(_3\)S [M+H]^+ : 445.1508 found 445.1590.

5-(2-Methoxyphenyl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-b]indole (2c): Yield: 75%; yellow solid; m.p. 125-127 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.75 (d, \(J = 8.0\) Hz, 2H), 7.45-7.39 (m, 3H), 7.30-7.24 (m, 3H), 7.13-7.01 (m, 5H), 6.83 (s, 1H), 3.92 (t, \(J = 5.2\) Hz, 2H), 3.73 (s, 3H), 3.02 (t, \(J = 5.2\) Hz, 2H), 2.39 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 158.0, 143.9, 136.7, 134.8, 132.4, 131.5, 129.9, 128.7, 127.1, 122.14, 122.12, 119.5, 118.0, 114.8, 112.8, 111.5, 110.6, 55.8, 46.3, 26.4, 21.6; HRMS-ESI (m/z): calcd for C\(_{26}\)H\(_{25}\)N\(_2\)O\(_3\)S [M+H]^+ : 445.1508 found 445.1566.

5-(p-Ttolyl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-b]indole (2d): Yield: 95%; yellow solid; m.p. 159-161 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.76 (d, \(J = 8.4\) Hz, 2H), 7.60 (s, 1H), 7.46 (d, \(J = 7.6\) Hz, 1H), 7.35-7.26 (m, 6H), 7.18-7.06 (m, 3H), 6.89 (s, 1H), 3.90 (t, \(J = 4.8\) Hz, 2H), 3.07 (t, \(J = 4.8\) Hz, 2H), 2.46 (s, 3H), 2.41 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 143.9, 137.7, 136.2, 135.5, 134.6, 130.8, 129.9, 129.6, 129.5, 128.4, 126.9, 126.3, 122.3, 119.5, 118.1, 117.9, 113.5, 110.5, 46.3, 26.4, 21.5, 21.1 HRMS-ESI (m/z): calcd for C\(_{26}\)H\(_{25}\)N\(_2\)O\(_2\)S [M+H]^+ : 429.1558 found 429.1624.
5-(4-Chlorophenyl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-b]indole (2e): Yield: 89%; yellow solid; m.p. 161-163 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.73 (d, $J = 8.4$ Hz, 2H), 7.48-7.43 (m, 4H), 7.38 (d, $J = 4$ Hz, 2H), 7.33 (d, $J = 8.0$ Hz, 2H), 7.17 (d, $J = 8.0$ Hz, 1H), 7.12 (t, $J = 8.0$ Hz, 1H), 7.07 (t, $J = 8.0$ Hz, 1H), 6.86 (s, 1H), 3.87 (t, $J = 4.0$ Hz, 2H), 3.04 (t, $J = 4.0$ Hz, 2H), 2.40 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 144.2, 137.1, 136.1, 134.8, 131.1, 130.3, 130.1, 129.3, 128.4, 127.0, 126.8, 122.6, 119.8, 118.0, 116.8, 114.1, 110.7, 46.4, 26.5, 21.6; HRMS-ESI (m/z): calcd for C$_{25}$H$_{22}$ClN$_2$O$_2$S [M+H]$^+$ : 449.1012 found 449.1080.

5-(3,4-Dichlorophenyl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-b]indole (2f): Yield: 82%; brown solid; m.p. 210-212 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.74 (d, $J = 8.8$ Hz, 2H), 7.55 (dd, $J = 6.4$, 4.0 Hz, 2H), 7.44 (d, $J = 7.6$ Hz, 2H), 7.33 (d, $J = 8.0$ Hz, 2H), 7.30-7.26 (m, 1H), 7.21 (t, $J = 6.8$ Hz, 1H), 7.08 (t, $J = 8.0$ Hz, 1H), 6.89 (s, 1H), 3.86 (t, $J = 4.8$ Hz, 2H), 3.03 (t, $J = 4.8$ Hz, 2H), 2.42 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 144.3, 138.7, 135.9, 134.9, 134.4, 131.6, 131.0, 129.1, 128.4, 127.16, 127.11, 122.8, 120.0, 118.0, 115.5, 114.4, 110.8, 46.5, 26.4, 21.6; HRMS-ESI (m/z): calcd for C$_{25}$H$_{21}$Cl$_2$N$_2$O$_2$S [M+H]$^+$ : 483.0623 found 483.0679.

5-(4-Fluorophenyl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-b]indole (2g): Yield: 80%; brown solid; m.p. 158-160 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.73 (d, $J = 8.4$ Hz, 2H), 7.45-7.38 (m, 4H), 7.30 (d, $J = 8.0$ Hz, 2H), 7.25 (s, 1H), 7.18-7.04 (m, 5H), 6.85 (s, 1H), 3.87 (t, $J = 4.4$ Hz, 2H), 3.04 (t, $J = 4.4$ Hz, 2H), 2.40 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 162.5 ($F = 245.8$ Hz), 144.1, 136.1, 134.7, 134.5, 134.4, 131.4 (d, $J = 10.0$ Hz), 130.5, 130.0, 128.4, 126.9, 126.6, 122.5, 119.7, 117.9, 116.9, 116.4 (d, $J = 20.0$ Hz), 113.8, 110.6, 46.3, 26.4, 21.5; HRMS-ESI (m/z): calcd for C$_{25}$H$_{21}$FN$_2$O$_2$S [M+H]$^+$ : 432.1308 found 432.1291.

5-(Naphthalen-2-yl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-b]indole (2h): Yield: 75%; yellow solid; m.p. 207-209 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.97-7.89 (m, 4H), 7.77 (d, $J = 8.4$ Hz, 2H), 7.60-7.54 (m, 4H), 7.48 (d, $J = 8.4$ Hz, 1H), 7.32 (d, $J = 8.0$ Hz, 2H), 7.15-7.07 (m, 3H), 7.01 (s, 1H), 3.94 (t, $J = 4.4$, 4.0 Hz, 2H), 3.11 (t, $J = 4.4$, 4.0 Hz, 2H), 2.42 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 144.04, 136.14, 135.98, 134.74, 133.50, 132.82, 130.73, 129.97, 128.62, 128.65, 128.40, 127.98, 127.73, 127.66, 126.98, 126.87, 126.66, 126.44, 122.42, 119.65, 118.10, 117.95, 113.81, 110.65, 46.47, 26.51, 21.52; HRMS-ESI (m/z): calcd for C$_{29}$H$_{25}$N$_2$O$_2$S [M+H]$^+$ : 465.1558 found 465.1613.
4-Methyl-5-phenyl-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-b]indole (2k): Yield: 57%; white solid; m.p. 201-203 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.59 (d, $J = 8.0$ Hz, 2H), 7.49-7.42 (m, 3H), 7.37 (d, $J = 8.0$ Hz, 1H), 7.23 (d, $J = 8.4$ Hz, 2H), 7.10-7.00 (m, 3H), 6.85 (d, $J = 8.0$ Hz, 3H), 4.01 (s, 2H), 3.27 (t, $J = 8.0$ Hz, 2H), 2.05 (d, $J = 8.0$ Hz, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 143.0, 138.1, 136.1, 134.6, 131.4, 130.0, 129.6, 129.1, 128.6, 128.3, 126.6, 122.8, 119.4, 118.6, 113.9, 110.3, 49.7, 26.4, 21.9, 21.2; HRMS-ESI (m/z): calcd for C$_{26}$H$_{24}$NaN$_2$O$_2$S [M+Na]$^+$ : 451.1558 found 451.1450.

