Mechanosynthesis of sulfonamides via a telescopic, environmentally friendly, and cost-effective process

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1. General information

Commercially available reagents were purchased from Acros, Aldrich, Strem Chemicals, Alfa-Aesar, TCI Europe and used as received. All reactions were monitored by thin-layer chromatography (TLC) performed on glass-backed silica gel 60 F254, 0.2 mm plates (Merck), and compounds were visualized under UV light (254 nm) or using cerium ammonium molybdate solution with subsequent heating. The eluents were technical grade. Mechanochemical reactions were carried out using a FormTech FTS-1000 Shaker Mill® apparatus. The reagents were milled using a zirconia SmartSnapTM grinding jar (15 mL) equipped with balls ($\phi = 8$ mm) of the same material. Precisely, the zirconium oxide of the vessels and balls used for all reactions accomplished in the mixer mill is yttrium oxide stabilized (ZrO₂-Y). These parameters were applied if not stated otherwise. ¹H and ¹³C liquid NMR spectra were recorded on a Varian 500 MHz and Bruker Avance III HD 600 MHz NMR spectrometer at 298 K and were calibrated using trimethylsylane (TMS). Proton chemical shifts are expressed in parts per million (ppm, δ scale) and are referred to the residual hydrogen in the solvent (CHCl₃, 7.27 ppm or DMSO 2.54 ppm). Data are represented as follows: chemical shift, multiplicity (s = singlet, d =doublet, t = triplet, q = quartet, m = multiplet and/or multiple resonances, bs = broad singlet, and combination of thereof), coupling constant (J) in Hertz (Hz) and integration. Carbon chemical shifts are expressed in parts per million (ppm, δ scale) and are referenced to the carbon resonances of the NMR solvent (CDCl₃, δ 77.0 ppm or δ DMSO-d6 δ 39.5 ppm). Deuterated NMR solvents were obtained from Aldrich. Samples were analyzed using an Agilent 5977B MS interfaced to the GC 7890B equipped with a DB-5ms column (J & W), injector temperature at 230 °C, detector temperature at 280 °C, helium carrier gas flow rate of 1 ml/min. The GC oven temperature program was 60°C initial temperature with 4 min hold time and ramping at 15°C/min to a final temperature of 270°C with 7 min hold time. 1 μ L of each sample was injected in split (1:20) mode. After a solvent delay of 3 minutes mass spectra were acquired in full scan mode using 2.28 scans/s with a mass range of 50–500 Amu. Retention times of different compounds were determined by injecting pure compound under identical conditions. All the experiments were carried out in duplicate to ensure reproducibility of the experimental data. Yields refer to pure isolated materials.

2. List of Compounds



Figure F1. List of the used organic reagents.

3. Mechanochemical synthesis of 2a-n and 3-15a-n

General Procedure A for the preparation of sulfonyl chloride (2a-n): a 15 mL ZrO₂ jar equipped with two ZrO₂ balls ($\Phi = 8 \text{ mm}$, mass_{tot} = 3.22 g) was charged with NaHSO₄ (10 mol%, 0.1 mmol), followed by disulfide (**1a-n**) (1.0 mmol) and NaOCl*5 H₂O (6 mmol). (*Attention!* It is crucial to add the reagents in the written order for the success of the reaction since a variable loss of gas could affect the yields). The jar was then closed and the mechanochemical reaction was conducted for 40 min at a frequency of 30 Hz. At the end of the reaction, the crude was recovered with 5 mL of AcOEt, filtered and concentrated under reduced pressure to afford the desired product **2a-n**. For the synthesis of **2h**, the amount of NaOCl*5H₂O had to be raised up to 7 mmol. For the synthesis of **2d-f** and **2i** the reaction time raised up to 180 min, and a short silica pad (Hexane/AcOEt: 3/7) was required for further purification.

General Procedure B for the preparation of sulfonamides (3-15 a-n): a 15 mL ZrO₂ jar equipped with two ZrO₂ balls ($\Phi = 8 \text{ mm}$, mass_{tot} = 3.22 g) was charged with sulphonyl chloride **2a-n** (1 mmol), amine **3-15** (1.1 mmol) and MgO (4 mmol) The jar was then closed, and the reaction was conducted for 120 min at a frequency of 30 Hz. At the end of the reaction, the crude was recovered with AcOEt (5 ml) from the jar and filtered. The filtrate was washed with an aqueous solution of citric acid 3x5 mL (10% w/w), to obtain the desired sulphonamide product **3-15a-n**. In some cases, a further extraction with AcOEt of the aqueous phase was required to improve the yields. A short silica pad (Hexane/AcOEt: 3/7) was required for compound **6k**. For the synthesis of sulfonamides **9d** and **10d**, *N*-methyl imidazole was added as LAG ($\eta = 0.6 \mu L/mg$) and the reaction was conducted for 180 min.

