One-pot three-component sulfone synthesis exploiting palladium-catalysed aryl halide aminosulfonylation

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Supporting Information

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1) General considerations

All glassware was dried in an oven (>200 °C) and allowed to cool under a positive nitrogen pressure (passed through a Drierite® filled tube) prior to use. All reactions were conducted with continuous magnetic stirring under a nitrogen atmosphere with anhydrous solvents unless otherwise stated. HPLC grade solvents were purchased from Sigma Aldrich, Fischer Scientific or Rathburn. Dichloromethane, tetrahydrofuran and toluene were obtained from an in-house solvent purification system having passed through anhydrous alumina columns. Water was purified by an Elix® UV-10 system. 1,4-Dioxane was distilled over calcium hydride and stored over 4 Å molecular sieves and degassed prior to use. Chemicals were purchased from Sigma Aldrich, Alfa Aesar, Acros or Fluorochem and used without prior purification, with the exception of 1,4-diazabicyclo[2.2.2]octane (DABCO), which was purified by sublimation (50 °C, 1 mbar). Et₂O refers to diethyl ether and petrol to 40-60 °C petroleum ether.

Thin Layer Chromatography (TLC) was performed on Merck Kieselgel 60 F254 pre-coated aluminium plates which were visualised using UV light (254 nm) and/or 1% aq. KMnO₄. Column chromatography was performed on silica gel (Fluka Kieselgel 60, particle size 0.040-0.063 nm) with the indicated eluents.

¹H, ¹³C and ¹⁹F Nuclear Magnetic Resonance experiments were carried out using Brüker DPX-200 (200 MHz), AVN-400 (400 MHz) or AVC-500 (500 MHz) spectrometers in the deuterated solvent stated. Chemical shifts (δ) are reported in parts per million (ppm) and referenced relative to the residual solvent peak(s). Assignments were made on the basis of chemical shifts, coupling constants, COSY, HSQC, NOE data and comparison with spectra of related compounds. The abbreviations s, d, dd, t, dt, td, tt, q, quin, br. s, m and app, denote singlet, doublet, doublet of doublets, triplet, doublet of triplets, triplet of doublets, triplet of triplets, quartet, quintet, broad singlet, multiplet and apparent. The coupling constants (J) are reported in Hertz (Hz) and rounded to the nearest 0.5 Hz.

Melting points were recorded on a Leica Gallen III hot-stage microscope and are uncorrected. Infra-red spectra were recorded neat on a Brüker Tensor 27 FT-IR spectrometer retrofitted with a diamond attenuated total reflectance (ATR) module, with an internal range of 600-4000 cm⁻¹. Low-resolution mass spectra (m/z) were recorded on a LCT Premier Open Access spectrometer (ESI). High resolution mass measurements were run on a Brüker MicroTOF or a Micromass GCT instrument by the mass spectrometry department of the Chemistry Research Laboratory, University of Oxford, UK. Values are quoted as ratio of mass to charge in Daltons and relative intensities of assignable peaks observed are quoted as a percentage value. High resolution values are calculated to four decimal places from the molecular formula, all found within a tolerance of 5 ppm.
2) General procedures

General procedure A
The electrophile (2 equiv), \(N\)-aminosulfonamide (1 equiv) and \(K_2CO_3\) (2 equiv) were added to a round-bottomed flask. 1,4-Dioxane [0.30 M] was added and the reaction mixture was stirred at 100 °C for the specified length of time. After cooling to rt, the reaction mixture was diluted with water (5 mL) and washed with \(CH_2Cl_2\) (3 \(\times\) 5 mL). The combined organic extracts were washed with brine (5 mL), dried over \(MgSO_4\), filtered and then concentrated in vacuo. Purification via column chromatography yielded the corresponding sulfone.

General procedure B
Benzyl bromide (0.95 equiv), \(N\)-aminosulfonamide (1 equiv) and \(K_2CO_3\) (2 equiv) were added to a round-bottomed flask. 1,4-Dioxane [0.30 M] was added and the reaction mixture was stirred at 50 °C for 1 h. The electrophile (1.5 equiv) was then added and the reaction mixture was warmed to 100 °C and stirred at this temperature for a further 15 h. After cooling to rt, the reaction mixture was diluted with water (5 mL) and washed with \(CH_2Cl_2\) (3 \(\times\) 5 mL). The combined organic extracts were washed with brine (5 mL), dried over \(MgSO_4\), filtered and then concentrated in vacuo. Purification via column chromatography yielded the corresponding sulfone.

General procedure C
An oven-dried tube was charged with tri-\(\text{tert}\)-butylphosphonium tetrafluoroborate (20 mol%), palladium(II) acetate (10 mol%) and the halogenated substrate (1 equiv). Either bis(sulfur dioxide)-1,4-diazabicyclo[2.2.2]octane (DABSO) (1.1 eq) or (DABSO) (0.6 eq) and 1,4-diazabicyclo[2.2.2]octane (DABCO) (0.5 equiv) were then added as specified. The solid reagents were weighed out in air. The tube was then evacuated and backfilled with \(N_2\). The hydrazine (1.2 equiv) and 1,4-dioxane [0.30 M] were added via microsyringe. The reaction mixture was stirred at 70 °C for 16 h. Either Method I or Method II was then followed as stated.

Method I: \(K_2CO_3\)\(_{aq}\) [2.40 M, 2.5 equiv] and the electrophile (2.5 equiv) were added and the reaction mixture stirred at 90 °C for the specified length of time.

Method II: \(K_2CO_3\)\(_{aq}\) [2.40 M, 2.5 equiv] and benzyl bromide (0.95 equiv) were added and the reaction mixture was stirred at 90 °C for 1 h. The second electrophile (2 equiv) was then added and the reaction mixture stirred at 90 °C for 19 h.

After cooling to rt, the suspension was filtered through a short pad of Celite® and washed sequentially with \(CH_2Cl_2\) (5 mL) and water (5 mL). The organic layer was separated and the aqueous layer extracted with \(CH_2Cl_2\) (2 \(\times\) 5 mL). The combined organic extracts were washed with brine, dried over \(MgSO_4\), filtered and then concentrated in vacuo. Purification via column chromatography yielded the corresponding sulfone.
3) Preparation of substrates

4-Ethoxy-N-(morpholin-4-yl)benzenesulfonamide, 9b

An oven-dried tube was charged with tri-tert-butylphosphonium tetrafluoroborate (14 mg, 20 mol%), palladium(II) acetate (5 mg, 10 mol%), 1-ethoxy-4-iodobenzene (60 mg, 0.24 mmol), DABSO (35 mg, 0.14 mmol) and DABCO (13 mg, 0.12 mmol). The solid reagents were weighed out in air. The tube was then evacuated and backfilled with N₂. Aminomorpholine (35 µL, 0.36 mmol) and 1,4-dioxane (1.6 mL) were added via microsyringe. The reaction mixture was stirred at 70 °C for 16 h. After cooling to rt, the suspension was filtered through a short pad of Celite® and the residue washed sequentially with CH₂Cl₂ (5 mL) and Et₂O (5 mL) before being concentrated in vacuo. Column chromatography (eluent: 50-100%, Et₂O in petrol) yielded the N-aminosulfonamide 9b as a white crystalline solid (63 mg, 92%); mp 144-145 °C (CH₂Cl₂); ν_max (neat)/cm⁻¹ 3209, 2987, 1595, 1577, 1496, 1463, 1327, 1303, 1264, 1183, 1160, 1111, 1041; δ_H (400 MHz, CDCl₃) 7.91-7.82 (2H, m, 2 × ArH), 7.00-6.91 (2H, m, 2 × ArH), 5.86 (1H, s, NH), 4.08 (2H, q, J 7.0, OC₃H₃CH₃), 3.58 (4H, t, J 4.5, N(CH₂CH₂)₂O), 2.61 (4H, t, J 4.5, N(C₃H₂CH₂)₂O), 1.43 (3H, t, J 7.0, OCH₂C₃H₃); δ_C (101 MHz, CDCl₃) 162.7, 130.3, 129.8, 114.4, 66.6, 64.0, 56.6, 14.6; m/z (ESI⁺) 595 ([2M + Na]⁺, 100%), 309 ([M + Na]⁺, 63%), 287 ([M + H]⁺, 93%); HRMS (ESI⁺) C₁₂H₁₄N₂NaO₄S⁺ ([M + Na]⁺) requires 309.8079; found 309.8078.

1-Ethoxy-3-iodobenzene

To a suspension of 3-iodophenol (1.50 g, 6.80 mmol) and K₂CO₃ (1.32 g, 9.50 mmol) in ethanol (10 mL) was added ethyl iodide (0.5 mL, 6.10 mmol) at rt. The reaction mixture was stirred at reflux for 24 h. The mixture was filtered and the filtrate concentrated in vacuo. The residue was diluted in Et₂O and the organic layer was washed sequentially with 1 M NaOH, 3 M HCl, sat. NaHCO₃(aq) and brine. The combined organic layers were dried over MgSO₄, filtered and concentrated in vacuo. Column chromatography (eluent: 4:1, petrol:Et₂O) yielded 1-ethoxy-3-iodobenzene as a colourless oil (1.30 g, 77%); ν_max (neat)/cm⁻¹ 3061, 2979, 2929, 2879, 1581, 1565, 1466, 1417, 1388, 1284, 1241, 1159, 1113, 1089, 1042; δ_H (500 MHz, CDCl₃) 7.31-7.27 (2H, m, 2 × ArH), 7.01 (1H, app t, J 8.0, ArH), 6.88 (1H, ddd, J 8.0, 2.5, 1.0, ArH), 4.00 (2H, q, J 7.0, OCH₂CH₃), 1.43 (3H, t, J 7.0, OCH₂CH₃); δ_C (126 MHz, CDCl₃) 159.6, 130.8, 129.7, 123.6, 114.2, 94.5, 63.7, 14.9; HRMS (FI⁺)
C$_8$H$_9$IO$^+$ ([M$^+$]) requires 247.9698; found 247.9702. $^1$H NMR spectroscopy data in accordance with literature.$^1$

1-Ethoxy-2-iodobenzene

![Structure of 1-Ethoxy-2-iodobenzene](image)

To a suspension of 2-iodophenol (0.77 mL, 6.80 mmol) and K$_2$CO$_3$ (1.32 g, 9.50 mmol) in ethanol (10 mL) was added ethyl iodide (0.5 mL, 6.10 mmol) at rt. The reaction mixture was stirred at reflux for 24 h. The mixture was filtered and the filtrate concentrated in vacuo. The residue was diluted in Et$_2$O and the organic layer was washed sequentially with 1 M NaOH, 3 M HCl, sat. NaHCO$_3$(aq) and brine. The combined organic layers were dried over MgSO$_4$, filtered and concentrated in vacuo.

Column chromatography (eluent: 4:1, petrol:Et$_2$O) yielded the 1-ethoxy-2-iodobenzene as a colourless oil (1.21 g, 72%); $\nu_{max}$ (neat)/cm$^{-1}$ 3060, 2980, 2930, 2882, 1581, 1568, 1465, 1438, 1389, 1275, 1243, 1161, 1108, 1088, 1049, 1016; $\delta_H$ (400 MHz, CDCl$_3$) 7.79 (1H, dd, $J$ 7.5, 1.5, Ar$H$), 7.35-7.27 (1H, m, Ar$H$), 6.82 (1H, dd, $J$ 8.5, 1.5, Ar$H$), 6.71 (1H, app td, $J$ 7.5, 1.5, Ar$H$), 4.10 (2H, q, $J$ 7.0, OCH$_2$CH$_3$), 1.50 (3H, t, $J$ 7.0, OCH$_2$C$_2$H$_3$); $\delta_C$ (101 MHz, CDCl$_3$) 157.5, 139.5, 129.4, 122.4, 122.4, 112.2, 86.8, 64.9, 14.8; HRMS (FI$^+$) C$_8$H$_9$O$I^+$ ([M$^+$]) requires 247.9698; found 247.9702. $^1$H NMR spectroscopy data in accordance with literature.$^1$

5-(2-Iodoethyl)-1H-indole, 12

![Chemical Reaction of 5-(2-Iodoethyl)-1H-indole](image)

A mixture of tert-butyl 5-bromo-1H-indole-1-carboxylate$^2$ (5.32 g, 18.0 mmol), Pd(OAc)$_2$ (202 mg, 0.90 mmol), 2-dicyclohexylphosphino-2',4',6'-triisopropylbiphenyl (XPhos) (0.86 g, 1.80 mmol), Cs$_2$CO$_3$ (17.6 g, 53.9 mmol) and diethyl malonate (2.7 mL, 18.0 mmol) in toluene (200 mL) was stirred under N$_2$ for 16 h at 100 °C. The reaction mixture was cooled to rt and concentrated in vacuo, then partitioned between EtOAc (150 mL) and water (100 mL). The mixture was filtered through Celite$^8$ and the aqueous layer was extracted with EtOAc (150 mL). The combined organic layers were dried over MgSO$_4$, filtered and concentrated in vacuo. Column chromatography (eluent: 15% Et$_2$O in petrol) yielded diethyl [1-(tert-butoxycarbonyl)-1H-indol-5-yl]propanedioate, 12a as a white crystalline solid (4.86 g, 72%); mp 80-81 °C (CH$_2$Cl$_2$); $\nu_{max}$ (neat)/cm$^{-1}$ 2985, 2160, 2026, 2009, 1977, 1748, 1721, 1467, 1443, 1385, 1367, 1335, 1314, 1299, 1283, 1256, 1223, 1172, 1143, 1129, 1115,
1079, 1030; δH (400 MHz, CDCl3) 8.12 (1H, d, J 8.5, ArH), 7.67-7.63 (2H, m, 2 × ArH), 7.35 (1H, dd, J 8.5, 2.0, ArH), 6.56 (1H, d, J 4.0, ArH), 4.70 (1H, s, CH(COOCH2CH3)2), 4.27-4.17 (4H, m, CH(COOCH2CH3)2), 1.66 (9H, s, O′Bu), 1.26 (6H, t, J 7.0, CH(COOCH2CH3)2); δc (101 MHz, CDCl3) 168.5, 149.7, 134.9, 130.8, 127.2, 126.5, 125.3, 121.8, 115.3, 107.4, 83.8, 61.8, 57.8, 28.2, 14.1; m/z (ESI⁺) 774 ([2M + Na⁺], 25%), 398 ([M + Na⁺], 10%), 376 ([M + H⁺], 100%); HRMS (ESI⁺) C19H25NaO6⁺ ([M + Na⁺]) requires 398.1574; found 398.1565.