4,5-Diphenyl-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-b]indole (2l): Yield: 30%; yellow solid; m.p. 274-276 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.53 (d, $J = 8.0$ Hz, 1H), 7.38 (s, 1H), 7.30-7.24 (m, 4H), 7.21-7.13 (m, 4H), 7.13-7.09 (m, 1H), 7.02-6.97 (m, 3H), 6.94-6.89 (m, 4H), 4.13 (s, 2H), 3.53 (s, 2H), 2.23 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 142.6, 137.9, 137.7, 137.5, 135.0, 130.9, 130.6, 130.5, 128.9, 128.8, 128.69, 128.67, 127.6, 127.1, 127.0, 126.9, 123.1, 119.6, 118.9, 114.4, 110.5, 49.9, 29.7, 28.5, 21.3; HRMS-ESI (m/z): calcd for C$_{31}$H$_{26}$NaN$_2$O$_2$S [M+Na]$^+$ : 513.1715 found 513.1096.

5,6-Diphenyl-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-b]indole (2m): Yield: 45%; yellow solid; m.p. 126-128 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.74 (d, $J = 8.0$ Hz, 2H), 7.54-7.52 (m, 1H), 7.31 (d, $J = 8.0$ Hz, 2H), 7.16-7.00 (m, 7H), 6.95-6.90 (m, 5H), 6.82-6.80 (m, 2H), 4.06 (t, $J = 4.8$ Hz, 2H), 3.09 (t, $J = 4.8$ Hz, 2H), 2.41 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 144.0, 139.9, 138.7, 138.5, 134.1, 129.8, 128.5, 128.4, 34.1, 127.9, 127.6, 127.5, 127.2, 126.9, 126.4, 126.3, 122.7, 120.3, 119.6, 118.8, 117.5, 110.5, 53.2, 29.6, 24.1, 21.5; HRMS-ESI (m/z): calcd for C$_{31}$H$_{27}$N$_2$O$_2$S [M+H]$^+$ : 491.1715 found 491.1779.

5-Phenyl-3-(phenylsulfonyl)-1,2,3,6-tetrahydroazepino[4,5-b]indole (2n): Yield: 79 %; yellow solid; m.p. 166-168 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.89-7.86 (m, 2H), 7.60-7.42 (m, 10H), 7.18-7.05 (m, 3H), 6.89 (s, 1H), 3.92 (t, $J = 4.4$ Hz, 2H), 3.86 (t, $J = 4.4$ Hz, 2H), 2.41 (t, $J = 5.2$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 139.1, 138.5, 134.6, 133.0, 130.5, 129.7, 129.3, 129.0, 128.4, 128.0, 126.9, 126.4, 122.4, 119.6, 118.4, 117.9, 113.7, 110.6, 46.4, 26.5; HRMS-ESI (m/z): calcd for C$_{24}$H$_{21}$N$_2$O$_2$S [M+H]$^+$ : 401.1245 found 401.1313.

3-((2-Nitrophenyl)sulfonyl)-5-phenyl-1,2,3,6-tetrahydroazepino[4,5-b]indole (2o): Yield: 75%; red solid; m.p. 205-207 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 8.07-8.05 (m, 1H), 7.73-7.65 (m, 3H), 7.60 (s, 1H), 7.52-7.43 (m, 6H), 7.19-7.06 (m, 3H), 6.81 (s, 1H), 4.26 (t, $J = 4.8$ Hz, 2H), 3.19(t, $J = 5.2$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 148.1, 138.2, 134.7, 134.0, 132.1, 131.9, 130.6, 130.3, 129.6, 129.7, 129.0, 128.3, 128.2, 125.8, 124.5, 122.7, 119.76, 119.74, 118.1, 113.8, 110.7, 46.8, 26.7; HRMS-ESI (m/z): calcd for C$_{24}$H$_{19}$N$_3$O$_4$S [M+H]$^+$ : 446.1096 found 446.1174.
3-benzyl-5-phenyl-1,2,3,6-tetrahydroazepino[4,5-b]indole (2p): Yield: 30%; brown oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 8.61 (bs, 1H), 7.44-7.33 (m, 11H), 7.32-7.24 (m, 1H), 7.04-7.00 (m, 2H), 6.40 (s, 2H), 3.43 (t, $J = 8.4$ Hz, 2H), 3.09-3.05 (m, 2H); HRMS-ESI (m/z): calcd for C$_{25}$H$_{22}$N$_2$ [M]$^+$ : 350.1783 found 350.1777. (Note: Compound started decomposing over a period of time; not enough stable to record $^{13}$C NMR)

Methyl 5-phenyl-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-b]indole-2-carboxylate (2q): Yield: 82%; yellow solid; m.p. 88-90 ºC; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.60 (d, $J = 8.4$ Hz, 2H), 7.55 (s, 1H), 7.49-7.40 (m, 6H), 7.32 (d, $J = 8.0$ Hz, 2H), 7.15-7.05 (m, 3H), 6.99 (d, $J = 0.8$ Hz, 1H), 5.82 (t, $J = 4.8$ Hz, 1H), 3.93 (dd, $J = 16.0$, 4.8 Hz, 1H), 3.28 (s, 3H), 2.81 (dd, $J = 16.0$, 4.8 Hz, 1H), 1.24 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 167.9, 144.3, 138.6, 135.5, 135.0, 131.1, 129.8, 129.6, 128.9, 128.3, 127.9, 127.2, 124.3, 122.3, 119.9, 118.6, 117.6, 116.4, 110.6, 57.6, 52.0, 28.2, 21.5; HRMS-ESI (m/z): calcd for C$_{27}$H$_{25}$N$_2$O$_4$S [M+H]$^+$ : 473.1457 found 473.1530.