General One Pot Procedure C for the preparation of sulfonamides (3-15a-n): a 15 mL ZrO₂ jar equipped with two ZrO₂ balls ($\Phi = 8$ mm, mass_{tot} = 3.22 g) was charged with NaHSO₄ (10 mol%, 0.1 mmol), followed by disulfide (**1a-n**) (1.0 mmol) and NaOCl*5H₂O (6 mmol). (*Attention!* It is crucial to add the reagents in the written order for the success of the reaction since a variable loss of gas could affect the yields). The jar was then closed and the mechanochemical reaction was conducted for 40 min at a frequency of 30 Hz (for the synthesis of **2h**, the amount of NaOCl*5 H₂O had to be raised up to 7 mmol. For the synthesis of **2d-f** and **2i** the reaction time raised up to 180 min). At the end of the first step, the jar was opened and the amine **3-15** (2.2 mmol) and MgO (4 mmol) were added to the mixture. The jar was then closed, and the reaction was conducted for 90-120 min at a milling frequency of 30 Hz. Once the second step was ended, the crude was recovered from the jar with 10 mL of AcOEt and filtered. The filtrate was washed with an aqueous solution of citric acid 3x5 mL (10% w/w), to obtain the desired sulphonamide product **3-15a-n**. In some cases, a further extraction with AcOEt of the aqueous phase was required to improve the yields. A short silica pad (Hexane/AcOEt: 3/7) was required for compound **6k**. For the synthesis of sulfonamides **9d** and **10d**, *N*-methyl imidazole was added as LAG ($\eta = 0.6 \mu L/mg$) and the reaction was conducted for 3h.

<u>Please note:</u> for the preparation of the non-commercially available compounds **1c**, **1g**, **1i** and **1m** a solution-based methodology has been used. Please see this link to the following paper to know more about it: https://pubs.rsc.org/en/content/articlelanding/2017/CC/C7CC02652H.

4. Screening







Figure F2. Plotted screening of bases used in relation to their equivalents.

Table T2. Reaction optimization for the pharmaceutical scope.



Mechanochemical conditions for this screening: Reaction conditions: sulfonyl chloride **2d** (1 mmol), amine **9** (1.1 equiv.), MgO (4.00 mmol) and a solvent (LAG, $\eta = 0.6 \,\mu$ L/mg) were placed in a 15 mL zirconia jar equipped with two zirconia balls ($\Phi = 8 \,\text{mm}$, 3.22 g) at a frequency of 30 Hz for 180 min.



Scheme S1. Mechanochemical oxidation reaction of disulfides mediated by NaOCI*5H₂O and divergent chlorination pathway promoted by atmospheric CO₂.

5. Compounds

Benzenesulphonyl chloride (2a)



The title compound was synthesized according to the general procedure A stated above. **1a** (218.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol) were used, to afford **2a** as a yellowish oil (349.69 mg, 0.99 mmol, 99%).

 $^{1}\textbf{H NMR} (600 \text{ MHz}, \text{CDCl}_{3}): \delta 8.06 - 8.04 \text{ (m, 2H)}, 7.77 - 7.74 \text{ (m, 1H)}, 7.65 - 7.62 \text{ (m, 2H)}.$

¹³C NMR (151 MHz, CDCl₃): δ 144.6, 135.4, 129.8, 127.1.

The spectroscopic data closely match the ones previously reported in the literature.¹

4-(Methyl)benzenesulphonyl chloride (2b)



The title compound was synthesized according to the general procedure A stated above. **1b** (246.39 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol) were used, to afford **2b** as a white solid. (369.84 mg, 0.97 mmol, 97%).

¹H NMR (600 MHz, CDCl₃) δ 7.92-7.90 (d, J = 8.3 Hz, 2H), 7.41-7.40 (d, J = 8.3 Hz, 2H), 2.49 (s, 3H).
¹³C NMR (151 MHz, CDCl₃) δ 147.0, 141.8, 130.4, 127.1, 21.9.

M.P.: 69-71 °C.

The spectroscopic data closely match the ones previously reported in the literature.¹

4-(*tert*-Butyl)benzenesulphonyl chloride (2c)



The title compound was synthesized according to the general procedure A stated above. **1c** (330.55 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol) were used, to afford **2c** as a white solid. (442.17 mg, 0.95 mmol, 95%).

¹**H NMR** (600 MHz, CDCl₃): δ 7.97 – 7.95 (m, 2H), 7.63 – 7.61 (m, 2H), 1.37 (s, 9H).

¹³C NMR (151 MHz, CDCl₃): δ 159.8, 141.7, 127.1, 126.8, 35.7, 31.1.