A solution of diethyl [1-(tert-butoxycarbonyl)-1H-indol-5-yl]propanedioate, 12a (4.84 g, 12.9 mmol) in THF (25 mL) and EtOH (2.5 mL) was treated with 2 M NaOH (5.6 mL). The solution was stirred at 0 °C for 16 h. The reaction mixture was concentrated in vacuo and the aqueous residue was acidified to pH 1 with 2 M HCl. The aqueous solution was washed with EtOAc (3 × 20 mL). The combined organic layers were dried over MgSO4, filtered and concentrated in vacuo to yield 1-(tert-butoxycarbonyl)-1H-indol-5-yl]acetic acid, 12b as a brown solid (2.66 g, 75%) that was used in the next step without further purification.

To a solution of 1-(tert-butoxycarbonyl)-1H-indol-5-yl]acetic acid, 12b (1.56 g, 5.44 mmol) in THF (9 mL) was added BH3·THF (18.8 mL, 1 M solution in THF) dropwise over 10 minutes at 0 °C. The reaction mixture was stirred for a further 90 min at 0 °C and then quenched by slow addition of MeOH. The reaction mixture was concentrated in vacuo. Chromatography (eluent: 3:7 to 1:1 Et2O:petrol) yielded tert-butyl 5-(2-hydroxyethyl)-1H-indole-1-carboxylate, 12c as a colourless oil (0.68 g, 46%); νmax (neat)/cm⁻¹ 3359, 2979, 2935, 1729, 1470, 1442, 1371, 1347, 1327, 1254, 1217, 1192, 1161, 1082, 1040, 1022; δH (400 MHz, CDCl3) 8.08 (1H, d, J 8.0, ArH), 7.58 (1H, d, J 4.0, ArH), 7.40 (1H, d, J 1.0, ArH), 7.17 (1H, dd, J 8.5, 1.5, ArH), 6.52 (1H, d, J 3.5, ArH), 3.85 (2H, t, J 6.5, ArCH2CH2OH), 2.93 (2H, t, J 6.5, ArCH2CH2OH), 2.20 (1H, s, ArCH2CH2OH), 1.67 (9H, s, O′Bu); δc (101 MHz, CDCl3) 149.8, 134.0, 132.8, 131.0, 126.2, 125.4, 121.1, 115.2, 107.2, 83.7, 64.0, 39.1, 28.2; m/z (ESI⁺) 545 ([2M + Na⁺], 100%), 284 ([M + Na⁺], 20%); HRMS (ESI⁺) C15H19NaO5⁺ ([M + Na⁺]) requires 284.1257; found 284.1254.

To a solution of PPh3 (0.84 g, 2.22 mmol) and 1H-imidazole (0.23 g, 3.35 mmol) in CH2Cl2 (6 mL) was added iodine (0.82 g, 3.22 mmol) at 0 °C. A solution of tert-butyl 5-(2-hydroxyethyl)-1H-indole-1-carboxylate, 12c (0.68 g, 2.48 mmol) in CH2Cl2 (3 mL) was added dropwise. The reaction mixture was stirred at rt for 16 h and then washed with H2O (10 mL), sat. NaHSO3(aq) (10 mL), and brine (10 mL). The combined organic layers were dried over MgSO4, filtered and concentrated in vacuo. Heptane (50 mL) was added and the mixture filtered through Celite®. The filtrate was concentrated in vacuo. Column chromatography (eluent: 0-10%, Et2O in petrol) yielded tert-butyl 5-(2-idoethyl)-1H-indole-1-carboxylate, 12d as a colourless oil (0.59 g, 64%); νmax (neat)/cm⁻¹ 2978, 2932, 1730, 1469, 1443, 1370, 1348, 1326, 1294, 1255, 1217, 1192, 1161, 1129, 1107, 1081, 1040, 1022; δH (400 MHz, CDCl3) 8.07 (1H, d, J 8.0, ArH), 7.59 (1H, d, J 4.0, ArH), 7.38 (1H, d, J 1.5, ArH), 7.14 (1H, dd, J 8.5, 1.5, ArH), 6.54 (1H, d, J 4.0, ArH), 3.42-3.36 (2H, m, ArCH2CH2I), 3.29-3.23 (2H, m, ArCH2CH2I), 1.67 (9H, s, O′Bu); δc (101 MHz, CDCl3) 149.7, 135.1, 134.2, 130.9,
126.4, 124.6, 120.5, 115.3, 107.1, 83.7, 40.3, 28.2, 6.5; m/z (ESI⁺) 394 ([M + Na]⁺, 100%); HRMS (ESI⁺) C₁₉H₁₇INaO₂⁺ ([M + Na]⁺) requires 394.0274; found 394.0263.

Trifluoroacetic acid (9.9 mL) was added dropwise to a solution of tert-butyl 5-(2-iodoethyl)-1H-indole-1-carboxylate, 12d (0.59 g, 1.59 mmol) in chloroform (2.0 mL). The reaction mixture was stirred for 30 min at rt and then concentrated in vacuo. EtOAc (20 mL) was added and washed with sat. NaHCO₃(aq) (20 mL). The organic layer was dried over MgSO₄, filtered and concentrated in vacuo. Column chromatography (eluent: 3:7, petrol:EtO) yielded 12 as a pale yellow solid (276 mg, 64%); mp 91–92 °C (CHCl₃); ν<sub>max</sub> (neat)/cm<sup>-1</sup> 3412, 2958, 2922, 2852, 1474, 1454, 1416, 1342, 1280, 1260, 1235, 1222, 1166, 1137, 1089, 1064, 1019; δ<sub>H</sub> (400 MHz, CDCl₃) 8.10 (1H, br. s, NH), 7.48 (1H, s, ArH), 7.34 (1H, d, J 8.5, ArH), 7.21 (1H, app t, J 3.0, ArH), 7.04 (1H, dd, J 8.5, 1.5, ArH), 6.55–6.52 (1H, m, ArH), 3.44–3.37 (2H, m, ArCH₂CH₂I), 3.31–3.25 (2H, m, ArCH₂CH₂I); δ<sub>C</sub> (101 MHz, CDCl₃) 134.8, 132.4, 128.2, 124.7, 122.7, 120.2, 111.2, 102.5, 40.8, 7.1; m/z (ESI⁺) 272 ([M + H]⁺, 100%); HRMS (ESI⁺) C₁₉H₁₇INN⁺ ([M – H]⁻) requires 269.9785; found 269.9779.

4) Synthesis of sulfones

Benzy1 4-methylphenyl sulfone, 10a and N-[(E)-phenylmethylidene]morpholin-4-amine, 11

Table 2. entry 4: General procedure A was followed by the use of 4-methyl-N-(morpholin-4-yl)benzenesulfonamide 9a<sup>1</sup> (123 mg, 0.48 mmol) and benzyl bromide (114 µL, 0.96 mmol). The reaction mixture was stirred at 100 °C for 1 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10a as a white crystalline solid (109 mg, 92%) and hydrazone 11 as a white crystalline solid (79 mg, 87%).

Table 2. entry 5: General procedure A was followed by the use of 4-methyl-N-(morpholin-4-yl)benzenesulfonamide 9a<sup>1</sup> (123 mg, 0.48 mmol) and benzyl chloride (110 µL, 0.96 mmol). The reaction mixture was stirred at 100 °C for 1 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10a as a white crystalline solid (82 mg, 69%).

Table 4. entry 1: General procedure C (Method I) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 4-iodotoluene (105 mg, 0.48 mmol). The reaction mixture was stirred at 90 °C for 5 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10a as a white crystalline solid (108 mg, 91%).

Sulfone 10a: mp 139–141 °C (CH₂Cl₂) {lit.<sup>4</sup> 141–142 °C}; δ<sub>H</sub> (400 MHz, CDCl₃) 7.54–7.48 (2H, m, 2 × ArH), 7.37–7.21 (5H, m, 5 × ArH), 7.14–7.06 (2H, m, 2 × ArH), 4.29 (2H, s, SCH₂), 2.42 (3H, s,
ArMe); δC (101 MHz, CDCl₃) 144.6, 135.0, 130.8, 129.5, 128.7, 128.6, 128.5, 128.3, 62.9, 21.6; m/z (ESI⁺) 515 ([2M + Na]⁺, 100%), 269 ([M + Na]⁺, 95%). Data in accordance with literature.⁴

Hydrazone 11: mp 85-87 °C (CH₂Cl₂) {lit.⁵ 89 °C (ethanol)}; δH (400 MHz, CDCl₃) 7.60-7.34 (2H, m, 3 × ArH), 7.32-7.26 (1H, m, ArHC=N), 3.90 (4H, t, J 5.0, N(CH₂CH₂)₂O), 3.19 (4H, t, J 5.0, N(CH₂CH₂)₂O); δC (101 MHz, CDCl₃) 136.2, 136.0, 128.4, 126.2, 66.5, 51.9; HRMS (ESI⁺) C₁₁H₁₄N₂O²⁺ ([M]⁺) requires 190.1106; found 190.1110. Data in accordance with literature.⁶

Methyl 4-methylphenyl sulfone, 10b

Table 2, entry 1: General procedure A was followed by the use of 4-methyl-N-(morpholin-4-yl)benzenesulfonamide 9a (123 mg, 0.48 mmol) and methyl iodide (179 µL, 2.88 mmol). The reaction mixture was stirred at 100 °C for 1 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10b as a white crystalline solid (63 mg, 77%); mp 85-87 °C (CH₂Cl₂) {lit.⁷ 88 °C (ethanol)}; δH (400 MHz, CDCl₃) 7.86-7.79 (2H, m, 2 × ArH), 7.39-7.35 (2H, m, 2 × ArH), 3.03 (3H, s, SMe), 2.45 (3H, s, ArMe); δC (101 MHz, CDCl₃) 144.6, 137.7, 129.9, 127.4, 44.6, 21.6; HRMS (EI⁺) C₈H₁₀O₂S⁺ ([M⁺]⁺) requires 170.0401; found 170.0402. Data in accordance with literature.⁸

4-Methylphenyl propyl sulfone, 10c

Table 2, entry 2: General procedure A was followed by the use of 4-methyl-N-(morpholin-4-yl)benzenesulfonamide 9a (123 mg, 0.48 mmol) and 1-iodopropane (94 µL, 0.96 mmol). The reaction mixture was stirred at 100 °C for 16 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10c as a white crystalline solid (58 mg, 61%); mp 50-52 °C {lit.⁹ 53-55 °C}; νmax (neat)/cm⁻¹ 2971, 2937, 2880, 1595, 1494, 1466, 1407, 1383, 1347, 1300, 1285, 1252, 1221, 1184, 1140, 1086, 1067, 1020; δH (400 MHz, CDCl₃) 7.73-7.69 (2H, m, 2 × ArH), 7.31-7.26 (2H, m, 2 × ArH), 3.00-2.95 (2H, m, SCH₂CH₂CH₃), 2.38 (3H, s, ArMe), 1.71-1.60 (2H, m, SCH₂CH₂CH₃), 0.91 (3H, t, J 7.5, SCH₂CH₂CH₃); δC (101 MHz, CDCl₃) 144.5, 136.2, 129.9, 128.1, 118.5, 116.6, 113.0; m/z (ESI⁻) 419 ([2M + Na]⁻, 100%), 221 ([M + Na]⁻, 67%); HRMS (ESI⁻) C₁₀H₁₄NaO₂S⁻ ([M + Na]⁻) requires 221.0607; found 221.0606. ¹H NMR spectroscopy data in accordance with literature.¹⁰
Hexyl 4-methylphenyl sulfone, 10d and hexyl 4-methylbenzenesulfinate, 10’d

Table 2, entry 3: General procedure A was followed by the use of 4-methyl-N-(morpholin-4-yl)benzenesulfonamide 9a (123 mg, 0.48 mmol) and 1-iodohexane (142 µL, 0.96 mmol). The reaction mixture was stirred at 100 °C for 16 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10d as a colourless oil (77 mg, 67%) and sulfinate ester 10’d as a pale yellow oil (9 mg, 8%).