Methyl 5-(4-methoxyphenyl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-b]indole-2-carboxylate (2r): Yield: 89%; yellow solid; m.p. 88-90 ºC; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.74 (d, $J = 8.4$ Hz, 2H), 7.51 (s, 1H), 7.46 (d, $J = 6.4$ Hz, 1H), 7.34-7.29 (m, 4H), 7.14-7.03 (m, 3H), 6.98-6.92 (m, 3H), 5.56 (t, $J = 3.6$ Hz, 1H), 3.90 (dd, $J = 16.0$, 4.8 Hz, 1H), 3.87 (s, 3H), 3.27 (s, 3H), 2.78 (dd, $J = 16.0$, 4.8 Hz, 1H), 2.40 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 168.0, 159.4, 144.2, 135.6, 134.9, 131.5, 130.87, 130.82, 129.8, 128.4, 127.2, 123.9, 119.6, 117.6, 116.1, 114.2, 110.5, 110.4, 57.6, 55.3, 52.0, 28.2, 21.5; HRMS-ESI (m/z): calcd for C$_{28}$H$_{26}$NaN$_2$O$_5$S [M+Na]$^+$ : 525.1562 found 525.1458.

Methyl 5-(4-chlorophenyl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-b]indole-2-carboxylate (2s): Yield: 69%; yellow solid; m.p. 94-96 ºC; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.60 (d, $J = 8.4$ Hz, 2H), 7.47-7.40 (m, 4H), 7.46-7.31 (m, 4H), 7.15-7.04 (m, 3H), 6.96 (d, $J = 0.8$ Hz, 1H), 5.56 (t, $J = 3.2$ Hz, 1H), 3.91 (dd, $J = 16.0$, 4.8 Hz, 1H) 3.25 (s, 3H), 2.77 (dd, $J = 16.0$, 4.8 Hz, 1H), 2.41 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 167.8, 144.4, 137.1, 135.4, 135.1, 133.9, 130.9, 130.8, 129.9, 129.1, 128.3, 127.3, 124.5, 122.5, 119.8, 117.6, 114.9, 110.8, 110.6, 57.6, 52.1, 28.2, 21.6; HRMS-ESI (m/z): calcd for C$_{27}$H$_{24}$ClN$_2$O$_4$S [M+H]$^+$ : 507.1067 found 507.1154.
Methyl 5-(naphthalen-2-yl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-b]indole-2-carboxylate (2t): Yield: 86%; yellow solid; m.p. 116-118 °C; 1H NMR (400 MHz, CDCl₃) δ 7.91-7.86 (m, 4H), 7.56-7.47 (m, 5H), 7.31 (d, J = 8.4 Hz, 2H), 7.10-7.05 (m, 4H), 5.61-5.58 (m, 1H), 3.94 (dd, J = 16.0, 4.8 Hz, 1H), 3.29 (s, 3H), 2.82 (dd, J = 16.0, 4.8 Hz, 1H), 2.41 (s, 3H); 13C NMR (100 MHz, CDCl₃) δ 167.9, 144.3, 136.0, 135.5, 135.1, 133.5, 132.8, 131.3, 129.9, 128.5, 128.4, 128.3, 128.0, 127.7, 127.5, 127.3, 126.6, 126.3, 124.6, 122.3, 119.7, 117.6, 116.3, 110.7, 110.6, 57.7, 52.1, 28.2, 21.5; HRMS-ESI (m/z): calcd for C₃₁H₂₆NaN₂O₄S [M+Na]⁺: 545.1613 found 545.1510.

1,5-diphenyl-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-b]indole (2u): Yield: 75%; white solid; m.p. 237-239 °C; 1H NMR (400 MHz, CDCl₃) δ 7.62 (s, 1H), 7.55-7.46 (m, 7H), 7.27-7.14 (m, 8H), 7.07-7.03 (m, 1H), 6.79 (d, J = 8.0 Hz, 1H), 6.89-6.85 (m, 2H), 4.75 (dd, J = 6.4, 2.8 Hz, 1H), 4.20 (dd, J = 13.2, 6.4Hz, 1H), 3.85 (dd, J = 13.6, 2.8 Hz, 1H), 2.38 (s, 3H); 13C NMR (100 MHz, CDCl₃) δ 143.7, 138.4, 135.9, 134.8, 131.2, 129.8, 129.7, 129.0, 128.48, 128.43, 128.3, 128.2, 128.0, 127.0, 126.7, 122.3, 119.5, 119.3, 118.2, 115.7, 110.5, 53.4, 44.4, 21.4; HRMS-ESI (m/z): calcd for C₃₁H₂₆NaN₂O₂S [M+Na]⁺: 513.1715 found 513.1611.

5-Phenyl-1-(p-tolyl)-3-tosyl-1,2,3,6-tetrahydroazepino[4,5-b]indole (2v): Yield: 71%; white solid; m.p. 178-180 °C; 1H NMR (400 MHz, CDCl₃) δ 7.60 (s, 1H), 7.54-7.46 (m, 3H), 7.20-7.13 (m, 3H), 7.07-6.97 (m, 6H), 6.89-6.85 (m, 2H), 4.71 (dd, J = 6.8, 2.8 Hz, 1H), 4.18 (dd, J = 6.8, 2.8 Hz, 1H), 3.84 (dd, J = 13.4, 2.8 Hz, 1H), 3.56 (t, J = 8.0 Hz, 2H), 3.00 (t, J = 8.0 Hz, 2H), 2.40 (s, 3H); 13C NMR (100 MHz, CDCl₃) δ 143.6, 138.6, 138.5, 136.1, 136.0, 134.8, 131.1, 129.79, 129.70, 129.1, 129.0, 128.4, 128.2, 128.1, 128.0, 127.0, 122.3, 119.5, 119.3, 118.2, 115.9, 110.4, 53.4, 44.0, 21.5, 21.1; HRMS-ESI (m/z): calcd for C₃₂H₂₈NaN₂O₂S [M+Na]⁺: 527.1871 found 527.1761.