M.P.: 80-82 °C.

The spectroscopic data closely match the ones previously reported in the literature.²

4-(Nitro)benzenesulphonyl chloride (2d)



The title compound was synthesized according to the general procedure A stated above. **1d** (308.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol) were used, to afford **2d** as a yellow solid. (345.71 mg, 0.78 mmol, 78%).

¹**H NMR** (600 MHz, CDCl₃) δ 8.49 – 8.47 (m, 2H), 8.27 – 8.26 (m, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 151.5, 148.8, 128.7, 125.2.

M.P.: 78 °C.

The spectroscopic data closely match the ones previously reported in the literature.²

3-(Nitro)benzenesulphonyl chloride (2e)



The title compound was synthesized according to the general procedure A stated above. **1e** (308.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol) were used, to afford **2e** as a yellow solid. (354.58 mg, 0.80 mmol, 80%).

¹**H NMR** (600 MHz, CDCl₃) δ 8.88-8.86 (t, J = 2.0 Hz, 1H), 8.62-8.60 (ddd, J = 8.3, 2.2, 1.0 Hz, 1H), 8.39-8.37 (ddd, J = 7.9, 1.9, 1.0 Hz, 1H), 7.93-7.90 (t, J = 8.1 Hz, 1H).

¹³C NMR (151 MHz, CDCl₃) δ 148.5, 145.6, 132.4, 131.5, 129.7, 122.5.

M.P.: 60-62 °C.

The spectroscopic data closely match the ones previously reported in the literature.²

2-(Nitro)benzenesulphonyl chloride (2f)



The title compound was synthesized according to the general procedure A stated above. **1f** (308.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), and NaHSO₄ (12.01 mg, 0.10 mmol) were used to afford **2f** as a yellow solid. (332.42 mg, 0.75 mmol, 75%).

¹**H NMR** (600 MHz, CDCl₃) δ 8.27 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.93 (ddd, *J* = 8.6, 7.3, 1.4 Hz, 1H), 7.90 – 7.87 (m, 1H), 7.86 – 7.84 (m, 1H).

¹³C NMR (151 MHz, CDCl₃) δ 146.2, 136.6, 136.2, 133.0, 130.6, 125.4.

M.P.: 65-67 °C.

The spectroscopic data closely match the ones previously reported in the literature.³

4-(Fluoro)benzenesulphonyl chloride (2g)



The title compound was synthesized according to the general procedure A stated above. **1g** (254.31 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol) were used to afford **2g** as a white solid. (350.28 mg, 0.90 mmol, 90%).

¹**H NMR** (600 MHz, CDCl₃) δ 8.10 – 8.07 (m, 2H), 7.32 – 7.29 (m, 2H).

¹³**C NMR** (151 MHz, CDCl₃) δ 166.4 (d, $J_{C-F} = 260.6$ Hz), 140.3 (d, $J_{C-F} = 3.0$ Hz), 130.1 (d, $J_{C-F} = 10.1$ Hz), 117.1 (d, $J_{C-F} = 23.2$ Hz).

M.P.: 30-31 °C.

The spectroscopic data closely match the ones previously reported in the literature.²

4-(Chloro)benzenesulphonyl chloride (2h)



The title compound was synthesized according to the general procedure A stated above. **1h** (287.22 mg, 1.00 mmol), NaOCl*5H₂O (1151.64 mg, 7.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol) were used, to afford **2h** as a white solid. (405.24 mg, 0.98 mmol, 98%).

¹**H NMR** (600 MHz, CDCl₃) δ 8.00 – 7.98 (m, 2H), 7.62 – 7.59 (m, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 142.8, 142.4, 130.2, 128.6.

M.P.: 54-56 °C.

The spectroscopic data closely match the ones previously reported in the literature.²

4-(Trifluoromethyl)benzenesulphonyl chloride (2i)



The title compound was synthesized according to the general procedure A stated above. **1i** (354.33 mg, 1.00 mmol), NaOCl $5H_2O$ (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol) were used, to afford **2i** as a white solid. (484.33 mg, 0.99 mmol, 99%).

¹**H NMR** (600 MHz, CDCl₃) δ 8.20 – 8.19 (m, 2H), 7.92 – 7.91 (m, 2H).

¹³**C** NMR (151 MHz, CDCl₃) δ 147.3, 137.0 (q, J_{C-F} = 33.3 Hz), 136.7, 127.0 (q, J_{C-F} = 3.7 Hz), 121.9 (q, J_{C-F} = 273.7 Hz).

M.P.: 31-33 °C.