**Sulfone 10d:** ν\(_{\text{max}}\) (neat)/cm\(^{-1}\) 2957, 2928, 2855, 1597, 1457, 1285, 1141, 1087; δ\(_{\text{H}}\) (400 MHz, CDCl\(_3\)) 7.79-7.75 (2H, m, 2 × ArH), 7.37-7.32 (2H, m, 2 × ArH), 3.08-3.01 (2H, m, C\(_\text{H}_2\)(CH\(_2\))\(_4\)CH\(_3\)), 2.44 (3H, s, ArMe), 1.76-1.61 (2H, m, OCH\(_2\)C\(_\text{H}_2\)(CH\(_2\))\(_3\)CH\(_3\)), 1.37-1.29 (2H, m, ArMe), 1.28-1.19 (4H, m, 2 × C\(_\text{H}_2\)), 0.84 (3H, t, J 7.0, O(CH\(_2\))\(_5\)C\(_\text{H}_3\)); δ\(_{\text{C}}\) (101 MHz, CDCl\(_3\)) 144.5, 136.2, 129.8, 128.0, 56.4, 31.1, 27.9, 22.7, 22.3, 21.6, 13.9; HRMS (FI\(^+\)) C\(_{13}\)H\(_{20}\)O\(_2\)S\(^+\) ([M]\(^+\)) requires 240.1184; found 240.1181.

**Sulfinate ester 10’d:** ν\(_{\text{max}}\) (neat)/cm\(^{-1}\) 2955, 2939, 2859, 1597, 1492, 1465, 1379, 1302, 1260, 1178, 1133, 1080, 1038, 1017; δ\(_{\text{H}}\) (400 MHz, CDCl\(_3\)) 7.64-7.57 (2H, m, 2 × ArH), 7.36-7.31 (2H, m, 2 × ArH), 4.02 (1H, dt, J 10.0, 6.5, OCH\(_a\)H\(_b\)), 3.61 (1H, dt, J 10.0, 6.5, OCH\(_a\)H\(_b\)), 2.43 (3H, s, ArMe), 1.66-1.58 (2H, m, OCH\(_2\)CH\(_2\)(CH\(_2\))\(_3\)CH\(_3\)), 1.38-1.20 (6H, m, 3 × CH\(_2\)), 0.87 (3H, t, J 7.0, O(CH\(_2\))\(_2\)CH\(_3\)); δ\(_{\text{C}}\) (101 MHz, CDCl\(_3\)) 142.6, 141.8, 129.7, 125.2, 64.6, 31.3, 29.7, 25.4, 22.5, 21.5, 14.0; HRMS (FI\(^+\)) C\(_{13}\)H\(_{20}\)O\(_2\)S\(^+\) ([M]\(^+\)) requires 240.1184; found 240.1184.

Benzyl 4-ethoxyphenyl sulfone, 10e

Table 2, entry 6: General procedure A was followed by the use of 4-ethoxy-N-(morpholin-4-yl)benzenesulfonamide, 9b (137 mg, 0.48 mmol) and benzyl bromide (114 µL, 0.96 mmol). The reaction mixture was stirred at 100 °C for 1 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10e as a white crystalline solid (124 mg, 94%).

**Benzyl 4-ethoxyphenyl sulfone, 10e:** ν\(_{\text{max}}\) (neat)/cm\(^{-1}\) 2955, 2939, 2859, 1597, 1492, 1465, 1379, 1302, 1260, 1178, 1133, 1080, 1038, 1017; δ\(_{\text{H}}\) (400 MHz, CDCl\(_3\)) 7.64-7.57 (2H, m, 2 × ArH), 7.36-7.31 (2H, m, 2 × ArH), 4.02 (1H, dt, J 10.0, 6.5, OCH\(_a\)H\(_b\)), 3.61 (1H, dt, J 10.0, 6.5, OCH\(_a\)H\(_b\)), 2.43 (3H, s, ArMe), 1.66-1.58 (2H, m, OCH\(_2\)CH\(_2\)(CH\(_2\))\(_3\)CH\(_3\)), 1.38-1.20 (6H, m, 3 × CH\(_2\)), 0.87 (3H, t, J 7.0, O(CH\(_2\))\(_2\)CH\(_3\)); δ\(_{\text{C}}\) (101 MHz, CDCl\(_3\)) 142.6, 141.8, 129.7, 125.2, 64.6, 31.3, 29.7, 25.4, 22.5, 21.5, 14.0; HRMS (FI\(^+\)) C\(_{13}\)H\(_{20}\)O\(_2\)S\(^+\) ([M]\(^+\)) requires 240.1184; found 240.1184.
(138 µL, 1.20 mmol) and 1-ethoxy-4-iodobenzene (119 mg, 0.48 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10e as a white crystalline solid (99 mg, 75%).

Table 4, entry 9: General procedure C (Method I) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 1-aminopiperidine (62 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 1-ethoxy-4-iodobenzene (119 mg, 0.48 mmol). The reaction mixture was stirred at 90 °C for 5 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10e as a white crystalline solid (117 mg, 88%).

Sulfone 10e: mp 93-96 °C ((CD₂)₂CO); νₘₚₓ (neat)/cm⁻¹ 2972, 2930, 2893, 1594, 1577, 1493, 1482, 1454, 1428, 1415, 1389, 1317, 1298, 1270, 1176, 1145, 1132, 1114, 1084, 1033, 1007; δₜ (400 MHz, (CD₂)₂CO) 7.62-7.54 (2H, m, 2 × ArH), 7.36-7.25 (3H, m, 3 × ArH), 7.21-7.15 (2H, m, 2 × ArH), 7.06-6.99 (2H, m, 2 × ArH), 4.45 (2H, s, SCH₂), 4.15 (2H, q, J₇.0, OCH₂CH₃), 1.40 (3H, t, J₇.0, OCH₂); δC (101 MHz, (CD₂)₂CO) 163.5, 131.4, 131.0, 130.7, 130.0, 128.6(2), 128.5(7), 114.8, 64.3, 62.4, 14.4; m/z (ESI⁺) 575 ([2M + Na]⁺, 100%), 299 ([M + Na]⁺, 88%); m/z (ESI⁻) 311 ([M + Cl]⁻, 100%); HRMS (ESI⁺) C₁₅H₁₆NaO₃S⁺ ([M + Na]⁺) requires 299.0712; found 299.0707.

Benzyl 4-(trifluoromethyl)phenyl sulfone, 10f

Table 2, entry 7: General procedure A was followed by the use of N-(morpholin-4-yl)-4-(trifluoromethyl)benzenesulfonamide³ (149 mg, 0.48 mmol) and benzyl bromide (114 µL, 0.96 mmol). The reaction mixture was stirred at 100 °C for 16 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10f as a white crystalline solid (101 mg, 70%).

Table 4, entry 15: General procedure C (Method I) was followed by the use of DABSO (127 mg, 0.53 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 4-iodobenzotrifluoride (71 µL, 0.48 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10f as a white crystalline solid (61 mg, 42%).

Sulfone 10f: mp 162-164 °C (CH₂Cl₂) {lit.²³ 163-165 °C}; δₜ (500 MHz, CDCl₃) 7.77-7.68 (4H, m, 4 × ArH), 7.38-7.33 (1H, m, ArH), 7.31-7.27 (2H, m, 2 × ArH), 7.13-7.06 (2H, m, 2 × ArH), 4.35 (2H, s, SCH₂); δC (126 MHz, CDCl₃) 141.3, 135.4 (q, J₇ CF 33.0), 130.8, 129.3, 129.1, 128.8, 127.5, 126.0 (q, J₇ CF 4.0), 123.1 (q, J₇ CF 273.0), 62.8; δF (377 MHz, CDCl₃) -63.2 (s, CF₃); m/z (ESI⁻) 623 ([2M + Na]⁻, 100%), 323 ([M + Na]⁺, 99%). Data in accordance with literature.²³
4-Methylphenyl propan-2-yl sulfone, 10g and propan-2-yl 4-methylbenzenesulfinate, 10’g

Table 2, entry 8: General procedure A was followed by the use of 4-methyl-N-(morpholin-4-yl)benzenesulfonamide 9a\(^3\) (123 mg, 0.48 mmol) and 2-iodopropane (96 µL, 0.96 mmol). The reaction mixture was stirred at 100 °C for 16 h. Column chromatography (eluent: 7:3, petrol:E\(_2\)O) yielded sulfone 10g as a white crystalline solid (45 mg, 47%) and sulfinate ester 10’g as a colourless oil (6 mg, 7%).

Table 2, entry 8: General procedure B was followed by the use of 4-methyl-N-(morpholin-4-yl)benzenesulfonamide 9a\(^3\) (123 mg, 0.48 mmol) and 2-iodopropane (72 µL, 0.72 mmol). Column chromatography (eluent: 7:3, petrol:E\(_2\)O) yielded sulfone 10g as a white crystalline solid (42 mg, 44%) and sulfinate ester 10’g as a colourless oil (5 mg, 5%).

Sulfone 10g: mp 81-83°C (CH\(_2\)Cl\(_2\)) \{lit.\(^1\) 81-83 °C\}; \(\delta\)\(_H\) (400 MHz, (CD\(_3\))\(_2\)SO) 7.72 (2H, d, \(J\) 8.0, 2 × ArH), 7.47 (2H, d, \(J\) 8.0, 2 × ArH), 3.42-3.30 (1H, m, CHMe\(_2\)), 2.42 (3H, s, ArMe), 1.13 (6H, d, \(J\) 7.0, CHMe\(_2\)); \(\delta\)\(_C\) (101 MHz, (CD\(_3\))\(_2\)SO) 145.2, 134.7, 130.7, 129.5, 55.0, 21.9, 16.1; m/z (ESI\(^+\)) 419 ([2M + Na]\(^+\), 100%), 221 ([M + Na]\(^+\), 45%). Data in accordance with literature.\(^1\)

Sulfinate ester 10’g: \(\nu_{\text{max}}\) (neat)/cm\(^{-1}\) 2950, 2940, 2861, 1594, 1491, 1470, 1377, 1301, 1265, 1179, 1131, 1051; \(\delta\)\(_H\) (400MHz, (CD\(_3\))\(_2\)SO) 7.48 (2H, d, \(J\) 7.0, 2 × ArH), 7.12 (2H, d, \(J\) 7.0, 2 × ArH), 3.82-3.71 (1H, m, CHMe\(_2\)), 2.29 (3H, s, ArMe), 1.03 (6H, d, \(J\) 6.5, CHMe\(_2\)); \(\delta\)\(_C\) (101 MHz, (CD\(_3\))\(_2\)SO) 138.6, 131.0, 129.0, 126.3, 62.9, 26.4, 21.7; m/z (ESI\(^+\)) 419 ([2M + Na]\(^+\), 100%), 221 ([M + Na]\(^+\), 45%). Data in accordance with literature.\(^1\)

Ethyl 2-methyl-2-[(4-methylphenyl)sulfonyl]propanoate, 10h

Table 2, entry 9: General procedure A was followed by the use of 4-methyl-N-(morpholin-4-yl)benzenesulfonamide 9a\(^3\) (123 mg, 0.48 mmol) and ethyl-\(\alpha\)-bromoisobutyrate (141 µL, 0.96 mmol). The reaction mixture was stirred at 100 °C for 16 h. Column chromatography (eluent: 7:3, petrol:E\(_2\)O) yielded sulfone 10h as a white crystalline solid (66 mg, 51%).

Table 2, entry 9: General procedure B was followed by the use of 4-methyl-N-(morpholin-4-yl)benzenesulfonamide 9a\(^3\) (123 mg, 0.48 mmol) and ethyl-\(\alpha\)-bromoisobutyrate (106 µL, 0.72 mmol). Column chromatography (eluent: 7:3, petrol:E\(_2\)O) yielded sulfone 10h as a white crystalline solid (75 mg, 58%).
**Sulfone 10h:** mp 73–75 °C (CH$_2$Cl$_2$); $\nu_{\text{max}}$ (neat)/cm$^{-1}$ 2988, 2940, 1733, 1688, 1593, 1544, 1493, 1467, 1443, 1402, 1386, 1364, 1312, 1302, 1272, 1186, 1157, 1127, 1113, 1077, 1021; $\delta_H$ (400 MHz, CDCl$_3$) 7.73 (2H, d, $J$ 8.0, 2 × ArH), 7.34 (2H, d, $J$ 8.0, 2 × ArH), 4.14 (2H, q, $J$ 7.0, OCH$_2$CH$_3$), 2.45 (3H, s, ArMe), 1.61 (6H, s, 2 × SCMe), 1.22 (3H, t, $J$ 7.0, OCH$_2$CMe$_3$); $\delta_C$ (101 MHz, CDCl$_3$) 168.9, 145.1, 132.7, 130.4, 129.3, 69.0, 62.2, 21.7, 20.3, 13.8; m/z (ESI$^+$) 564 ([2M + Na]$^+$, 100%), 293 ([M + Na]$^+$, 75%), 270 ([M + H]$^+$, 65%); HRMS (ESI$^+$) C$_{13}$H$_{18}$NaO$_4$S$^+$ ([M + Na]$^+$) requires 293.0818; found 293.0810.

**Ethenyl 4-methylphenyl sulfone, 10i**

![Ethenyl 4-methylphenyl sulfone](image_url)

Table 2, entry 6: General procedure A was followed by the use of 4-methyl-N-(morpholin-4-yl)benzenesulfonamide 9a (123 mg, 0.48 mmol) and 1,2-dibromoethane (83 µL, 0.96 mmol). The reaction mixture was stirred for 1 h at 100 °C, then triethylamine (67 µL, 0.48 mmol) was added and the reaction stirred for a further 15 h. Column chromatography (eluent: 7:3, petrol:Et$_2$O) yielded sulfone 10i as a white crystalline solid (16 mg, 18%).