Spectral Characterization Data for Starting materials (1a-v):

N-(2-(1H-Indol-3-yl)ethyl)-4-methyl-N-(2-oxo-2-phenylethyl)benzenesulfonamide (1a): Yield: 90%; white solid; m.p. 139-141 °C; 1H NMR (400 MHz, CDCl₃) δ 8.02 (s, 1H), 7.82-7.79 (m, 2H), 7.75 (d, J = 8.0 Hz, 2H), 7.58-7.54 (m, 1H), 7.48 (d, J = 7.6 Hz, 1H), 7.41 (t, J = 8.4 Hz, 2H), 7.28 (dd, J = 16.0, 8.4 Hz, 3H), 7.14 (t, J = 8.0 Hz, 1H), 7.05 (t, J = 8.0 Hz, 1H), 6.95 (s, 1H), 4.70 (s, 2H), 3.56 (t, J = 8.0 Hz, 2H), 3.00 (t, J = 8.0 Hz, 2H), 2.40 (s, 3H); 13C NMR (100 MHz, CDCl₃) δ 194.3, 143.4, 136.8, 136.2, 134.9, 133.7, 129.6, 128.5, 128.0, 127.2, 122.2, 122.1, 119.5, 118.7, 112.4, 56.6, 48.8, 24.8, 21.6; HRMS-ESI (m/z): calcd for
C_{25}H_{24}NaN_{2}O_{3}S [M+Na]^+ : 455.1508 found 455.1391.

**N-(2-(1H-Indol-3-yl)ethyl)-N-(2-(4-methoxyphenyl)-2-oxoethyl)-4-methylbenzenesulfonamide (1b)**

Yield: 92%; yellow solid; m.p. 175-177 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.99 (s, 1H), 7.83 (d, J = 8.0 Hz, 2H), 7.75 (d, J = 8.0 Hz, 2H), 7.49 (d, J = 8.0 Hz, 2H), 7.28 (dd, J = 16.0, 8.0 Hz, 3H), 7.14 (t, J = 12.0, 8.0 Hz, 1H), 7.06 (t, J = 16.0, 8.0 Hz, 1H), 6.96 (s, 1H), 6.89 (d, J = 12.0 Hz, 2H), 4.64 (s, 2H), 3.85 (s, 3H), 3.54 (t, J = 16.0, 8.0 Hz, 2H), 2.99 (t, J = 12.0, 8.0 Hz, 1H), 2.40 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 192.7, 163.8, 143.3, 136.6, 136.1, 130.4, 130.4, 129.5, 127.9, 127.5, 127.1, 122.09, 122.04, 119.4, 118.6, 113.9, 112.4, 111.0, 55.4, 53.3, 48.8, 24.7, 21.5; HRMS-ESI (m/z): calcd for C_{26}H_{26}NaN_{2}O_{4}S [M+Na]^+ : 485.1613 found 485.1488.

**N-(2-(1H-Indol-3-yl)ethyl)-N-(2-(2-methoxyphenyl)-2-oxoethyl)-4-methylbenzenesulfonamide (1c)**

Yield: 90%; white solid; m.p. 164-166 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.98 (s, 1H), 7.74 (d, J = 8.0 Hz, 2H), 7.67 (dd, J = 8.0, 1.2 Hz, 1H), 7.51-7.44 (m, 2H), 7.30 (d, J = 8.0 Hz, 1H), 7.24 (t, J = 8.0, 4.0 Hz, 2H), 7.14 (t, J = 12.0, 4.0 Hz, 1H), 7.04 (t, J = 12.0, 8.0 Hz, 1H), 6.99-6.95 (m, 2H), 6.91 (d, J = 8.0 Hz, 1H), 4.72 (s, 2H), 3.81 (s, 3H), 3.57 (t, J = 16.0, 8.0 Hz, 2H), 3.03 (t, J = 16.0, 8.0 Hz, 2H), 2.39 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 159.0, 143.0, 137.4, 136.2, 134.4, 130.7, 129.4, 127.6, 127.3, 125.6, 122.13, 122.10, 120.9, 119.4, 118.8, 112.8, 111.5, 111.1, 57.4, 55.5, 48.7, 24.8, 21.6; HRMS-ESI (m/z): calcd for C_{26}H_{26}NaN_{2}O_{4}S [M+Na]^+ : 485.1613 found 485.1488.

**N-(2-(1H-Indol-3-yl)ethyl)-4-methyl-N-(2-oxo-2-(p-tolyl)ethyl)benzenesulfonamide (1d)**

Yield: 80 %; yellow solid; m.p. 144-146 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.02 (s, 1H), 7.75 (t, J = 16.0, 8.0 Hz, 3H), 7.50 (d, J = 8.0 Hz, 1H), 7.31 (d, J = 12.0 Hz, 1H), 7.27 (d, J = 8.0 Hz, 2H), 7.23 (d, J = 8.0 Hz, 2H), 7.18-7.14 (m, 1H), 7.09-7.05 (m, 1H), 6.97 (d, J = 2.0 Hz, 1H), 4.69 (s, 2H), 3.57 (dd, J = 12.0, 8.0 Hz, 2H), 2.41 (s, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 193.9, 144.7, 143.4, 136.8, 136.2, 132.5, 129.6, 129.5, 128.2, 127.6, 127.2, 122.2, 122.1, 119.5, 118.7, 112.5, 111.2, 53.4, 48.9, 24.8, 21.8, 21.6; HRMS-ESI (m/z): calcd for C_{26}H_{27}N_{2}O_{3}S [M+H]^+ : 447.1664 found 447.1736.

**N-(2-(1H-indol-3-yl)ethyl)-N-(2-(4-chlorophenyl)-2-oxoethyl)-4-methylbenzenesulfonamide (1e)**

Yield: 65 %; sticky brown oil; ¹H NMR (400 MHz, DMSO-d₆) δ 10.7 (bs, 1H), 7.99 (d, J = 8.4 Hz, 2H), 7.75 (d, J = 8.4 Hz, 2H), 7.61 (d, J = 8.4 Hz, 2H), 7.38 (t, J = 7.6 Hz, 3H), 7.30 (d, J = 8.0 Hz, 1H), 7.08-7.02 (m, 2H), 6.94 (t, J = 7.2 Hz, 1H), 4.94 (s, 2H), 3.42-3.38 (m, 2H), 2.90-2.88 (m, 2H), 2.38 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 193.7, 143.0, 138.5, 136.9, 136.9, 135.8, 134.4, 133.9, 133.3, 132.5, 131.6, 130.9, 129.5, 128.6, 128.2, 127.6, 127.2, 122.2, 122.1, 119.5, 118.7, 112.5, 111.2, 53.4, 48.9, 24.8, 21.8, 21.6; HRMS-ESI (m/z): calcd for C_{26}H_{26}NaN_{2}O_{4}S [M+Na]^+ : 485.1613 found 485.1488.
136.1, 133.4, 129.9, 129.6, 128.8, 126.9, 126.8, 122.8, 118.2, 117.9, 11.3, 110.5, 53.8, 49.2, 24.1, 20.9; HRMS-ESI (m/z): calcd for C_{25}H_{23}ClN_{2}O_{3}S [M+Na]^+: 489.1010 found 489.0999.