The spectroscopic data closely match the ones previously reported in the literature.²

4-(Methoxy)benzenesulphonyl chloride (2j)



The title compound was synthesized according to the general procedure A stated above. **1j** (354.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol) were used, to afford **2j** as a white solid. (409.15 mg, 0.99 mmol, 99%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.99 – 7.97 (m, 2H), 7.06 – 7.04 (m, 2H), 3.92 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 165.0, 136.3, 129.7, 114.9, 56.1.

M.P.: 40-41 °C

The spectroscopic data closely match the ones previously reported in the literature.^{1,2}

Dimethylsolfonyl chloride (2k)



The title compound was synthesized according to the general procedure A stated above. **11** (94.19 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol) were used, to afford **21** as a colourless oil. (GC-MS conversion: 99%).

2-Isopropylsolfonyl chloride (21)



The title compound was synthesized according to the general procedure A stated above. **11** (150.30 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol) were used, to afford **21** as a white solid. (282.35 mg, 0.99 mmol, 99%).

¹**H** NMR (600 MHz, CDCl₃) δ 3.80 – 3.70 (hept, J = 6.7 Hz, 1H), 1.61 – 1.58 (dd, J = 6.7, 1.1 Hz, 6H).

¹³C NMR (151 MHz, CDCl₃) δ 67.5, 17.4.

The spectroscopic data closely match the ones previously reported in the literature.⁴

2,4,6-Trimethylbenzenesulfonyl chloride (**2m**)

The title compound was synthesized according to the general procedure A stated above. **1m** (302,49 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol) were used, to afford **2n** as a pale yellow solid. (411,12 mg, 0.94 mmol, 94%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.03 (s, 2H), 2.73 (s, 6H), 2.35 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 145.6, 140.1, 139.6, 132.4, 23.0, 21.3.

M.P.: 58 °C

Me

MeO

Pyridine-2-sulfonyl chloride (2n)

 $\begin{array}{c} O \\ S \\ C \\ V \end{array} \begin{array}{c} \mathsf{N} \\ \mathsf{C} \mathsf{I} \end{array} \begin{array}{c} \mathsf{The title compound was synthesized according to the general procedure A stated above.$ **1n**(220,31 mg, 1.00 mmol), NaOCI*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol) were used, to afford**2n** $as a viscous yellow oil. (310,13 mg, 0.87 mmol, 87%). \end{array}$

¹**H** NMR (600 MHz, CDCl₃) δ 8.81 (d, J = 4.7 Hz, 1H), 8.12 – 8.03 (m, 2H), 7.72 – 7.66 (m, 1H).

¹³C NMR (151 MHz, CDCl₃) δ 159.2, 150.8, 139.2, 129.3, 122.0.

N,*N*-dibenzyl-benzenesulphonamide (**3a**)



The title compound was synthesized according to the general procedure B stated above. **2a** (176.61 mg, 1.00 mmol), **3** (217.01 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **3a** as a white solid (303.70 mg, 0.90 mmol, 90%).

The title compound was synthesized according to the general procedure C stated above. **1a** (218.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **3** (434.02 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **3a** as a white solid (607.40 mg, 1.8 mmol, 90%).

¹**H NMR** (600 MHz, CDCl₃): δ 7.87 – 7.85 (m, 2H), 7.59 – 7.58 (m, 1H), 7.53 – 7.50 (m, 2H), 7.22 – 7.21 (m, 6H), 7.05 – 7.04 (m, 4H), 4.34 (s, 4H).

¹³C NMR (151 MHz, CDCl₃): δ 141.0, 135.7, 132.6, 129.3, 129.2, 128.7, 128.6, 127.8, 50.6.

M.P.: 74-76 °C

The spectroscopic data closely match the ones previously reported in the literature.⁵

N,*N*-Dibenzyl-4-(methyl)benzenesulphonamide (**3b**)



The title compound was synthesized according to the general procedure B stated above. **2b** (190.64 mg, 1.00 mmol), **3** (217.01 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **3b** as a white solid (309.28 mg, 0.88 mmol, 88%).

The title compound was synthesized according to the general procedure C stated above. **1b** (246.39 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **3** (434.02 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **3b** as a white solid (618.57 mg, 1.76 mmol, 88%).

¹**H** NMR (600 MHz, CDCl₃): δ 7.76 – 7.75 (d, *J* = 7.9 Hz, 2H), 7.32 (d, *J* = 7.9 Hz, 2H), 7.23 – 7.22 (m, 6H), 7.08 – 7.06 (dd, *J* = 6.5, 3.1 Hz, 4H), 4.33 (s, 4H), 2.45 (s, 3H).

¹³C NMR (151 MHz, CDCl₃): δ 143.4, 137.8, 135.8, 128.7, 128.6, 128.4, 127.8, 127.7, 50.7, 21.6.