Table 2, entry 6: General procedure B was followed by the use of 4-methyl-N-(morpholin-4-yl)benzenesulfonamide 9a (123 mg, 0.48 mmol) and 1,2-dibromoethane (62 µL, 0.72 mmol). The reaction mixture was stirred for 1 h at 100 °C, then triethylamine (67 µL, 0.48 mmol) was added and the reaction stirred for a further 15 h. Column chromatography (eluent: 7:3, petrol:Et$_2$O) yielded sulfone 10i as a white crystalline solid (46 mg, 53%).

**Sulfone 10i:** mp 63–65°C (CH$_2$Cl$_2$) {lit.$^{14,15}$ 65.0-66.0 °C (2-PrOH)}; $\delta_H$ (400 MHz, CDCl$_3$) 7.80-7.75 (2H, m, 2 × ArH), 7.37-7.32 (2H, m, 2 × ArH), 6.64 (1H, dd, $J$ 16.5, 10.0, CH=CH$_2$), 6.42 (1H, d, $J$ 16.5, CH=CH$_{trans}$H), 6.00 (1H, d, $J$ 10.0, CH=CH$_{cis}$H), 2.44 (3H, s, ArMe); $\delta_C$ (101 MHz, CDCl$_3$) 144.7, 138.7, 136.6, 130.0, 128.0, 127.2, 21.7; m/z (ESI$^+$) 205 ([M + Na]$^+$, 100%), 183 ([M + H]$^+$, 10%). Data in accordance with literature.$^{14,15}$

**2-[(4-Methylphenyl)sulfonyl]-5-(trifluoromethyl)benzaldehyde, 10j**

![2-[(4-Methylphenyl)sulfonyl]-5-(trifluoromethyl)benzaldehyde](image_url)

Table 2, entry 11: General procedure B was followed by the use of 4-methyl-N-(morpholin-4-yl)benzenesulfonamide 9a (123 mg, 0.48 mmol) and 2-fluoro-5-(trifluoromethyl)benzaldehyde (101 µL, 0.72 mmol). Column chromatography (eluent: 7:3, petrol:Et$_2$O) yielded sulfone 10j as a white crystalline solid (63 mg, 40%); mp 64-66 °C (CH$_2$Cl$_2$); $\nu_{\text{max}}$ (neat)/cm$^{-1}$ 2930, 1692, 1598, 1494,
1323, 1297, 1251, 1157, 1125, 1078, 1046, 1020; δH (500 MHz, CDCl3) 10.90 (1H, s, CHO), 8.30-8.26 (2H, m, 2 × ArH), 7.99 (1H, dd, J 8.5, 2.0, ArH), 7.82-7.78 (2H, m, 2 × ArH), 7.37 (2H, d, J 8.0, 2 × ArH), 2.45 (3H, s, ArMe); δC (126 MHz, CDCl3) 188.0, 146.0, 145.7, 137.4, 135.5 (q, JCF 35.0), 134.4, 130.5, 130.2 (q, JCF 3.5), 130.1, 127.8, 126.6 (q, JCF 3.5), 122.5 (q, JCF 273.5), 21.7; δF (377 MHz, CDCl3) –63.4 (s, C3F3); HRMS (FI+ +) C15H11F3O3S+ ([M]+) requires 328.0381; found 328.0388.

1-Methyl-4-(phenylsulfonyl)benzene, 10k

Table 2, entry 12: General procedure A was followed by the use of 4-methyl-N-(morpholin-4-yl)benzenesulfonamide 9a3 (123 mg, 0.48 mmol) and diphenyliodonium triflate16 (413 mg, 0.96 mmol). The reaction mixture was stirred at 100 °C for 16 h. Column chromatography (eluent: 7:3, petrol:Et2O) yielded sulfone 10k as a white crystalline solid (22 mg, 20%).

Table 2, entry 12: General procedure B was followed by the use of 4-methyl-N-(morpholin-4-yl)benzenesulfonamide 9a3 (123 mg, 0.48 mmol) and diphenyliodonium triflate16 (320 mg, 0.72 mmol). Column chromatography (eluent: 7:3, petrol:Et2O) yielded sulfone 10k as a white crystalline solid (66 mg, 59%).

Sulfone 10k: mp 124-125 °C (CH2Cl2) {lit.17 125-127 °C}; δH (400 MHz, CDCl3) 7.95-7.91 (2H, m, ArH), 7.57-7.52 (1H, m, ArH), 7.51-7.46 (2H, m, ArH), 7.29 (2H, d, J 8.0, ArH), 2.39 (3H, s, ArMe); δC (126 MHz, CDCl3) 144.1, 142.0, 138.6, 133.0, 129.9, 129.2, 127.7, 127.5, 21.5; m/z (ESI+) 255 ([M + Na]+, 100%); HRMS (FI+) C15H11O2S+ ([M]+) requires 232.0558; found 232.0555. Data in accordance with literature.17

Benzyl phenyl sulfone, 10l

Table 4, entry 2: General procedure C (Method I) was followed by the use of DABSO (127 mg, 0.53 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and iodobenzene (54 µL, 0.48 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et2O) yielded sulfone 10l as a white crystalline solid (72 mg, 65%); mp 147-148 °C (CH2Cl2) {lit.18 147-148 °C (ethanol)}; δH (400 MHz, CDCl3) 7.68-7.56 (3H, m, 3 × ArH), 7.50-7.42 (2H, m, 2 × ArH), 7.36-7.23 (3H, m, 3 × ArH), 7.08 (2H, d, J 7.5, 2 × ArH),
4.32 (2H, s, SCH₂); δ_C (101 MHz, CDCl₃) 137.8, 133.7, 130.8, 128.9, 128.8, 128.6(2), 128.5(7), 128.1, 62.9; m/z (ESI⁺) 255 ([M + Na]⁺, 100%). Data in accordance with literature.¹⁹

**Benzyl 4-tert-butylphenyl sulfone, 10m**

![Structure of Benzyl 4-tert-butylphenyl sulfone, 10m]

Table 4, entry 3: General procedure C (Method I) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 4-tert-butylidobenzene (85 µL, 0.48 mmol). The reaction mixture was stirred at 90 °C for 10 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10m as a white crystalline solid (120 mg, 87%); mp 98-101 °C (CH₂Cl₂); ν_max (neat)/cm⁻¹ 3062, 3032, 2964, 2924, 2871, 1922, 1594, 1493, 1473, 1456, 1400, 1363, 1317, 1304, 1288, 1266, 1204, 1164, 1154, 1132, 1106, 1085, 1028, 1015; δ_H (400 MHz, CDCl₃) 7.60-7.54 (2H, m, 2 × ArH), 7.49-7.43 (2H, m, 2 × ArH), 7.36-7.23 (3H, m, 3 × ArH), 7.11 (2H, d, J 8.0, 2 × ArH), 4.30 (2H, s, SCH₂), 1.33 (9H, s, Ar'tBu); δ_C (101 MHz, CDCl₃) 157.7, 135.0, 130.9, 128.7, 128.5, 128.4, 128.2, 125.9, 62.9, 35.2, 31.1; m/z (ESI⁺) 311 ([M + Na]⁺, 100%); HRMS (ESI⁺) C₁₇H₂₀NaO₂S⁺ ([M + Na]⁺) requires 311.1076; found 311.1074.

**4-(Benzylsulfonyl)biphenyl, 10n**

![Structure of 4-(Benzylsulfonyl)biphenyl, 10n]

Table 4, entry 4: General procedure C (Method I) was followed by the use of DABSO (127 mg, 0.53 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 4-iodobiphenyl (134 mg, 0.48 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10n as a white crystalline solid (115 mg, 78%); mp 204-207 °C (CH₂Cl₂); ν_max (neat)/cm⁻¹ 2947, 1562, 1491, 1479, 1454, 1403, 1325, 1305, 1284, 1201, 1183, 1147, 1119, 1090, 1038, 1025, 1005; δ_H (500 MHz, CDCl₃) 7.71-7.65 (4H, m, 4 × ArH), 7.63-7.59 (2H, m, 2 × ArH), 7.51-7.47 (2H, m, 2 × ArH), 7.46-7.42 (1H, m, ArH), 7.36-7.32 (2H, m, 2 × ArH), 7.31-7.27 (1H, m, ArH), 7.16-7.12 (2H, m, 2 × ArH), 4.36 (2H, s, SCH₂); δ_C (126 MHz, CDCl₃) 146.6, 139.0, 136.4, 130.9, 129.2, 129.1, 128.8, 128.7, 128.6, 128.1, 127.4(1), 127.3(5), 63.0; m/z (ESI⁺) 331 ([M + Na]⁺, 100%); HRMS (ESI⁺) C₁₉H₁₆NaO₂S⁺ ([M + Na]⁺) requires 331.0763; found 331.0761.
Benzyl 2-naphthyl sulfone, 10o

Table 4, entry 5: General procedure C (Method I) was followed by the use of DABSO (127 mg, 0.53 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 2-iodonaphthalene (70 µL, 0.48 mmol). The reaction mixture was stirred at 90 ºC for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10o as a white crystalline solid (98 mg, 72%); mp 190-191 ºC (CH₂Cl₂) {lit.20 192-193 ºC}; ν max (neat)/cm⁻¹ 3062, 2968, 2922, 1593, 1508, 1495, 1458, 1401, 1346, 1314, 1256, 1201, 1186, 1158, 1075, 1030; δH (500 MHz, CDCl₃) 8.78 (1H, d, J 8.5, ArH), 8.09 (1H, d, J 8.5, ArH), 8.00-7.95 (2H, m, 2 × ArH), 7.70 (1H, ddd, J 8.5, 7.0, 1.5, ArH), 7.64 (1H, ddd, J 8.0, 7.0, 1.0, ArH), 7.44 (1H, app t, J 8.0, ArH), 7.27-7.23 (1H, m, ArH), 7.18-7.14 (2H, m, 2 × ArH), 6.97-6.93 (2H, m, 2 × ArH), 4.52 (2H, s, SC₂H₂); δC (126 MHz, CDCl₃) 135.2, 134.0, 133.0, 131.5, 130.6, 129.2(3), 129.1(7), 128.6(9), 128.4, 128.0, 126.9, 124.2, 124.1, 62.3; m/z (ESI⁺) 305 ([M + Na]⁺, 100%); HRMS (ESI⁺) C₁₇H₁₄NaO₂S⁺ ([M + Na]⁺) requires 305.0607; found 305.0608. ¹H NMR spectroscopy data in accordance with literature.21

Benzyl 2,4-dimethylphenyl sulfone, 10p

Table 4, entry 6: General procedure C (Method I) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 4-iodo-m-xylene (68 µL, 0.48 mmol). The reaction mixture was stirred at 90 ºC for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10p as a white crystalline solid (110 mg, 88%); mp 81-84 ºC (CH₂Cl₂); ν max (neat)/cm⁻¹ 2979, 2937, 2924, 2879, 1592, 1574, 1496, 1476, 1465, 1415, 1402, 1384, 1347, 1311, 1290, 1261, 1214, 1180, 1138, 1115, 1088, 1038; δH (400 MHz, CDCl₃) 7.60 (1H, d, J 8.0, ArH), 7.41-7.24 (3H, m, 3 × ArH), 7.13-7.08 (3H, m, 3 × ArH), 7.05 (1H, d, J 8.0, ArH), 4.33 (2H, s, SCH₂), 2.48 (3H, s, ArMe), 2.38 (3H, s, ArMe); δC (101 MHz, CDCl₃) 144.6, 138.5, 133.1, 131.0, 130.9, 128.7, 128.6, 128.1, 127.1, 127.0, 62.3, 21.4, 20.3; m/z (ESI⁺) 283 ([M + Na]⁺, 100%); HRMS (ESI⁺) C₁₅H₁₆NaO₂S⁺ ([M + Na]⁺) requires 283.0763; found 283.0751.
Benzyl 4-methoxyphenyl sulfone, 10q

Table 4, entry 7: General procedure C (Method I) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 4-iodoanisole (112 mg, 0.48 mmol). The reaction mixture was stirred at 90 °C for 5 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10q as a white crystalline solid (110 mg, 87%).

Table 4, entry 8: General procedure C (Method I) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 4-bromoanisole (60 µL, 0.48 mmol). The reaction mixture was stirred at 90 °C for 5 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10q as a white crystalline solid (58 mg, 46%).

Sulfone 10q: mp 82-83 °C (CH₂Cl₂) {lit. 83 °C}; δₓ (400 MHz, CDCl₃) 7.53 (2H, d, J 8.0, 2 × ArH), 7.36-7.24 (3H, m, 3 × ArH), 7.09 (2H, d, J 7.5, 2 × ArH), 6.90 (2H, d, J 8.0, 2 × ArH), 4.29 (2H, s, SC₆H₅), 3.86 (3H, s, OMe); δₓ (101 MHz, CDCl₃) 163.7, 130.8 (3), 130.8 (2), 129.4, 128.7, 128.6, 128.5, 114.0, 63.1, 55.7; m/z (ESI⁺) 285 ([M + Na]⁺, 100%). Data in accordance with literature.