N-(2-(1H-indol-3-yl)ethyl)-N-(2-(3,4-dichlorophenyl)-2-oxoethyl)-4-methylbenzenesulfonamide (1f): The crude was taken into next without any purification and the product was confirmed by HRMS. HRMS-ESI (m/z): calcd for C_{25}H_{23}Cl_{2}N_{2}O_{3}S [M+H]^+: 501.0801 found 501.0789.

N-(2-(1H-indol-3-yl)ethyl)-N-(2-(4-fluorophenyl)-2-oxoethyl)-4-methylbenzenesulfonamide (1g): The crude was taken into next without any purification and the product was confirmed by HRMS. HRMS-ESI (m/z): calcd for C_{25}H_{23}FNaN_{2}O_{3}S [M+Na]^+: 473.1063 found 473.2012.

N-(2-(1H-indol-3-yl)ethyl)-4-methyl-N-(2-(naphthalen-2-yl)-2-oxoethyl)benzenesulfonamide (1h): Yield: 75%; yellow solid; m.p. 184-186 °C; $^1$H NMR (400 MHz, CDCl$_3$) δ 8.31 (s, 1H), 7.92-7.85 (m, 4H), 7.77 (d, $J$ = 8.0 Hz, 2H), 7.67-7.54 (m, 3H), 7.47 (d, $J$ = 8.0 Hz, 1H), 7.36-7.23 (m, 3H), 7.12-7.08 (m, 1H), 7.02-6.97 (m, 2H), 4.83 (s, 2H), 3.60 (t, $J$ = 16.0, 8.0 Hz, 2H), 3.02 (t, $J$ = 16.0, 8.0 Hz, 2H), 2.42 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 193.9, 143.0, 136.9, 136.5, 136.0, 135.4, 129.5, 129.3, 129.27, 129.22, 128.5, 128.3, 127.4, 127.1, 126.7, 126.63, 126.61, 123.1, 122.4, 122.2, 121.3, 118.7, 118.6, 118.0, 111.3, 111.1, 53.2, 48.6, 43.0, 39.9, 39.7, 25.3, 24.5, 21.2, 21.1; HRMS-ESI (m/z): calcd for C$_{29}$H$_{26}$NaN$_{2}$O$_{3}$S [M+Na]$^+$ : 505.1664 found 505.1545.

N-(2-(1H-indol-3-yl)ethyl)-N-(3,3-dimethyl-2-oxobutyl)-4-methylbenzenesulfonamide (1i): Yield: 85%; pale-yellow sticky mass; $^1$H NMR (400 MHz, CDCl$_3$) δ 8.00 (bs, 1H), 7.71 (d, $J$ = 8.0 Hz, 2H), 7.49 (d, $J$ = 8.0 Hz, 1H), 7.34 (d, $J$ = 8.0 Hz, 1H), 7.26-7.24 (m, 3H), 7.17 (t, $J$ = 8.0 Hz, 1H), 7.08 (t, $J$ = 8.4 Hz, 1H), 7.01 (s, 1H), 4.20 (s, 1H), 3.51 (t, $J$ = 7.2 Hz, 2H), 3.01 (t, $J$ = 7.2 Hz, 2H), 2.40 (s, 3H), 0.99 (s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 209.7, 143.2, 137.2, 136.3, 129.4, 127.5, 127.1, 122.2, 119.6, 118.6, 118.2, 111.2, 51.4, 48.2, 43.0, 39.7, 26.2, 25.2, 21.6; HRMS-ESI (m/z): calcd for C$_{23}$H$_{28}$NaN$_{2}$O$_{3}$S [M+Na]$^+$ : 435.1718 found 435.1822.
1-((2-(1H-indol-3-yl)ethyl)(benzyl)amino)butan-2-one (1j): Yield: 65%; brown sticky mass; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.06 (bs, 1H), 7.48 (d, \(J = 8.0\) Hz, 1H), 7.36-7.22 (m, 6H), 7.15 (td, \(J = 8.0, 1.2\) Hz, 1H), 7.06 (td, \(J = 8.0, 0.8\) Hz, 1H), 6.94 (d, \(J = 2.0\) Hz, 1H), 2.97-2.85 (m, 4H), 2.37 (q, \(J = 7.2\) Hz, 2H), 0.95 (t, \(J = 7.2\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 211.8, 138.7, 136.1, 128.9, 128.2, 127.3, 127.1, 121.8, 121.6, 119.1, 118.6, 113.8, 111.0, 63.3, 58.8, 55.1, 33.1, 23.2, 7.5; HRMS-ESI (m/z): calcd for C\(_{21}\)H\(_{24}\)N\(_2\)O \([\text{M}+\text{H}]^+\): 321.1967 found 321.1928.

N-(2-(1H-indol-3-yl)ethyl)-4-methyl-N-(1-oxo-1-phenylpropan-2-yl)benzenesulfonamide (1k): Yield: 70%; yellow sticky mass; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.12 (d, \(J = 8.0\) Hz, 2H), 8.00 (s, 1H), 7.71 (d, \(J = 8.0\) Hz, 2H), 7.62-7.55 (m, 2H), 7.47 (t, \(J = 8.0\) Hz, 2H), 7.30 (d, \(J = 8.0\) Hz, 1H), 7.24-7.10 (m, 4H), 6.90 (s, 1H), 5.66 (dd, \(J = 16.0, 8.0\) Hz, 1H), 3.49-3.30 (m, 2H), 3.02-2.93 (m, 1H), 2.37 (s, 3H), 1.19 (d, \(J = 8.0\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 197.9, 143.5, 136.9, 136.1, 135.2, 133.5, 129.6, 128.9, 128.7, 127.4, 127.2, 122.0, 121.9, 119.4, 118.8, 112.7, 111.0, 55.6, 45.6, 27.2, 21.4, 13.7; HRMS-ESI (m/z): calcd for C\(_{26}\)H\(_{26}\)NaN\(_2\)O\(_3\)S \([\text{M}+\text{Na}]^+\): 469.1664 found 469.1556.

N-(2-(1H-Indol-3-yl)ethyl)-4-methyl-N-(2-oxo-1,2-diphenylethyl)benzenesulfonamide (1l): Yield: 72%; yellow solid; m.p. 135-137 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.90 (s, 1H), 7.74-7.71 (m, 2H), 7.67-7.61 (m, 2H), 7.51-7.46 (m, 1H), 7.41-7.32 (m, 6H), 7.28-7.09 (m, 5H), 7.02-6.98 (m, 1H), 6.76 (s, 1H), 6.72 (d, \(J = 2.4\) Hz, 1H), 3.73-3.65 (m, 1H), 3.54-3.46 (m, 1H), 3.07-2.99 (m, 1H), 2.36 (s, 3H), 2.14-2.06 (m, 1H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 196.3, 143.2, 136.9, 136.0, 135.3, 134.8, 133.4, 129.9, 129.5, 129.4, 129.3, 128.9, 128.6, 128.4, 127.3, 127.2, 126.9, 121.76, 121.73, 119.0, 118.8, 113.1, 110.9, 65.1, 47.6, 27.0, 21.4; HRMS-ESI (m/z): calcd for C\(_{31}\)H\(_{28}\)NaN\(_2\)O\(_3\)S \([\text{M}+\text{Na}]^+\): 531.1821 found 531.1712.