M.P.: 75-77 °C

The spectroscopic data closely match the ones previously reported in the literature.⁶

N,*N*-Dibenzyl-4-(tert-buthyl)benzenesulphonamide (**3c**)



The title compound was synthesized according to the general procedure B stated above. **2c** (232.72 mg, 1.00 mmol), **3** (217.01 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **3c** as a white solid (334.52 mg, 0.85 mmol, 85%).

The title compound was synthesized according to the general procedure C stated above. **1c** (330.55 mg, 1.00 mmol), NaOCl $*5H_2O$ (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **3** (434.02 mg, 2.2 mmol), MgO (161.20 mg, 1.20 mg, 1.20 mg)

4.00 mmol) were used, to afford 3c as a white solid (669.04 mg, 1.70 mmol, 85%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.78 – 7.76 (m, 2H), 7.52 – 7.50 (m, 2H), 7.22 – 7.18 (m, 6H), 7.04 – 7.01 (m, 4H), 4.33 (s, 4H), 1.37 (s, 9H).

¹³C NMR (151 MHz, CDCl₃) δ 156.5, 137.9, 135.9, 128.7, 128.5, 127.8, 127.2, 126.2, 50.6, 35.3, 31.3.

N,*N*-Dibenzyl-4-(nitro)benzenesulphonamide (**3d**)



The title compound was synthesized according to the general procedure B stated above. **2d** (221.61 mg, 1.00 mmol), **3** (217.01 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **3d** as a brown solid (378.61 mg, 0.99 mmol, 99%).

The title compound was synthesized according to the general procedure C stated above. **1d** (308.33 mg, 1.00 mmol), NaOCl $*5H_2O$ (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **3** (434.02 mg, 2.2 mmol), MgO (161.20 mg,

4.00 mmol) were used, to afford **3d** as a brown solid (757.21 mg, 1.98 mmol, 99%).

¹**H NMR** (600 MHz, CDCl₃) δ 8.29 – 8.27 (m, 2H), 7.92 – 7.91 (m, 2H), 7.26 – 7.25 (m, 6H), 7.10 – 7.08 (m, 4H), 4.35 (s, 4H).

¹³C NMR (151 MHz, CDCl₃) δ 149.8, 146.8, 134.9, 128.7, 128.6, 128.3, 128.1, 124.2, 50.8.

M.P.: 126 – 127 °C

The spectroscopic data closely match the ones previously reported in the literature.⁷

N,*N*-Dibenzyl-3-(nitro)benzenesulphonamide (**3e**)



The title compound was synthesized according to the general procedure B stated above. **2e** (221.61 mg, 1.00 mmol), **3** (217.01 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **3e** as a brown solid (370.96 mg, 0.97 mmol, 97%).

The title compound was synthesized according to the general procedure C stated above. **1e** (308.33 mg, 1.00 mmol), NaOCl $*5H_2O$ (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **3** (434.02 mg, 2.2 mmol), MgO (161.20 mg,

4.00 mmol) were used, to afford **3e** as a brown solid (741.91 mg, 1.94 mmol, 97%).

¹**H** NMR (600 MHz, CDCl₃) δ 8.55 – 8.52 (t, *J* = 2.0 Hz, 1H), 8.38 – 8.37 (ddd, *J* = 8.0, 2.0, 1.0 Hz, 1H), 8.07 – 8.06 (ddd, *J* = 8.0, 2.0, 1.0 Hz, 1H), 7.67 – 7.64 (t, *J* = 8.0 Hz, 1H), 7.28 – 7.26 (m, 6H), 7.15 – 7.13 (m, 4H), 4.44 (s, 4H).

¹³C NMR (151 MHz, CDCl₃) δ 148.3, 143.3, 135.0, 132.6, 130.4, 128.8, 128.7, 128.3, 126.9, 122.4, 51.0.

M.P.: 105-106°C

N,*N*-Dibenzyl-4-(fluoro)benzenesulphonamide (**3**g)



The title compound was synthesized according to the general procedure B stated above. 2g (194.60 mg, 1.00 mmol), 3 (217.01 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford 3g as a white solid (351.88 mg, 0.99 mmol, 99%).

The title compound was synthesized according to the general procedure C stated above. **1g** (254.31 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **3** (434.02 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **3g** as a white solid (703.75 mg, 1.98 mmol, 99%).

¹**H NMR** (600 MHz, CDCl₃): δ 7.84 – 7.81 (m, 2H), 7.26 – 7.22 (m, 6H), 7.18 – 7.14 (m, 2H), 7.07 – 7.06 (m, 4H), 4.34 (s, 4H).