Benzyl 3-ethoxyphenyl sulfone 10r

Table 4, entry 10: General procedure C (Method I) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 1-ethoxy-3-iodobenzene (119 mg, 0.48 mmol). The reaction mixture was stirred at 90 °C for 5 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10r as a white crystalline solid (115 mg, 87%); mp 103-105 °C (CH₂Cl₂); νₓ (neat)/cm⁻¹ 2987, 2922, 2878, 1594, 1580, 1492, 1480, 1467, 1456, 1437, 1390, 1307, 1286, 1239, 1200, 1181, 1164, 1151, 1126, 1096, 1072, 1048; δₓ (500 MHz, CDCl₃) 7.37-7.30 (2H, m, ArH), 7.30-7.22 (3H, m, 3 × ArH), 7.13-7.09 (3H, m, 3 × ArH), 7.05 (1H, app t, J 2.0, ArH), 4.30 (2H, s, SCH₂), 3.92 (2H, q, J 7.0, OCH₂CH₃), 1.38 (3H, t, J 7.0, OCH₂CH₃); δₓ (126 MHz, CDCl₃) 159.0, 138.8, 130.8, 129.9, 128.7, 128.5, 128.2, 121.1, 120.6, 113.2, 63.9, 62.9, 14.5; m/z (ESI⁺) 299 ([M + Na]⁺, 100%); m/z (ESI⁻) 311 ([M + Cl]⁻, 100%); HRMS (ESI⁺) C₁₅H₁₆NaO₃S⁺ ([M + Na]⁺) requires 299.0712; found 299.0717.
Benzyl 2-ethoxyphenyl sulfone, 10s

Table 4, entry 11: General procedure C (Method I) was followed by the use of DABSO (127 mg, 0.53 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 1-ethoxy-2-iodobenzene (119 mg, 0.48 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10s as a white crystalline solid (99 mg, 75%); mp 78-80 °C (CH₂Cl₂); ν_max (neat)/cm⁻¹ 3064, 3030, 2987, 2934, 2885, 1592, 1577, 1483, 1469, 1445, 1401, 1391, 1198, 1153, 1138, 1118, 1061, 1037; δ_H (400 MHz, CDCl₃) 7.68 (1H, dd, J 8.0, 1.5, ArH), 7.53 (1H, app t, J 8.0, ArH), 4.63 (2H, s, SC₄H₂), 4.29 (2H, q, J 7.0, OC₄H₂CH₃), 1.61 (3H, t, J 7.0, OCH₂C₄H₃); δ_C (101 MHz, CDCl₃) 156.7, 135.5, 130.9, 130.7, 128.6, 128.5, 128.3, 126.3, 120.6, 113.0, 65.2, 60.4, 14.8; m/z (ESI⁺) 299 ([M + Na]⁺, 100%); HRMS (ESI⁺) C₁₅H₁₆NO₃S⁺ ([M + Na]⁺) requires 299.0712; found 299.0709.

Benzyl 4-(methylsulfanyl)phenyl sulfone, 10t

Table 4, entry 12: General procedure C (Method I) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 4-iodothioanisole (120 mg, 0.48 mmol). The reaction mixture was stirred at 90 °C for 5 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10t as a white crystalline solid (119 mg, 89%); mp 150-153 °C (CH₂Cl₂); ν_max (neat)/cm⁻¹ 3060, 3030, 2998, 2947, 2921, 1579, 1477, 1455, 1428, 1404, 1394, 1310, 1295, 1284, 1274, 1190, 1150, 1094, 1074, 1026, 1012; δ_H (500 MHz, CDCl₃) 7.50-7.46 (2H, m, 2 × ArH), 7.35-7.31 (1H, m, ArH), 7.30-7.26 (2H, m, 2 × ArH), 7.23-7.20 (2H, m, 2 × ArH), 7.12-7.09 (2H, m, 2 × ArH), 4.30 (2H, s, SCH₂), 2.51 (3H, s, SMe); δ_C (126 MHz, CDCl₃) 147.2, 133.4, 130.8, 128.9, 128.7, 128.6, 128.2, 124.9, 63.0, 14.7; HRMS (EI⁺) C₁₄H₁₆O₂S₂⁺ ([M⁺]⁺) requires 278.0435; found 278.0448.

4-(Benzylsulfonyl)aniline, 10u
Table 4, entry 13: General procedure C (Method I) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 4-iodoaniline (105 mg, 0.48 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10u as a white crystalline solid (68 mg, 57%); mp 218-220 °C (CH₂Cl₂); \( \nu \text{max} \) (neat)/cm⁻¹ 3471, 3377, 3033, 2971, 2918, 1638, 1591, 1504, 1456, 1436, 1401, 1327, 1303, 1280, 1253, 1200, 1184, 1163, 1144, 1081, 1031, 1002; \( \delta_H \) (400 MHz, (CD₃)₂SO) 7.29-7.21 (5H, m, 5×ArH), 7.12-7.08 (2H, m, 2×ArH), 6.55-6.52 (2H, m, 2×ArH), 6.08 (2H, s, NH₂), 4.41 (2H, s, SCH₂); \( \delta_C \) (126 MHz, (CD₃)₂SO) 153.5, 130.9, 129.9, 129.6, 128.0(5), 128.0(1), 123.0, 112.4, 61.5; \( m/z \) (ESI⁺) 517 ([2M + Na]⁺, 25%), 270 ([M + Na]⁺, 100%); HRMS (ESI⁺) C₁₃H₁₃NNaO₂S⁺ ([M + Na]⁺) requires 270.0559; found 270.0568. \(^1\)H NMR spectroscopy data in accordance with literature.\(^{24}\)

4-(Benzylsulfonyl)-N,N-dimethylaniline, 10v

Table 4, entry 14: General procedure C (Method I) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 4-iodo-N,N-dimethylaniline\(^{25}\) (119 mg, 0.48 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10v as a white crystalline solid (97 mg, 74%); mp 147-149 °C (CH₂Cl₂); \( \nu \text{max} \) (neat)/cm⁻¹ 2926, 1592, 1550, 1561, 1493, 1455, 1442, 1403, 1374, 1331, 1300, 1285, 1229, 1201, 1159, 1135, 1086, 1062, 1029; \( \delta_H \) (500 MHz, CDCl₃) 7.43-7.38 (2H, m, 2×ArH), 7.33-7.25 (3H, m, 3×ArH), 7.13-7.10 (2H, m, 2×ArH), 6.60-6.56 (2H, m, 2×ArH), 4.26 (2H, s, SCH₂), 3.04 (6H, s, NMe₂); \( \delta_C \) (126 MHz, CDCl₃) 153.3, 130.9, 130.3, 129.1, 128.4, 123.0, 110.5, 63.3, 40.0; \( m/z \) (ESI⁺) 298 ([M + Na]⁺, 100%); HRMS (ESI⁺) C₁₅H₁₇NNaO₂S⁺ ([M + Na]⁺) requires 298.0872; found 298.0864.

Benzyl 4-chlorophenyl sulfone, 10w

Table 4, entry 16: General procedure C (Method I) was followed by the use of DABSO (127 mg, 0.53 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 1-chloro-4-iodobenzene (114 mg, 0.48 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10w as a white crystalline solid
(86 mg, 67%); mp 139-142 °C (CH₂Cl₂) {lit.²⁶ 142.5-143.0 °C (methanol)}; δH (400 MHz, CDCl₃) 7.54 (2H, d, J 8.5, 2 × ArH), 7.41 (2H, d, J 8.5, 2 × ArH), 7.37-7.25 (3H, m, 3 × ArH), 7.09 (2H, d, J 7.5, 2 × ArH), 4.32 (2H, s, SC₂H₂); δC (101 MHz, CDCl₃) 140.5, 136.2, 130.8, 130.1, 129.2, 129.0, 128.7, 127.8, 62.9; m/z (ESI⁺) 291 ([M(³⁷Cl) + Na]⁺, 40%), 289 ([M(³⁵Cl) + Na]⁺, 100%). Data in accordance with literature.²⁷

**Benzyl thiophen-3-yl sulfone, 10x**

![](benzyl_thiophen-3-yl_sulfone.png)

**Table 4, entry 17:** General procedure C (Method I) was followed by the use of DABSO (127 mg, 0.53 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 3-iodothiophene (49 µL, 0.48 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10x as a white crystalline solid (71 mg, 62%); mp 123-125 °C (CH₂Cl₂); νmax (neat)/cm⁻¹ 3117, 3093, 3032, 2973, 2920, 1603, 1496, 1457, 1405, 1363, 1295, 1259, 1207, 1186, 1165, 1150, 1120, 1094, 1073, 1030; δH (400 MHz, CDCl₃) 7.75 (1H, d, J 2.0, ArH)), 7.40-7.25 (4H, m, 4 × ArH)), 7.18-7.06 (3H, m, 3 × ArH)), 4.34 (2H, s, SC₂H₂); δC (101 MHz, CDCl₃) 138.2, 133.4, 130.7, 128.9, 128.6, 128.2, 127.7, 126.4, 63.1; m/z (ESI⁺) 261 ([M + Na]⁺, 100%); HRMS (ESI⁺) C₁₁H₁₀NaO₂S₂⁺ ([M + Na]⁺) requires 261.0014; found 261.0012.

**2-(Benzylsulfonyl)dibenzo[b,d]furan, 10y**

![](2-benzylsulfonyl)dibenzo[b,d]furan.png)

**Table 4, entry 18:** General procedure C (Method I) was followed by the use of DABSO (127 mg, 0.53 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 2-iododibenzofuran (141 mg, 0.48 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10y as white needles (99 mg, 64%); mp 205-208 °C (CH₂Cl₂); νmax (neat)/cm⁻¹ 2960, 2927, 2852, 1584, 1442, 1422, 1406, 1346, 1327, 1307, 1295, 1260, 1244, 1199, 1184, 1164, 1154, 1118, 1104, 1071, 1021; δH (200 MHz, CDCl₃) 8.25 (1H, d, J 2.0, ArH), 7.92 (1H, d, J 8.0, ArH), 7.73-7.51 (5H, m, 5 × ArH), 7.47-7.38 (1H, m, ArH), 7.34-7.19 (2H, m, 2 × ArH), 7.15-7.03 (2H, m, 2 × ArH), 4.40 (2H, s, SCH₂); δC (126 MHz, CDCl₃) 158.7, 157.0, 132.3, 130.8, 128.8, 128.6(2), 128.6(0), 128.3, 127.6, 124.8, 123.7, 122.9, 122.3, 121.1, 112.1, 112.0, 63.3; m/z (ESI⁺) 345 ([M + Na]⁺, 100%); HRMS (ESI⁺) C₁₉H₁₄NaO₃S⁺ ([M + Na]⁺) requires 345.0556; found 345.0552.
5-(Benzylsulfonyl)-2-methoxypyridine, 10z

Table 4, entry 19: General procedure C (Method I) was followed by the use of DABSO (127 mg, 0.53 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 5-iodo-2-methoxypyridine (113 mg, 0.48 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10z as a white crystalline solid (88 mg, 70%); mp 114-116 °C (CH₂Cl₂); ν max (neat)/cm⁻¹ 2977, 2950, 2930, 1589, 1559, 1484, 1454, 1431, 1408, 1375, 1310, 1286, 1272, 1257, 1200, 1163, 1098, 1071, 1029, 1006; δH (500 MHz, CDCl₃) 8.39 (1H, dd, J 2.5, 0.5, ArH), 7.60 (1H, dd, J 9.0, 2.5, ArH), 7.37-7.33 (1H, m, ArH), 7.32-7.28 (2H, m, 2 × ArH), 7.14-7.12 (2H, m, 2 × ArH), 6.71 (1H, dd, J 9.0, 0.5, ArH), 4.33 (2H, s, SC₂H₂), 3.99 (3H, s, OMe); δC (126 MHz, CDCl₃) 167.1, 149.2, 138.5, 130.9, 129.0, 128.7, 127.0, 111.0, 63.4, 54.4; m/z (ESI⁺) 286 ([M + Na]⁺, 100%), 264 ([M + H]⁺, 60%); HRMS (ESI⁺) C₁₃H₁₃NNaO₃S⁺ ([M + Na]⁺) requires 286.0508; found 286.0509.

5-(Benzylsulfonyl)-1H-indole, 10aa

Table 4, entry 20: General procedure C (Method I) was followed by the use of DABSO (127 mg, 0.53 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 5-iodoindole (117 mg, 0.48 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10aa as a white crystalline solid (47 mg, 36%); mp 229-231 °C (CH₂Cl₂); ν max (neat)/cm⁻¹ 3366, 3142, 3082, 3031, 2916, 1606, 1503, 1467, 1455, 1424, 1407, 1348, 1327, 1291, 1254, 1204, 1162, 1150, 1133, 1112, 1095, 1060, 1030; δH (500 MHz, CDCl₃) 10.80 (1H, br. s, NH), 8.00-7.99 (1H, m, ArH), 7.59-7.55 (2H, m, ArH), 7.44 (1H, dd, J 8.5, 2.0, ArH), 7.33-7.23 (3H, m, 3 × ArH), 7.18-7.15 (2H, m, 2 × ArH), 6.66-6.64 (1H, m, ArH), 4.47 (2H, s, SCH₂); δC (126 MHz, CDCl₃) 139.4, 131.9, 130.7, 130.5, 128.9, 128.4, 128.3, 123.2, 121.7, 112.4, 104.0, 79.2, 63.2; m/z (ESI⁺) 270 ([M – H]⁻, 100%); HRMS (ESI⁺) C₁₅H₁₃NNaO₃S⁻ ([M + Na]⁻) requires 294.0559; found 294.0558.