4-Methyl-N-(2-oxo-2-phenylethyl)-N-(2-(1-phenyl-1H-indol-3-yl)ethyl)benzenesulfonamide (1m): Yield: 76 %; yellow sticky mass; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.85-7.82 (m, 2H), 7.76 (d, \(J = 8.0\) Hz, 2H), 7.57-7.37 (m, 10H), 7.33-7.24 (m, 3H), 7.20-7.10 (m, 3H), 4.75 (s, 2H), 3.64-3.60 (m, 2H), 3.06 (t, \(J = 8.0\) Hz, 2H), 2.39 (s, 3H); 194.2, 143.3, 139.5, 136.7, 135.9, 134.8, 133.6, 129.6, 129.5, 128.7, 128.5, 127.9, 127.4, 127.0, 126.1, 125.8, 123.9, 122.5, 120.1, 118.9, 113.5, 110.5, 53.5, 48.7, 24.6, 21.5; HRMS-ESI (m/z): calcd for C\(_{31}\)H\(_{28}\)NaN\(_2\)O\(_3\)S \([\text{M}+\text{Na}]^+\): 531.1821 found 531.1694.
N-(2-(1H-Indol-3-yl)ethyl)-N-(2-oxo-2-phenylethyl)benzenesulfonamide (1n): Yield: 86 %; yellow solid; m.p. 142-144 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.00 (s, 1H), 7.87 (d, \(J = 8.0\) Hz, 2H), 7.89 (d, \(J = 2.0\) Hz, 2H), 7.57-7.54 (m, 2H), 7.50-7.45 (m, 3H), 7.41 (t, \(J = 8.0\) Hz, 2H), 7.30 (d, \(J = 8.0\) Hz, 1H), 7.14 (t, \(J = 8.0\) Hz, 1H), 7.05 (t, \(J = 8.0\) Hz, 1H), 6.95 (d, \(J = 4.0\) Hz, 1H), 4.71 (s, 2H), 3.60 (dd, \(J = 12.0, 8.0\) Hz, 2H), 3.01 (t, \(J = 8.0\) Hz, 2H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 194.2, 139.8, 136.2, 134.9, 133.8, 132.7, 129.0, 128.8, 128.0, 127.5, 127.1, 127.0, 122.26, 122.21, 119.6, 118.6, 112.4, 111.2, 53.5, 48.9, 24.8; HRMS-ESI (m/z): calcd for C\(_{24}\)H\(_{22}\)NaN\(_2\)O\(_3\)S \([\text{M+Na}]^+\) : 441.1351 found 441.1235.

N-(2-(1H-Indol-3-yl)ethyl)-2-nitro-N-(2-oxo-2-phenylethyl)benzenesulfonamide (1o): Yield: 78 %; yellow solid; m.p. 140-142 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.05 (d, \(J = 8.0\) Hz, 1H), 7.97 (s, 1H), 7.77 (d, \(J = 8.0\) Hz, 2H), 7.60-7.47 (m, 5H), 7.41 (t, \(J = 8.0\) Hz, 2H), 7.28 (d, \(J = 8.0\) Hz, 1H), 7.13 (t, \(J = 8.0\) Hz, 1H), 7.04 (t, \(J = 8.0\) Hz, 1H), 6.98 (s, 1H), 4.86 (s, 2H), 3.78 (t, \(J = 8.0\) Hz, 2H), 3.03 (t, \(J = 8.0\) Hz, 2H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 193.9, 147.7, 136.2, 134.7, 133.8, 133.6, 133.2, 131.7, 130.8, 128.8, 127.9, 127.0, 124.0, 122.4, 122.2, 119.6, 118.5, 112.0, 56.3, 48.9, 24.6; HRMS-ESI (m/z): calcd for C\(_{24}\)H\(_{21}\)NaN\(_3\)O\(_5\)S \([\text{M+Na}]^+\) : 468.1202 found 468.1088.

2-((2-(1H-indol-3-yl)ethyl)(benzyl)amino)-1-phenylethan-1-one (1p): Yield: 82 %; brown sticky mass; m.p. 140-142 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.29 (bs, 1H), 7.76 (d, \(J = 7.2\) Hz, 2H), 7.54-7.48 (m, 2H), 7.49-7.24 (m, 9H), 7.12 (td, \(J = 8.0, 1.2\) Hz, 1H), 7.05 (td, \(J = 7.6, 0.8\) Hz, 1H), 6.96 (d, \(J = 1.2\) Hz, 1H), 4.11 (bs, 4H), 3.25-3.23 (m, 2H), 3.13-3.09 (m, 2H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 136.1, 133.5, 129.8, 128.5, 128.0, 127.1, 121.2, 119.2, 118.6, 119.1, 58.4, 54.4, 22.6; HRMS-ESI (m/z): calcd for C\(_{25}\)H\(_{24}\)N\(_2\)O \([\text{M+H}]^+\) : 369.1967 found 369.1959.