¹³**C NMR** (151 MHz, CDCl₃): δ 165.1 (d, $J_{C-F} = 255.2$ Hz), 137.1, 137.0, 135.5, 130.0 (d, $J_{C-F} = 15.1$ Hz), 128.7, 127.9, 116.4 (d, $J_{C-F} = 30.2$ Hz), 50.6.

M.P.: 89-90 °C

The spectroscopic data closely match the ones previously reported in the literature.⁸

N,*N*-dibenzyl-4-(chloro)benzenesulphonamide (**3h**)



The title compound was synthesized according to the general procedure B stated above. **2h** (211.06 mg, 1.00 mmol), **3** (217.01 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **3h** as a white solid (345.85 mg, 0.93 mmol, 93%).

The title compound was synthesized according to the general procedure C stated above. **1h** (287.22 mg, 1.00 mmol), NaOCl $*5H_2O$ (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **3** (434.02 mg, 2.2 mmol), MgO (161.20 mg, 4.00

mmol) were used, to afford **3h** as a white solid (691.70 mg, 1.86 mmol, 93%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.75 – 7.73 (m, 2H), 7.46 – 7.45 (m, 2H), 7.26 – 7.23 (m, 6H), 7.07 – 7.06 (m, 4H), 4.33 (s, 4H).

¹³C NMR (151 MHz, CDCl₃) δ 139.2, 138.8, 135.3, 129.3, 128.7, 128.6, 128.5, 127.9, 50.6.

M.P.: 92 °C

The spectroscopic data closely match the ones previously reported in the literature.⁹

N,*N*-Dibenzyl-4-(trifluoromethyl)benzenesulphonamide (**3i**)



The title compound was synthesized according to the general procedure B stated above. **2i** (244.61 mg, 1.00 mmol), **3** (217.01 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **3i** as a white solid (381.11 mg, 0.94 mmol, 94%).

The title compound was synthesized according to the general procedure C stated above. **1i** (354.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **3** (434.02 mg, 2.2 mmol), MgO (161.20 mg,

4.00 mmol) were used, to afford **3i** as a white solid (762.23 mg, 1.88 mmol, 94%).

¹**H** NMR (600 MHz, CDCl₃) δ 7.90 – 7.89 (d, *J* = 8.1 Hz, 2H), 7.72 – 7.71 (d, *J* = 8.1 Hz, 2H), 7.24 – 7.20 (dd, *J* = 5.1, 2.0 Hz, 6H), 7.07 – 7.05 (m, 4H), 4.37 (s, 4H).

¹³**C NMR** (151 MHz, CDCl₃) δ 144.6, 135.2, 134.2 (q, J_{C-F} = 287 Hz), 128.3 (q, J_{C-F} = 136 Hz), 126.3, 126.1, 126.0, 124.3, 122.5, 50.8.

M.P.: 91 - 92 °C

N,*N*-Dibenzyl-4-(methoxy)benzenesulphonamide (**3j**)



The title compound was synthesized according to the general procedure B stated above. **2j** (206.64 mg, 1.00 mmol), **3** (217.01 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **3j** as a white solid (304.99 mg, 0.94 mmol, 94%).

The title compound was synthesized according to the general procedure C stated above. **1j** (278.38 mg, 1.00 mmol), NaOC1*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **3** (434.02 mg, 2.2 mmol), MgO (161.20 mg,

4.00 mmol) were used, to afford **3j** as a white solid (609.98 mg, 1.66 mmol, 94%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.79 – 7.78 (m, 2H), 7.26 – 7.21 (m, 6H), 7.07 – 7.06 (m, 4H), 6.98 – 6.96 (m, 2H), 4.31 (s, 4H), 3.89 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 162.9, 135.9, 132.6, 129.5, 128.7, 128.5, 127.9, 114.4, 55.8, 50.6.

M.P.: 63-65 °C

The spectroscopic data closely match the ones previously reported in the literature.¹⁰

N-Benzyl-benzenesulphonamide (4a)



The title compound was synthesized according to the general procedure B stated above. **2a** (176.61 mg, 1.00 mmol), **4** (117.87 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **4a** as a white solid (244.84 mg, 0.99 mmol, 99%).

The title compound was synthesized according to the general procedure C stated above. **1a** (218.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **4** (235.73 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used,

to afford 4a as a white solid (489.68 mg, 1.98 mmol, 99%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.85 – 7.83 (m, 2H), 7.56 – 7.54 (m, 1H), 7.53 – 7.51 (m, 2H), 7.50 – 7.47 (m, 3H), 7.46 – 7.45 (m, 2H), 5.23 – 5.21 (t, *J* = 6.3 Hz, 1H), 4.11 – 4.10 (d, *J* = 6.3 Hz, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 140.0, 136.4, 132.7, 129.2, 128.7, 127.9, 127.8, 127.1, 47.3.