Benzyl (1E)-oct-1-en-1-yl sulfone 10ab
Table 4, entry 21: General procedure C (Method I) was followed by the use of DABSO (127 mg, 0.53 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and trans-1-iodo-1-octene (114 mg, 0.48 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10ab as a white crystalline solid (59 mg, 46%); mp 28-29 °C (CH₂Cl₂); ν\text{max} (neat)/cm⁻¹ 2928, 2870, 2858, 1496, 1456, 1376, 1303, 1285, 1256, 1221, 1202, 1161, 1151, 1113, 1072, 1030; δH (500 MHz, CDCl₃) 7.39-7.33 (5H, m, 5 × ArH), 6.71 (1H, dt, J 15.0, 7.0, CH=CHS), 6.13 (1H, d, J 15.0, CH=CHS), 4.21 (2H, s, SCH₂), 2.21-2.15 (2H, m, CH₂(CH₂)₄CH₃), 1.37 (2H, app quin, J 6.5, CH₂), 1.28 (2H, app quin, J 6.5, CH₂) overlapping 1.26-1.15 (4H, m, 2 × CH₂), 0.95-0.81 (3H, m, (CH₃)$_₂$CH); δC (126 MHz, CDCl₃) 151.3, 131.3, 129.2(4), 129.2(0), 128.8, 127.2, 61.9, 32.0, 31.9, 29.1, 27.9, 22.9, 14.5; m/z (ESI⁺) 289 ([M + Na]$^+$, 100%); HRMS (ESI⁺) C₁₃H₂₂NaO₂S⁺ ([M + Na]$^+$) requires 289.1233; found 289.1223.

[[Cyclohexylenemethyl]sulfonyl][methyl]benzene benzyl cyclohexylenemethyl sulfone, 10ac

Table 4, entry 22: General procedure C (Method I) was followed by the use of DABSO (127 mg, 0.53 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and (iodomethylidene)cyclohexane$^{28}$ (106 mg, 0.48 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10ac as a white crystalline solid (104 mg, 87%); mp 74-76 °C (CH₂Cl₂); ν\text{max} (neat)/cm⁻¹ 2922, 2855, 1495, 1450, 1439, 1407, 1357, 1339, 1317, 1298, 1251, 1234, 1202, 1179, 1159, 1150, 1115, 1073, 1029; δH (400 MHz, CDCl₃) 7.39-7.33 (5H, m, 5 × ArH), 5.85 (1H, s, C=CHS), 4.20 (2H, s, SCH₂), 2.37 (2H, t, J 6.0, CH₂C=CH), 2.15 (2H, t, J 6.0, CH₂C=CH), 1.66-1.59 (2H, m, CH₂), 1.54-1.47 (2H, m, CH₂), 1.43-1.35 (2H, m, CH₂); δC (101 MHz, CDCl₃) 164.6, 131.1, 128.7(1), 128.6(8), 128.6(5), 119.4, 62.4, 37.7, 29.0, 28.3, 27.3, 25.7; m/z (ESI⁺) 523 ([2M + Na]$^+$, 100%), 273 ([M + Na]$^+$, 50%); HRMS (ESI⁺) C₁₃H₁₈NaO₂S⁺ ([M + Na]$^+$) requires 273.0920; found 273.0922.

Benzyl cyclohept-1-en-1-yl sulfone, 10ad

Table 4, entry 23: General procedure C (Method I) was followed by the use of DABSO (127 mg, 0.53 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), benzyl bromide (142 µL, 1.20 mmol) and 1-iodocycloheptene$^{29}$ (107 mg, 0.48 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10ad as a white crystalline solid (97 mg, 81%); mp 57-58 °C (CH₂Cl₂); ν\text{max} (neat)/cm⁻¹ 3063, 2922, 2849, 1602, 1494, 1445, 1415,
1360, 1297, 1284, 1224, 1147, 1128, 1114, 1077, 1044, 1031; δH (400 MHz, CDCl3) 7.38-7.30 (5H, m, 5 × ArH), 6.85 (1H, t, J 6.5, SC=CH2), 4.16 (2H, s, SCHR2), 2.48-2.43 (2H, m, CH2), 2.26-2.20 (2H, m, CH2), 1.78-1.71 (2H, m, CH2), 1.61-1.54 (2H, m, CH2), 1.54-1.47 (2H, m, CH2); δC (101 MHz, CDCl3) 146.2, 142.1, 130.7, 128.7(3), 128.6(7), 128.4, 59.5, 31.1, 28.7, 28.2, 26.1, 25.3; m/z (ESI+) 523 ([2M + Na]+, 50%), 273 ([M + Na]+, 100%); HRMS (ESI+) C14H18O2S+ ([M + Na]+) requires 273.0920; found 273.0925.

4-Ethoxyphenyl 4-(trifluoromethyl)benzyl sulfone, 10ae

Table 5, entry 1: General procedure C (Method I) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), 1-ethoxy-4-iodobenzene (119 mg, 0.48 mmol) and 4-(trifluoromethyl)benzyl bromide (186 µL, 1.20 mmol). The reaction mixture was stirred at 90 °C for 5 h. Column chromatography (eluent: 7:3, petrol:Et2O) yielded sulfone 10ae as a white crystalline solid (150 mg, 91%); mp 226-228 °C (CH2Cl2); νmax (neat)/cm⁻¹ 2987, 2922, 1592, 1575, 1493, 1477, 1443, 1412, 1394, 1313, 1301, 1292, 1281, 1262, 1244, 1202, 1178, 1152, 1132, 1114, 1083, 1045, 1024; δH (500 MHz, CDCl3) 7.56-7.52 (4H, m, 4 × ArH), 7.26-7.22 (2H, m, 2 × ArH), 6.95-6.84 (2H, m, 2 × ArH), 4.33 (2H, s, SCHR2), 4.09 (2H, q, J 7.0, OCHR2CH3), 1.45 (3H, t, J 7.0, OCHR2CH3); δC (126 MHz, CDCl3) 163.4, 132.5, 131.2, 131.1 (q, JCF 32.0), 130.7, 128.8, 125.4 (q, JCF 4.0), 123.8 (q, JCF 272.5), 114.6, 64.1, 62.6, 14.5; δF (470 MHz, CDCl3) −62.9 (s, CF3); m/z (ESI+) 343 ([M – H]+, 100%); HRMS (ESI+) C16H15F3NaO3S+ ([M + Na]+) requires 367.0586; found 367.0588.

2-Bromobenzyl 4-ethoxyphenyl sulfone, 10af

Table 5, entry 2: General procedure C (Method I) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), 1-ethoxy-4-iodobenzene (119 mg, 0.48 mmol) and 2-bromobenzylbromide (300 mg, 1.20 mmol). The reaction mixture was stirred at 90 °C for 5 h. Column chromatography (eluent: 7:3, petrol:Et2O) yielded sulfone 10af as a white crystalline solid (153 mg, 90%); mp 226-228 °C (CH2Cl2); νmax (neat)/cm⁻¹ 2987, 2922, 1592, 1575, 1493, 1477, 1443, 1412, 1394, 1313, 1301, 1292, 1281, 1262, 1244, 1202, 1178, 1152, 1132, 1114, 1083, 1045, 1024; δH (500 MHz, CDCl3) 7.56-7.51 (2H, m, 2 × ArH), 7.49 (1H, dd, J 8.0, 1.5, ArH), 7.46 (1H, dd, J 8.0, 1.5, ArH), 7.32 (1H, app td, J 7.5, 1.5, ArH), 7.18 (1H,
app td, $J$ 7.5, 1.5, Ar$H$), 6.90-6.86 (2H, m, 2 × Ar$H$), 4.57 (2H, s, SCH$_2$), 4.09 (2H, q, $J$ 7.0, OCH$_2$CH$_3$), 1.44 (3H, t, $J$ 7.0, OCH$_2$CH$_3$); $\delta_c$ (126 MHz, CDCl$_3$) 163.4, 132.9(3), 132.9(0), 131.0, 130.3, 129.4, 128.7, 127.7, 126.0, 114.5, 64.0, 61.8, 14.6; HRMS (FI$^+$) C$_{15}$H$_{15}$OBrS$^+$ ([M(79Br)]$^+$) requires 355.9905; found 355.9903.

4-Ethoxyphenyl naphthalen-2-ylmethyl sulfone, 10ag

![Structural Diagram]

Table 5, entry 3: General procedure C (Method I) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), 1-ethoxy-4-iodobenzene (119 mg, 0.48 mmol) and 2-(bromomethyl)naphthalene (265 mg, 1.20 mmol). The reaction mixture was stirred at 90 °C for 5 h. Column chromatography (eluent: 7:3, petrol:Et$_2$O) yielded sulfone 10ag as a white crystalline solid (146 mg, 93%); mp 128-130 °C (CH$_2$Cl$_2$); $\nu$max (neat)/cm$^{-1}$ 3052, 2977, 2938, 1596, 1579, 1498, 1477, 1442, 1210, 1365, 1313, 1295, 1256, 1211, 1179, 1146, 1114, 1089, 1044; $\delta_h$ (500 MHz, CDCl$_3$) 7.84-7.81 (1H, m, Ar$H$), 7.75 (1H, app br. s, Ar$H$), 7.54-7.46 (4H, m, 4 × Ar$H$), 7.20 (1H, dd, $J$ 8.5, 2.0, Ar$H$), 6.87-6.83 (2H, m, 2 × Ar$H$), 4.45 (2H, s, SCH$_2$), 4.06 (2H, q, J 7.0, OCH$_2$CH$_3$), 1.43 (3H, t, J 7.0, OCH$_2$CH$_3$); $\delta_c$ (126 MHz, CDCl$_3$) 163.1, 133.0, 130.8, 130.5, 129.2, 128.2, 128.0, 127.9, 127.6, 126.6, 126.3, 126.0, 114.5, 64.0, 63.3, 14.5; m/z (ESI$^+$) 349 ([M + Na]$^+$, 100%); HRMS (ESI$^+$) C$_{19}$H$_{18}$NaO$_3$S$^+$ ([M + Na]$^+$) requires 349.0869; found 349.0879.

4-Ethoxyphenyl (2E)-3-phenylprop-2-en-1-yl sulfone, 10ah

![Structural Diagram]

Table 5, entry 4: General procedure C (Method I) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), 1-ethoxy-4-iodobenzene (119 mg, 0.48 mmol) and 3-bromo-1-phenyl-1-propene (178 µL, 1.20 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et$_2$O) yielded sulfone 10ah as a white crystalline solid (75 mg, 51%).

Table 5, entry 4: General procedure C (Method II) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), 1-ethoxy-4-iodobenzene (119 mg, 0.48 mmol) and 3-bromo-1-phenyl-1-propene (142 µL, 0.96 mmol). Column chromatography (eluent: 7:3, petrol:Et$_2$O) yielded sulfone 10ah as a white crystalline solid (120 mg, 83%).
**Sulfone 10ah:** mp 102-104 °C (CH₂Cl₂); ν<sub>max</sub> (neat)/cm<sup>-1</sup> 2938, 1595, 1574, 1494, 1471, 1453, 1414, 1390, 1315, 1296, 1262, 1178, 1141, 1087, 1039; δ<sub>H</sub> (500 MHz, CDCl₃) 7.80-7.76 (2H, m, 2 × ArH), 7.32-7.28 (5H, m, 5 × ArH), 6.98-6.94 (2H, m, 2 × ArH), 6.38 (1H, app d, J 16.0, SCH₂CH=CH), 6.11 (1H, dt, J 16.0, 7.5, SCH₂CH=CH), 4.09 (2H, q, J 7.0, OCH₂CH₃), 3.92 (2H, dd, J 7.5, 1.0, SC₂H=CH), 1.44 (3H, t, J 7.0, OCH₂CH₃); δ<sub>C</sub> (126 MHz, CDCl₃) 163.6, 139.4, 136.3, 131.1, 130.2, 129.1, 128.9, 127.1, 116.0, 115.1, 64.5, 61.2, 15.0; m/z (ESI<sup>+</sup>) 325 ([M + Na]<sup>+</sup>), 100%; HRMS (ESI<sup>+</sup>) C₁₇H₁₈NaO₃S<sup>+</sup> ([M + Na]<sup>+</sup>) requires 325.0869; found 325.0866.

Cyclohex-2-en-1-yl 4-ethoxyphenyl sulfone, 10ai

![Cyclohex-2-en-1-yl 4-ethoxyphenyl sulfone](image)

**Table 5, entry 5:** General procedure C (Method II) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), 1-ethoxy-4-iodobenzene (119 mg, 0.48 mmol) and 3-bromocyclohexene (110 µL, 0.96 mmol). Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10ai as a white crystalline solid (70 mg, 55%); mp 128-130 °C (CH₂Cl₂); ν<sub>max</sub> (neat)/cm<sup>-1</sup> 2962, 2933, 2863, 1591, 1575, 1491, 1471, 1456, 1428, 1410, 1392, 1312, 1285, 1261, 1238, 1216, 1188, 1173, 1137, 1124, 1113, 1105, 1083, 1040, 1018; δ<sub>H</sub> (400 MHz, CDCl₃) 7.80-7.75 (2H, m, 2 × ArH), 7.01-6.96 (2H, m, 2 × ArH), 6.06 (1H, app dtd, J 10.0, 4.0, 2.5, CH=C), 5.78 (1H, app dq, J 10.0, 2.5, CH=C), 4.10 (2H, q, J 7.0, OCH₂CH₃), 3.75-3.68 (1H, m, SCHR), 2.03-1.93 (3H, m), 1.89-1.70 (2H, m), 1.54-1.46 (1H, m) overlapping 1.45 (3H, t, J 7.0, OCH₂CH₃); δ<sub>C</sub> (101 MHz, CDCl₃) 163.1, 135.0, 131.3, 128.6, 118.9, 114.5, 64.0, 62.0, 24.4, 22.8, 19.6, 14.6; m/z (ESI<sup>+</sup>) 289 ([M + Na]<sup>+</sup>), 100%; HRMS (ESI<sup>+</sup>) C₁₄H₁₈NaO₃S<sup>+</sup> ([M + Na]<sup>+</sup>) requires 289.0869; found 289.0874.