Methyl N\(^\alpha\)-(2-oxo-2-phenylethyl)-N\(^\alpha\)-tosyl-L-tryptophanate (1q): Yield: 70 %; yellow solid; m.p. 118-120 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.02 (s, 1H), 7.96 (d, \(J = 4.0\) Hz, 2H), 7.83 (d, \(J = 12.0\) Hz, 2H), 7.60 (t, \(J = 16.0, 8.0\) Hz, 1H), 7.48 (t, \(J = 16.0, 8.0\) Hz, 2H), 7.40 (d, \(J = 8.0\) Hz, 1H), 7.31 (d, \(J = 8.0\) Hz, 1H), 7.23 (t, \(J = 16.0, 8.0\) Hz, 2H), 7.15 (t, \(J = 12.0, 8.0\) Hz, 1H), 7.08-7.00 (m, 1H), 6.94 (d, \(J = 4.0\) Hz, 1H), 5.10 (s, 2H), 4.65 (dd, \(J = 8.0, 4.0\) Hz, 1H), 3.41 (s, 3H), 3.27-3.12 (m, 2H), 2.40 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 194.3, 171.3, 143.6, 136.6, 136.1, 135.2, 133.6, 129.5, 129.3, 128.8, 128.2, 128.0, 127.1, 127.0, 123.3, 122.1, 119.6, 118.5, 111.2, 109.8, 59.2, 52.0, 50.5, 26.8, 21.6; HRMS-ESI (m/z): calcd for C\(_{27}\)H\(_{26}\)N\(_2\)O\(_5\)S \([\text{M+Na}]^+\) : 513.1562 found 513.1443.
Methyl Nα-(2-(4-methoxyphenyl)-2-oxoethyl)-Nα-tosyl-L-tryptophanate (1r): Yield: 77%; yellow solid; m.p. 100-102 °C; 1H NMR (400 MHz, CDCl3) δ 8.12 (s, 1H), 7.95-7.92 (m, 2H), 7.84-7.81 (m, 2H), 7.39 (d, J = 8.0 Hz, 1H), 7.29-7.20 (m, 3H), 7.16-7.12 (m, 1H), 7.07-7.03 (m, 1H), 6.96-6.92 (m, 3H), 5.05 (s, 2H), 4.64 (dd, J = 12.0, 8.0, Hz, 1H), 3.87 (s, 3H), 3.40 (s, 3H), 3.26-3.11 (m, 2H), 2.39 (s, 3H); 13C NMR (100 MHz, CDCl3) δ 192.6, 171.2, 163.8, 143.4, 136.6, 136.0, 130.2, 129.2, 128.03, 128.9, 123.2, 121.9, 119.4, 118.3, 113.9, 111.1, 109.7, 59.2, 55.4, 51.8, 49.5, 26.7, 21.5; HRMS-ESI (m/z): calcd for C28H28NaN2O6S [M+Na]+: 546.1668 found 546.1560.

Methyl Nα-(2-(4-chlorophenyl)-2-oxoethyl)-Nα-tosyl-L-tryptophanate (1s): Yield: 54%; yellow solid; m.p. 80-82 °C; 1H NMR (400 MHz, CDCl3) δ 7.99 (s, 1H), 7.90-7.86 (m, 2H), 7.81 (d, J = 8.0 Hz, 2H), 7.44 (d, J = 8.0 Hz, 2H), 7.39 (d, J = 4.0 Hz, 1H), 7.29 (d, J = 8.0 Hz, 1H), 7.23 (d, J = 8.0 Hz, 2H), 7.18-7.14 (m, 1H), 7.08-7.04 (m, 1H), 6.94 (d, J = 4.0 Hz, 2H), 5.03 (s, 2H), 4.65 (dd, J = 8.0, 4.0 Hz, 1H), 3.41 (s, 3H), 3.23 (dd, J = 12.0, 8.0 Hz, 1H), 3.15-3.10 (m, 1H), 2.40 (s, 3H); 13C NMR (100 MHz, CDCl3) δ 193.2, 171.2, 143.6, 140.0, 136.3, 136.0, 133.8, 129.36, 129.31, 129.0, 128.0, 126.9, 123.1, 122.1, 119.5, 118.3, 111.1, 109.6, 59.0, 51.9, 49.8, 26.7, 21.5; HRMS-ESI (m/z): calcd for C27H25ClNaN2O5S [M+Na]+: 547.1173 found 547.1085.

Methyl Nα-(2-(naphthalen-2-yl)-2-oxoethyl)-Nα-tosyltryptophanate (1t): The crude was taken to next without any purification and the product was confirmed by HRMS. HRMS-ESI (m/z): calcd for C31H28N2NaO5S [M+Na]+: 563.1611 found 563.1611.

N-(2-(1H-Indol-3-yl)-2-phenylethyl)-4-methyl-N-(2-oxo-2-phenylethyl)benzenesulfonamide (1u): Yield: 88%; white solid; m.p. 162-164 °C; 1H NMR (400 MHz, CDCl3) δ 8.06 (s, 1H), 7.69 (d, J = 8.0 Hz, 2H), 7.56-7.48 (m, 3H), 7.40 (d, J = 8.0 Hz, 1H), 7.34-7.09 (m, 1H), 7.05 (d, J = 4.0 Hz, 1H), 7.00-6.96 (m, 1H), 4.66 (d, J = 16.0 Hz, 1H), 4.56 (t, J = 16.0, 8.0 Hz, 1H), 4.24-4.13 (m, 2H), 3.76 (dd, J = 12.0 8.0 Hz, 1H), 2.39 (s, 3H); 13C NMR (100 MHz, CDCl3) δ 194.3, 143.2, 142.1, 136.8, 136.3, 134.7, 133.5, 19.4, 128.59, 128.56, 128.1, 127.6, 127.5, 126.7, 126.6, 122.1, 122.0, 119.5, 119.2, 116.1, 111.0, 53.2, 52.4, 42.6, 21.5; HRMS-ESI (m/z): calcd for C31H28NaN2O6S [M+Na]+: 531.1821 found 531.1713.

N-(2-(1H-Indol-3-yl)-2-(p-tolyl)ethyl)-4-methyl-N-(2-oxo-2-phenylethyl)benzenesulfonamide (1v):
Yield: 77%; yellow solid; m.p. 139-141 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.03 (s, 1H), 7.69 (d, $J = 8.0$ Hz, 2H), 7.57-7.48 (m, 3H), 7.41 (d, $J = 8.0$ Hz, 1H), 7.34-7.22 (m, 5H), 7.13-7.09 (m, 3H), 7.04-6.96 (m, 4H), 4.65 (d, $J = 16.0$ Hz, 1H), 4.23-4.16 (m, 2H), 7.74 (dd, $J = 12.0$ 8.0 Hz, 1H), 2.39 (s, 3H), 2.24 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 194.3, 143.2, 139.1, 136.8, 136.3, 136.2, 134.7, 133.4, 129.4, 129.2, 128.5, 128.0, 127.6, 127.5, 126.6, 122.1, 122.0, 119.5, 119.3, 116.4, 111.0, 53.1, 52.3, 42.2, 21.5, 20.9; HRMS-ESI (m/z): calcd for C$_{32}$H$_{30}$N$_2$O$_3$S [M+Na]$^+$ : 545.1977 found 545.1872.