M.P.: 85-86 °C

The spectroscopic data closely match the ones previously reported in the literature.¹¹

N-benzyl-4-(methyl)benzenesulphonamide (**4b**)



The title compound was synthesized according to the general procedure B stated above. **2b** (190.64 mg, 1.00 mmol), **4** (117.87 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **4b** as a white solid (240.43 mg, 0.92 mmol, 92%).

The title compound was synthesized according to the general procedure C stated above. **1b** (246.39 mg, 1.00 mmol), NaOCl $*5H_2O$ (987.12 mg, 6.00 mmol), NaHSO₄

(12.01 mg, 0.10 mmol), **4** (235.73 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **4b** as a white solid (480.87 mg, 1.84 mmol, 92%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.79 – 7.77 (m, 2H), 7.34 – 7.32 (m, 3H), 7.30 – 7.29 (m, 2H), 7.28 – 7.27 (m, 2H), 4.63 – 4.61 (s, 1H), 4.15 – 4.14 (d, *J* = 5.9 Hz, 2H), 2.46 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 143.7, 137.0, 136.4, 129.9, 128.8, 128.1, 128.0, 127.4, 47.5, 21.7.

M.P.: 112 – 113 °C

The spectroscopic data closely match the ones previously reported in the literature.¹¹

N-Benzyl-4-(*tert*-buthyl)benzenesulphonamide (**4c**)



The title compound was synthesized according to the general procedure B stated above. **2c** (232.72 mg, 1.00 mmol), **4** (117.87 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **4c** as a white solid (276.11 mg, 0.91 mmol, 91%).

The title compound was synthesized according to the general procedure C stated above. **1c** (330.55 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **4** (235.73 mg, 2.2 mmol), MgO (161.20 mg, t_{12} = 1.01 mg) is the state of t

4.00 mmol) were used, to afford **4c** as a white solid (552.22 mg, 1.82 mmol, 91%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.82 – 7.79 (m, 2H), 7.54 – 7.52 (m, 2H), 7.30 – 7.29 (m, 3H), 7.28 – 7.26 (m, 2H), 4.63 (s, 1H), 4.18 – 4.17 (d, *J* = 4.8 Hz, 2H), 1.37 (s, 9H).

¹³C NMR (151 MHz, CDCl₃) δ 156.7, 137.0, 136.4, 128.8, 128.0, 127.9, 127.1, 126.3, 47.5, 35.3, 31.2.

M.P.: 110 °C

The spectroscopic data closely match the ones previously reported in the literature.¹²

N-benzyl-4-(nitro)benzenesulphonamide (**4d**)



The title compound was synthesized according to the general procedure B stated above. **2d** (221.61 mg, 1.00 mmol), **4** (117.87 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **4d** as a brown solid (289.39 mg, 0.99 mmol, 99%).

The title compound was synthesized according to the general procedure C stated above. **1d** (308.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol),

NaHSO₄ (12.01 mg, 0.10 mmol), **4** (235.73 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **4d** as a brown solid (578.77 mg, 1.98 mmol, 99%).

¹**H** NMR (600 MHz, CDCl₃) δ 8.31 – 8.30 (m, 2H), 8.00 – 7.99 (m, 2H), 7.27 – 7.26 (m, 3H), 7.18 – 7.16 (m, 2H), 4.97 (t, *J* = 6.0 Hz, 1H), 4.24 – 4.23 (d, *J* = 6.0 Hz, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 150.1, 146.2, 135.6, 129.0, 128.5, 128.4, 128.0, 124.5, 47.6.

M.P.: 124-126 °C.

The spectroscopic data closely match the ones previously reported in the literature.¹³

N-Benzyl-2-nitrobenzenesulfonamide (4f)

The title compound was synthesized according to the general procedure B stated above. **2f** (221.61 mg, 1.00 mmol), **4** (117.87 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **4d** as a brown solid (222.16 mg, 0.76 mmol, 76%).

The title compound was synthesized according to the general procedure C stated above. **1d** (308.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10

mmol), **4** (235.73 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **4d** as a brown solid (444.31 mg, 1.52 mmol, 76%).

¹**H** NMR (600 MHz, CDCl₃) δ 8.05 - 8.03 (dd, *J* = 7.7, 1.6 Hz, 1 H), 7.86 - 7.84 (dd, J = 7.7, 1.6 Hz, 1 H), 7.68 - 7.65 (dd, J = 7.7, 1.6 Hz, 2 H), 7.25 - 7.18 (m, 5 H), 5.72 (t, J = 6.0 Hz, 1 H), 4.32 (d, J = 6.0 Hz, 2 H).