2-[(4-Ethoxyphenyl)sulfonyl]cyclohexanol, 10aj

![2-[(4-Ethoxyphenyl)sulfonyl]cyclohexanol](image)

**Table 5, entry 6:** General procedure C (Method I) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), 1-ethoxy-4-iodobenzene (119 mg, 0.48 mmol) and cyclohexene oxide (121 µL, 1.20 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10aj as a white crystalline solid (48 mg, 35%, >20:1 dr by <sup>1</sup>H NMR spectroscopy).

**Table 5, entry 6:** General procedure C (Method II) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), 1-ethoxy-4-iodobenzene (119 mg, 0.48 mmol) and cyclohexene oxide (97 µL, 0.96 mmol). Column
chromatography (eluent: 7:3 to 1:1, petrol:Et₂O) yielded sulfone 10aj as a white crystalline solid (102 mg, 75%, >20:1 dr by ¹H NMR spectroscopy).

**Sulfone 10aj:** mp 69-72 °C (CH₂Cl₂); ν_max (neat)/cm⁻¹ 3511, 2985, 2861, 1592, 1574, 1494, 1468, 1449, 1394, 1357, 1333, 1262, 1211, 1177, 1131, 1113, 1086, 1062, 1040; δ_H (400 MHz, CDCl₃) 7.83-7.77 (2H, m, 2 × ArH), 7.08-6.99 (2H, m, 2 × ArH), 4.40 (1H, br. s, OCH₃), 4.12 (2H, q, J 7.0, OCH₂CH₃), 3.86 (1H, td, J 10.0, 5.0, C(2)OH), 2.98-2.90 (1H, m, SCH₂CH₂CH₃), 1.80-1.66 (2H, m, SCH₂C₆H₄), 1.46 (3H, t, J 7.0, OCH₂CH₃), 0.99 (3H, t, J 7.5, SCH₂CH₂CH₃); δ_C (101 MHz, CDCl₃) 163.1, 130.4, 130.2, 114.8, 69.0, 68.3, 64.2, 34.1, 25.8, 24.6, 23.6, 14.6; m/z (ESI⁺) 307 ([M + Na]⁺, 100%); HRMS (ESI⁺) C₁₄H₂₀NaO₄S⁺ ([M + Na]⁺) requires 307.0975; found 307.0980.

### 4-Ethoxyphenyl propyl sulfone 10ak

![4-Ethoxyphenyl propyl sulfone](image)

**Table 5, entry 7:** General procedure C (Method I) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), 1-ethoxy-4-iodobenzene (119 mg, 0.48 mmol) and 1-iodopropane (117 µL, 1.20 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10ak as a white crystalline solid (60 mg, 55%); mp 39-40 °C (CH₂Cl₂); ν_max (neat)/cm⁻¹ 2979, 2937, 2879, 1593, 1574, 1496, 1476, 1465, 1402, 1384, 1348, 1311, 1290, 1261, 1214, 1180, 1138, 1115, 1089, 1038; δ_H (400 MHz, CDCl₃) 7.85-7.78 (2H, m, 2 × ArH), 7.04-6.96 (2H, m, 2 × ArH), 4.11 (2H, q, J 7.0, OCH₂CH₃), 3.07-3.02 (2H, m, SC(2)H₂CH₃), 1.80-1.66 (2H, m, SCH₂CH₂CH₃), 1.46 (3H, t, J 7.0, OCH₂CH₃), 0.99 (3H, t, J 7.5, SCH₂CH₂CH₃); δ_C (101 MHz, CDCl₃) 163.1, 130.4, 130.2, 114.8, 64.0, 58.3, 16.7, 14.6, 12.9; m/z (ESI⁺) 251 ([M + Na]⁺, 100%); HRMS (ESI⁺) C₁₄H₂₀NaO₃S⁺ ([M + Na]⁺) requires 251.0712; found 251.0707.

### 4-Ethoxyphenyl hexyl sulfone, 10al

![4-Ethoxyphenyl hexyl sulfone](image)

**Table 5, entry 8:** General procedure C (Method I) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), 1-ethoxy-4-iodobenzene (119 mg, 0.48 mmol) and 1-iodohexane (177 µL, 1.20 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10al as a colourless oil (82 mg, 63%); ν_max (neat)/cm⁻¹ 2931, 2871, 1595, 1578, 1496, 1475, 1395, 1314, 1296, 1257, 1216, 1138, 1115, 1089, 1039; δ_H (400 MHz, CDCl₃) 7.84-7.70 (2H, m, 2 × ArH), 7.03-6.95
(2H, m, 2 × ArH), 4.10 (2H, q, J 7.0, OCH2CH3), 3.10-2.99 (2H, m, SCH2(CH2)2CH3), 1.75-1.61 (2H, m, SCH2(CH2)2CH3), 1.44 (3H, t, J 7.0, OCH2CH3), 1.33 (2H, app quin, J 7.0, CH2) overlapping 1.29-1.18 (4H, m, 2 × CH2), 0.84 (3H, t, J 7.0, S(CH2)2CH3); δc (101 MHz, CDCl3) 163.0, 130.5, 130.2, 114.8, 64.0, 56.6, 31.2, 27.9, 22.8, 22.3, 14.6, 13.9; m/z (ESI+) 293 ([M + Na]+, 100%), 271 ([M + H]+, 70%); HRMS (ESI+) C14H12NaO3S+ ([M + Na]+) requires 323.0924; found 323.0913.

**Ethyl 2-[(4-ethoxyphenyl)sulfonyl]-2-methylpropanoate, 10am**

![Chemical structure](image)

Table 5, entry 9: General procedure C (Method I) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), 1-ethoxy-4-iodobenzene (119 mg, 0.48 mmol) and ethyl α-bromoisobutyrate (176 µL, 1.20 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (eluent: 7:3, petrol:Et2O) yielded sulfone 10am as a white crystalline solid (82 mg, 57%); mp 86-89 °C (CH2Cl2); νmax (neat)/cm⁻¹ 2983, 2941, 1731, 1594, 1577, 1497, 1473, 1415, 1394, 1367, 1316, 1298, 1259, 1153, 1128, 1079, 1038, 1023; δt (500 MHz, CDCl3) 7.78-7.74 (2H, m, 2 × ArH), 7.02-6.92 (2H, m, 2 × ArH), 4.15 (2H, q, J 7.0, COOCH2CH3) overlapping 4.12 (2H, q, J 7.0, ArOCH2CH3), 1.61 (6H, s, 2 × SCMe), 1.46 (3H, t, J 7.0, ArOCH2CH3), 1.24 (3H, t, J 7.0, COOCH2CH3); δc (126 MHz, CDCl3) 169.5, 164.0, 133.0, 127.2, 114.7, 69.4, 64.5, 62.7, 20.8, 15.0, 14.3; m/z (ESI+) 323 ([M + Na]+, 100%); HRMS (ESI+) C14H20NaO3S+ ([M + Na]+) requires 323.0924; found 323.0913.

**2-[(4-Ethoxyphenyl)sulfonyl]-5-(trifluoromethyl)benzaldehyde, 10an**

![Chemical structure](image)

Table 5, entry 10: General procedure C (Method II) was followed by the use of DABSO (69 mg, 0.29 mmol), DABCO (27 mg, 0.24 mmol), 4-aminomorpholine (55 µL, 0.58 mmol), 1-ethoxy-4-iodobenzene (119 mg, 0.48 mmol) and 2-fluoro-5-(trifluoromethyl)benzaldehyde (136 µL, 0.96 mmol). Column chromatography (eluent: 7:3, petrol:Et2O) yielded sulfone 10an as a pale yellow crystalline solid (60 mg, 35%); mp 88-90 °C (CH2Cl2); νmax (neat)/cm⁻¹ 2987, 2920, 1705, 1593, 1579, 1496, 1473, 1446, 1417, 1397, 1328, 1256, 1178, 1166, 1151, 1128, 1077, 1039; δt (500 MHz, CDCl3) 10.92 (1H, s, ArCHO), 8.26 (1H, dd, J 1.5, 0.5, ArH), 8.24 (1H, d, J 8.0, ArH), 7.97 (1H, dd, J 8.0, 1.5, ArH), 7.85-7.81 (2H, m, 2 × ArH), 7.05-6.98 (2H, m, 2 × ArH), 4.10 (2H, q, J 7.0, OCH2CH3), 1.44 (3H, t, J 7.0, OCH2CH3); δc (126 MHz, CDCl3) 188.1, 163.7, 146.5, 135.3 (q, JCF 34.0), 134.3, 131.2, 130.1(3) (q, JCF 3.5), 130.1(0), 129.8, 126.6 (q, JCF 3.5), 122.6 (q, JCF 273.0),
115.5, 64.3, 14.5; δν (470 MHz, CDCl3) –63.4 (s, CF3); m/z (ESI⁺) 413 ([M + Na + MeOH]⁺, 100%); m/z (ESI⁻) 425 ([M + Cl + MeOH]⁻, 100%); HRMS (ESI⁻) C16H13F3NaO6S⁺ ([M + Na⁺]) 381.0379; found 381.0367.

5-[2-(Phenylsulfonyl)ethyl]-1H-indole, 13

General procedure C (Method I) was followed by the use of DABSO (53 mg, 0.22 mmol), 4-aminomorpholine (25 µL, 0.24 mmol), iodobenzene (22 µL, 0.20 mmol) and 5-(2-iodoethyl)-1H-indole, 12 (136 mg, 0.50 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (elucent: 7:3 to 3:7, petrol:Et2O) yielded sulfone 13 as an off-white crystalline solid (25 mg, 43%); νmax (neat)/cm⁻¹: 3394, 2920, 2851, 1447, 1304, 1149, 1085; δν (200 MHz, CDCl3) 8.14 (1H, br. s, NH), 8.01-7.92 (2H, m, 2 × ArH), 7.69-7.53 (3H, m, 3 × ArH), 7.40-7.36 (1H, m, ArH), 7.31 (1H, d, J 8.5, ArH), 7.23-7.19 (1H, m, ArH), 6.94 (1H, dd, J 8.5, 1.5, ArH), 6.47 (1H, ddd, J 3.0, 2.0, 1.0, ArH), 3.48-3.37 (2H, m, SCH2CH2Ar), 3.19-3.08 (2H, m, SCH2CH2Ar); δc (126 MHz, CDCl3) 139.1, 134.7, 133.7, 129.3, 128.7, 128.2, 128.1, 124.8, 122.4, 120.1, 111.3, 102.3, 58.4, 28.9; HRMS (ESI⁺) C16H13NNaO6S⁺ ([M + Na⁺]) requires 308.0716; found 308.0718. Data in accordance to literature.³⁰

5-[2-[(4-Ethoxyphenyl)sulfonyl]ethyl]-1H-indole, 14

General procedure C (Method I) was followed by the use of DABSO (29 mg, 0.12 mmol), DABCO (11 mg, 0.10 mmol), 4-aminomorpholine (25 µL, 0.24 mmol), 4-ethoxy-N-(morpholin-4-yl)benzenesulfonamide (56 mg, 0.20 mmol) and 5-(2-iodoethyl)-1H-indole, 12 (136 mg, 0.50 mmol). The reaction mixture was stirred at 90 °C for 20 h. Column chromatography (elucent: 7:3 to 3:7, petrol:Et2O) yielded sulfone 14 as an off-white crystalline solid (41 mg, 63%); mp 89-91 °C (CH2Cl2); νmax (neat)/cm⁻¹: 3400, 2981, 2929, 1594, 1578, 1496, 1475, 1454, 1416, 1395, 1342, 1313, 1260, 1180, 1137, 1087, 1039; δν (400 MHz, CDCl3) 8.13 (1H, br. s, NH), 7.88-7.83 (2H, m, 2 × ArH), 7.38-7.35 (1H, m, ArH), 7.29 (1H, d, J 8.5, ArH), 7.21-7.18 (1H, m, ArH), 7.03-6.98 (2H, m, 2 × ArH), 6.94 (1H, dd, J 8.5, 1.5, ArH), 6.46 (1H, ddd, J 3.0, 2.0, 1.0, ArH), 4.11 (2H, q, J 7.0, ArOCH2CH3), 3.41-3.35 (2H, m, SCH2CH2Ar), 3.14-3.07 (2H, m, SCH2CH2Ar); δc (126 MHz, CDCl3) 163.1, 134.7, 130.4, 130.2, 128.9, 128.2, 124.7, 122.5, 120.1, 114.8, 111.3, 102.3, 64.0, 58.6, 29.1, 14.6; m/z (ESI⁻) 681 ([2M + Na⁻], 40%), 352 ([M + Na⁺], 40%).
Synthesis of benzyl 4-methylphenyl sulfone, 10a using potassium metabisulfite/TBAB and 4-iodotoluene.