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Date: Jul 6 2010

Solvent: cdc12

Ambient temperature

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STANDARD 1H OBSERVE

Solvent: CDCl3
Ambient temperature
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<td>Tri_Freq</td>
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<td>5.0 (ppm)</td>
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<td>Tri_Domain</td>
<td>Proton</td>
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Pulse Sequence: 52pul
Mercury-400BB "MerPlus400"
Date: Aug 30 2016
Solvent: dmso
Ambient temperature
Total 32 repetitions
Hou-C1-1

Pulse Sequence: s2pul
Mercury-400BB "MerPlus400"
Date: Aug 30 2018
Solvent: ddeo
Ambient temperature
Total 672 repetitions
Pulse Sequence: s2pu1
Mercury-6000B "HerPlus600"
Date: Aug 5, 2015
Solvent: dcdCl3
 Ambient temperature
Total 32 repetitions
Pulse Sequence: s2pul
Instrument: Mercury-400D, "SorFlo-100"
Date: Aug 8, 2018
Solvent: CDCl3
 Ambient Temperature
Total 1584 repetitions
MR-085

Pulse Sequence: s2pul
Mercury-400MR "HerPlus400"
Date: Nov 25 2016
Solvent: cdCl3
Ambient temperature
Total 32 repetitions
Pulse Sequence: H2ps1
Mercury-10288 "NMRPlus600"
Date: Aug 1 2018
Solvent: ccd13
Ambient temperature
Total 32 repetitions
Pulse Sequence: s2pul
Mercury-400BR "NMR Plus 400"
Date: Aug 1 2016
Solvent: CDCl3
Ambient temperature
Total 1952 repetitions
Pulse Sequences DEPT
Pulse Sequence: 60ps
Mercury-400B "MerPlus 400"
Date: Aug 8 2016
Solvent: CDCl3
Ambient temperature
Total 32 repetitions
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<td>Tri_Mode</td>
<td>OFF</td>
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Pulse Sequence: st2pul
Mercury-4000B "MerPlus400"
Date: Nov 28 2016
Solvent: cdc13
Ambient temperature
Total 32 repetitions
Pulse Sequence: 62pul
Mercury-600BB "MerPlus400"
Date: Nov 25 2016
Solvent: cdCl3
Ambient temperature
Total 1156 repetitions
Pulse Sequence: s2pul
Mercury-4000 & "HerPlus400"
Date: Aug. 1 2016
Solvent: cdcl3
Ambient temperature
Total 32 repetitions
Hou-93-17

Pulse Sequence: 45ppm
Mercury-4000S "MorPlus88"
Date: Aug 1 2016
Solvent: d6d13
Ambient temperature
Total: 64000 repetitions
Pulse sequence: s2pul
Mercury-400BB "Varian 400"
Date: Aug 8 2019
Solvent: CDCl3
Ambient temperature
File: n0175-12
Total 12 repetitions
Pulse Sequence: s2pu3
Mercury-400 and "MerPlus80"
Date: Aug 8 2006
Solvent: cdc13
Ambient temperature
Total 764 repetitions
Pulse Sequence: ztop
Mercury-600BB *WarpPlus400*
Date: Aug 1 2016
Solvent: cdc19
Ambient Temperature
Total 32 repetitions
Pulse Sequence: s2pu1
Mercury-400BB "MerPlus400"
Date: Aug 1 2018
Solvent: ccd3
Ambient temperature
Total 1600 repetitions
Hou-03-43

Pulse Sequence: s1psl
Mercury-460BB "MerPlus400"
Date: Aug 3, 2018
Solvent: cdcl3
Ambient Temperature
Total 32 repetitions
Pulse Sequence: s2pul
Mercury-20388 "KemPlus400"
Date: Aug. 1 2016
Solvent: cdc32
Ambient temperature
Total: 2416 repetitions
Hou-02-43

Pulse Sequence: DFT

1v
checkCIF/PLATON report

Structure factors have been supplied for datablock(s) nb7154

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: nb7154

Bond precision:  C-C = 0.0022 Å  Wavelength=0.7107 Å

Cell:  
\[ a=8.8691(4) \text{ Å} \quad b=9.8046(5) \text{ Å} \quad c=12.4019(6) \text{ Å} \]
\[ \alpha=99.836(2)^\circ \quad \beta=102.889(2)^\circ \quad \gamma=94.239(2)^\circ \]  
Temperature: 150 K

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<tr>
<td>Space group</td>
<td>P -1</td>
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<tr>
<td>Hall group</td>
<td>-P 1</td>
</tr>
<tr>
<td>Moiety formula</td>
<td>C25 H22 N2 O2 S</td>
</tr>
<tr>
<td>Sum formula</td>
<td>C25 H22 N2 O2 S</td>
</tr>
<tr>
<td>Mr</td>
<td>414.51</td>
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<tr>
<td>Dx, g cm⁻³</td>
<td>1.338</td>
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<tr>
<td>Z</td>
<td>2</td>
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<tr>
<td>Mu (mm⁻¹)</td>
<td>0.182</td>
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<tr>
<td>F000</td>
<td>436.0</td>
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<td>436.41</td>
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<td>Tmin,Tmax</td>
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<td>Tmin’</td>
<td>0.920</td>
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</table>

Correction method= # Reported T Limits: Tmin=0.921 Tmax=0.937  
AbsCorr = MULTI-SCAN

Data completeness= 0.996  
\[ \text{Theta}(\text{max})= 26.420^\circ \]

R(reflections)= 0.0380(3632)  
\[ wR2(reflections)= 0.1117(4204) \]

S = 1.000  
Npar= 275

The following ALERTS were generated. Each ALERT has the format test-name_ALERT_alert-type_alert-level.  
Click on the hyperlinks for more details of the test.
Alert level A

DENSX01_ALERT_1_A The ratio of the calculated to measured crystal density lies outside the range 0.80 <> 1.20
Calculated density = 1.338
Measured density = 0.000

PLAT902_ALERT_1_A No (Interpretable) Reflections found in FCF .... Please Check

Alert level G

PLAT005_ALERT_5_G No Embedded Refinement Details found in the CIF Please Do!
PLAT066_ALERT_1_G Predicted and Reported Tmin&Tmax Range Identical ? Check
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note) 0.002 Degree
PLAT380_ALERT_4_G Incorrectly? Oriented X(sp2)-Methyl Moiety ..... C7 Check
PLAT899_ALERT_4_G SHELXL97 is Deprecated and Succeeded by SHELXL 2014 Check

2 ALERT level A = Most likely a serious problem - resolve or explain
0 ALERT level B = A potentially serious problem, consider carefully
0 ALERT level C = Check. Ensure it is not caused by an omission or oversight
5 ALERT level G = General information/check it is not something unexpected

4 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
0 ALERT type 2 Indicator that the structure model may be wrong or deficient
0 ALERT type 3 Indicator that the structure quality may be low
2 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation); however, if you intend to submit to Acta Crystallographica Section C or E or IUCrData, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the Notes for Authors of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 11/08/2016; check.def file version of 04/08/2016