An oven-dried tube was charged with tri-tert-butylphosphonium tetrafluoroborate (15 mg, 10 mol%), palladium(II) acetate (5 mg, 5 mol%), 4-iodotoluene (109 mg, 0.50 mmol), potassium metabisulfite (111 mg, 0.50 mmol), HBF$_4$ (14 µL, 0.10 mmol, HBF$_4$ 54 wt. % in Et$_2$O), and tetrabutylammonium bromide (242 mg, 0.75 mmol). 4-Aminomorpholine (58 µL, 0.60 mmol) and 1,4-dioxane (2.0 mL) were added. The reaction mixture was stirred at 80 °C for 21 h (reaction completion by TLC was not observed after 21 h).$^{31}$ K$_2$CO$_3$(aq) (0.52 mL, 2.40 M) and benzyl bromide (149 µL, 1.25 mmol) were added and the reaction mixture stirred at 90 °C for 5 h. After cooling to rt, the suspension was filtered through a short pad of Celite® and washed sequentially with CH$_2$Cl$_2$ (5 mL) and water (5 mL). The organic layer was separated and the aqueous layer extracted with CH$_2$Cl$_2$ (2 × 5 mL). The combined organic extracts were washed with brine, dried over MgSO$_4$, filtered and then concentrated in vacuo. Column chromatography (eluent: 7:3, petrol:Et$_2$O) yielded sulfone 10a as a white crystalline solid (125 mg, 72%).

Synthesis of benzyl 4-methylphenyl sulfone, 10a using potassium metabisulfite/TBAB and 4-bromotoluene.

An oven-dried tube was charged with tri-tert-butylphosphonium tetrafluoroborate (44 mg, 30 mol%), palladium(II) acetate (11 mg, 10 mol%), 4-bromotoluene (62 µL, 0.50 mmol), potassium metabisulfite (222 mg, 1.00 mmol), and tetrabutylammonium bromide (242 mg, 0.75 mmol). 4-Aminomorpholine (72 µL, 1.00 mmol) and 1,4-dioxane (2.0 mL) were added. The reaction mixture was stirred at 100 °C for 21 h (reaction completion by TLC was not observed after 21 h).$^{31}$ K$_2$CO$_3$(aq) (0.52 mL, 2.40 M) and benzyl bromide (149 µL, 1.24 mmol) were added and the reaction mixture stirred at 90 °C for 5 h. After cooling to rt, the suspension was filtered through a short pad of Celite® and washed sequentially
with CH₂Cl₂ (5 mL) and water (5 mL). The organic layer was separated and the aqueous layer extracted with CH₂Cl₂ (2 × 5 mL). The combined organic extracts were washed with brine, dried over MgSO₄, filtered and then concentrated in vacuo. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10a as a white crystalline solid (83 mg, 68%).

**Synthesis of benzyl 4-methoxyphenyl sulfone, 10q using DABSO and 4-methoxyphenylboronic acid.**

An oven-dried tube was charged with 4-methoxyphenylboronic acid (152 mg, 1.00 mmol), palladium(II) acetate (5 mg, 5 mol%), DABSO (240 mg, 1.00 mmol), and tetrabutylammonium bromide (242 mg, 0.75 mmol). 4-Aminomorpholine (48 µL, 0.50 mmol) and 1,4-dioxane (2.0 mL) were added. The reaction mixture was stirred at 80 °C for 16 h under a balloon of O₂. K₂CO₃ (0.52 mL, 2.40 M) and benzyl bromide (149 µL, 1.25 mmol) were added and the reaction mixture stirred at 90 °C for 5 h. After this time, K₂CO₃ (69 mg, 0.50 mmol) and benzyl bromide (59 µL, 0.50 mmol) were added and the reaction mixture stirred at 90 °C for a further 16 h. After cooling to rt, the suspension was filtered through a short pad of Celite® and washed sequentially with CH₂Cl₂ (5 mL) and water (5 mL). The organic layer was separated and the aqueous layer extracted with CH₂Cl₂ (2 × 5 mL). The combined organic extracts were washed with brine, dried over MgSO₄, filtered and then concentrated in vacuo. Column chromatography (eluent: 7:3, petrol:Et₂O) yielded sulfone 10q as a white crystalline solid (113 mg, 86%).

### 5) Mechanistic investigation

To a round-bottomed flask, benzyl bromide (114 µL, 0.96 mmol) was added to a solution of 4-methyl-N-(morpholin-4-yl)benzenesulfonamide 9a (123 mg, 0.48 mmol) and Cs₂CO₃ (313 mg, 0.96 mmol) in 1,4-dioxane (1.6 mL). The reaction mixture was stirred at 40 °C for 20 min and then stirred at 50 °C for 20 min. After cooling to rt, the suspension was filtered through filter paper and the residue washed with CH₂Cl₂ (5 mL). The filtrate was concentrated in vacuo to afford a thick pale yellow oil that was determined by ¹H NMR spectroscopy to be a mixture of the presumed trialkylaminosulfonamide intermediate, N-benzyl-4-methyl-N-morpholinobenzenesulfonamide and unreacted benzyl bromide. To this oil was added benzyl bromide (57 µL, 0.48 mmol), Cs₂CO₃ (150 mg, 0.48 mmol) and 1,4-dioxane (1.6 mL). The reaction mixture was stirred at 100 °C for 1 h and then cooled to rt. The
reaction mixture was diluted with water (5 mL) and washed with CH$_2$Cl$_2$ (3 × 5 mL). The combined organic extracts were washed with brine (5 mL), dried over MgSO$_4$, filtered and then concentrated \textit{in vacuo}. Column chromatography (eluent: 7:3, petrol:Et$_2$O) yielded sulfone 10a as a white crystalline solid (108 mg, 91%) and hydrazone 11 (77 mg, 85%) as a white crystalline solid.
6) $^1$H and $^{13}$C NMR Spectra

4-Ethoxy-N-(morpholin-4-yl)benzenesulfonamide, 9b

$^1$H CDCl$_3$ 400 MHz

$^{13}$C CDCl$_3$ 101 MHz
Diethyl [1-(tert-butoxycarbonyl)-1H-indol-5-yl]propanedioate, 12a

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$, 101 MHz
tert-Butyl 5-(2-hydroxyethyl)-1H-indole-1-carboxylate, 12c

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$, 101 MHz
**tert-Butyl 5-(2-iodoethyl)-1H-indole-1-carboxylate, 12d**

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$, 101 MHz
5-(2-Iodoethyl)-1H-indole, 12

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$, 101 MHz

N
H
I

ppm
Benzyl 4-methylphenyl sulfone, 10a

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$, 101 MHz
$N$-[(E)-Phenylmethyldiene]morpholin-4-amine, 11

$^1$H CDCl$_3$, 400 MHz

$^13$C CDCl$_3$, 101 MHz
Methyl 4-methylphenyl sulfone, 10b

$^1$H CDCl$_3$ 400 MHz

$^{13}$C CDCl$_3$ 101 MHz
4-Methylphenyl propyl sulfone, 10c

$^1$H CDCl$_3$ 400 MHz

$^{13}$C CDCl$_3$ 101 MHz
Hexyl 4-methylphenyl sulfone, 10d

$^1$H CDCl$_3$ 400 MHz

$^{13}$C CDCl$_3$ 101 MHz
Hexyl 4-methylbenzenesulfinate, 10’d

$^1\text{H} \text{CDCl}_3, 400 \text{ MHz}$

$^{13}\text{C} \text{CDCl}_3 101 \text{ MHz}$
Benzyl 4-ethoxyphenyl sulfone, 10e

$^1$H (CD$_3$)$_2$CO 400 MHz

$^{13}$C (CD$_3$)$_2$CO 101 MHz
Benzyl 4-(trifluoromethyl)phenyl sulfone, 10f

$^1$H CDCl$_3$ 500 MHz

$^{13}$C CDCl$_3$ 126 MHz
4-Methylphenyl propan-2-yl sulfone, 10g

$^1$H (CD$_3$)$_2$SO 400 MHz

$^{13}$C (CD$_3$)$_2$SO 101 MHz
Ethyl 2-methyl-2-[(4-methylphenyl)sulfonyl]propanoate, 10h

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$, 101 MHz
Ethenyl 4-methylphenyl sulfone, 10i

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$, 101 MHz
2-[(4-Methylphenyl)sulfonyl]-5-(trifluoromethyl)benzaldehyde, 10j

$^1$H CDCl$_3$ 500 MHz

$^13$C CDCl$_3$ 126 MHz

S47
1-Methyl-4-(phenylsulfonyl)benzene, 10k

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$, 126 MHz
Benzyl phenyl sulfone, 10l

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$, 101 MHz
Benzyl 4-tert-butylphenyl sulfone, 10m

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$, 101 MHz
4-(Benzylsulfonyl)biphenyl, 10n

$^1$H CDCl$_3$, 500 MHz

$^{13}$C CDCl$_3$, 126 MHz
Benzyl 2-naphthyl sulfone, 10o

$^1$H CDCl$_3$, 500 MHz

$^{13}$C CDCl$_3$, 126 MHz
Benzyl 2,4-dimethylphenyl sulfone, 10p

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$, 101 MHz
Benzyl 4-methoxyphenyl sulfone, 10q

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$, 101 MHz
Benzyl 3-ethoxyphenyl sulfone, 10r

$^1$H CDCl$_3$, 500 MHz

$^{13}$C CDCl$_3$, 126 MHz
Benzyl 2-ethoxyphenyl sulfone, 10s

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$, 101 MHz
Benzyl 4-(methylsulfanyl)phenyl sulfone, 10t

$^1$H CDCl$_3$, 500 MHz

$^{13}$C CDCl$_3$, 126 MHz
4-(Benzylsulfonyl)aniline, 10u

$^1$H (CD$_3$)$_2$SO 400 MHz

$^{13}$C (CD$_3$)$_2$SO 126 MHz
4-(Benzylsulfonyl)-N,N-dimethylaniline, 10v

$^1$H CDCl$_3$, 500 MHz

$^{13}$C CDCl$_3$, 126 MHz
Benzyl 4-chlorophenyl sulfone, 10w

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$, 101 MHz
Benzyl thiophen-3-yl sulfone, 10x

$^1$H CDCl$_3$ 400 MHz

$^1^3$C CDCl$_3$ 101 MHz
2-(Benzylsulfonyl)dibenz[\(b,d\)]furan, 10y

\(^1\)H CDCl\(_3\) 200 MHz

\(^{13}\)C CDCl\(_3\) 126 MHz
5-(Benzylsulfonyl)-2-methoxypyridine, 10z

$^1$H CDCl$_3$, 500 MHz

$^{13}$C CDCl$_3$, 126 MHz
5-(Benzylsulfonyl)-1H-indole, 10aa

$^1H$ (CD$_3$)$_2$CO 500 MHz

$^{13}C$ (CD$_3$)$_2$CO 126 MHz

S64
Benzyl (1E)-oct-1-en-1-yl sulfone, 10ab

$^1$H CDCl$_3$ 500 MHz

$^{13}$C CDCl$_3$ 126 MHz
{[(Cyclohexylidenemethyl)sulfonyl]methyl}benzene benzyl cyclohexylidenemethyl sulfone, 10ac

$^1H$ CDCl$_3$ 400 MHz

$^{13}C$ CDCl$_3$ 101 MHz
Benzyl cyclohept-1-en-1-yl sulfone, 10ad

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$, 101 MHz
4-Ethoxyphenyl 4-(trifluoromethyl)benzyl sulfone, 10ae

$^1$H CDCl$_3$ 500 MHz

$^{13}$C CDCl$_3$ 126 MHz
2-Bromobenzyl 4-ethoxyphenyl sulfone, 10af

$^1$H CDCl$_3$, 500 MHz

$^{13}$C CDCl$_3$, 126 MHz
4-Ethoxyphenyl naphthalen-2-ylmethyl sulfone, 10ag

$^1$H CDCl$_3$ 500 MHz

$^{13}$C CDCl$_3$ 126 MHz
4-Ethoxyphenyl (2E)-3-phenylprop-2-en-1-yl sulfone, 10ah

$^1$H CDCl$_3$, 500 MHz

$^{13}$C CDCl$_3$, 126 MHz
Cyclohex-2-en-1-yl 4-ethoxyphenyl sulfone, 10ai

$^1$H CDCl$_3$, 400 MHz

$^1$H NMR spectrum of Cyclohex-2-en-1-yl 4-ethoxyphenyl sulfone, 10ai in CDCl$_3$ at 400 MHz.

$^{13}$C CDCl$_3$, 101 MHz

$^{13}$C NMR spectrum of Cyclohex-2-en-1-yl 4-ethoxyphenyl sulfone, 10ai in CDCl$_3$ at 101 MHz.
2-[(4-Ethoxyphenyl)sulfonyl]cyclohexanol, 10aj

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$, 101 MHz
4-Ethoxyphenyl propyl sulfone, 10ak

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$, 101 MHz
4-Ethoxyphenyl hexyl sulfone, 10al

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$ 101 MHz

$\text{EtO}$

$\text{Me}$
Ethyl 2-[(4-ethoxyphenyl)sulfonyl]-2-methylpropanoate, 10am

$^1$H CDCl$_3$ 500 MHz

$^{13}$C CDCl$_3$ 126 MHz
2-[(4-Ethoxyphenyl)sulfonyl]-5-(trifluoromethyl)benzaldehyde, 10an

$^1$H CDCl$_3$ 500 MHz

$^{13}$C CDCl$_3$ 126 MHz
5-[2-(Phenylsulfonyl)ethyl]-1H-indole, 13

$^1$H CDCl$_3$, 200 MHz

$^{13}$C CDCl$_3$, 126 MHz
5-{2-[(4-Ethoxyphenyl)sulfonyl]ethyl}-1H-indole, 14

$^1$H CDCl$_3$, 400 MHz

$^{13}$C CDCl$_3$, 126 MHz
7) References