¹³C NMR (151 MHz, CDCl₃) δ 147.6, 135.9, 134.1, 133.6, 133.4, 129.8, 128.9, 127.7, 127.6, 125.2, 47.7.

M.P.: 43-44 °C.

N-Benzyl-4-(fluoro)benzenesulphonamide (4g)



The title compound was synthesized according to the general procedure B stated above. **2g** (194.60 mg, 1.00 mmol), **4** (117.87 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **4g** as a white solid (257.34 mg, 0.97 mmol, 97%).

The title compound was synthesized according to the general procedure C stated above. **1g** (254.31 mg, 1.00 mmol), NaOCl $*5H_2O$ (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **4** (235.73 mg, 2.2 mmol), MgO (161.20 mg, 4.00

mmol) were used, to afford **4g** as a white solid (514.68 mg, 1.94 mmol, 97%).

¹**H** NMR (600 MHz, CDCl₃) δ 7.88 – 7.86 (m, 2H), 7.28 – 7.26 (m, 3H), 7.19 – 7.16 (m, 4H), 4.74 – 4.72 (t, J = 6.1 Hz, 1H), 4.17 – 4.16 (d, J = 6.1 Hz, 2H).

¹³**C** NMR (151 MHz, CDCl₃) δ 166.3 (d, $J_{C-F} = 256.7$ Hz), 136.1, 130.0, 129.9 (d, $J_{C-F} = 15.1$ Hz), 128.1 (d, $J_{C-F} = 30.2$ Hz), 127.9, 116.5, 116.4, 47.5.

M.P.: 99 °C

The spectroscopic data closely match the ones previously reported in the literature.^{11,12}

N-Benzyl-4-(chloro)benzenesulphonamide (4h)



The title compound was synthesized according to the general procedure B stated above. **2h** (194.60 mg, 1.00 mmol), **4** (117.87 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **4h** as a white solid (267.66 mg, 0.95 mmol, 95%).

The title compound was synthesized according to the general procedure C stated above. **1h** (254.31 mg, 1.00 mmol), NaOCl $*5H_2O$ (987.12 mg, 6.00 mmol),

NaHSO₄ (12.01 mg, 0.10 mmol), **4** (235.73 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **4h** as a white solid (535.33 mg, 1.90 mmol, 95%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.79 – 7.77 (m, 2H), 7.47 – 7.46 (m, 2H), 7.28 – 7.26 (m, 3H), 7.19 – 7.17 (m, 2H), 4.76 (m, 1H), 4.16 – 4.15 (d, *J* = 6.2 Hz, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 139.2, 138.6, 135.9, 129.4, 128.8, 128.6, 128.1, 127.9, 47.3.

M.P.: 108-109 °C

The spectroscopic data closely match the ones previously reported in the literature.¹¹

N-Benzyl-4-(trifluoromethyl)benzenesulphonamide (4i)



The title compound was synthesized according to the general procedure B stated above. **2i** (244.61 mg, 1.00 mmol), **4** (117.87 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **4i** as a white solid (302.70 mg, 0.96 mmol, 96%).

The title compound was synthesized according to the general procedure C stated above. **1i** (354.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol),

NaHSO₄ (12.01 mg, 0.10 mmol), **4** (235.73 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **4i** as a white solid (605.40 mg, 1.92 mmol, 96%).

¹**H** NMR (600 MHz, CDCl₃) δ 7.97 – 7.95 (m, 2H), 7.76 – 7.74 (m, 2H), 7.29 – 7.25 (m, 3H), 7.18 – 7.16 (m, 2H), 4.79 – 4.77 (t, *J* = 6.0 Hz, 1H), 4.21 – 4.20 (d, *J* = 6.0 Hz, 2H).

¹³**C** NMR (151 MHz, CDCl₃) δ 143.9, 135.8, 134.6 (q, $J_{C-F} = 35.0$ Hz), 128.9, 128.4, 128.0, 127.7, 126.3 (q, $J_{C-F} = 135.9$ Hz), 124.3, 47.6.

M.P.: 121 - 122 °C

The spectroscopic data closely match the ones previously reported in the literature.^{8,11,12}

N-Benzyl-4-(methoxy)benzenesulphonamide (4j)



The title compound was synthesized according to the general procedure B stated above. **2j** (206.64 mg, 1.00 mmol), **4** (117.87 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **4j** as a white solid (246.83 mg, 0.89 mmol, 89%).

The title compound was synthesized according to the general procedure C stated above. **1j** (278.38 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol),

NaHSO₄ (12.01 mg, 0.10 mmol), **4** (235.73 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **4j** as a white solid (605.40 mg, 1.78 mmol, 89%).

¹**H** NMR (600 MHz, CDCl₃) δ 7.82 – 7.81 (m, 2H), 7.30 – 7.21 (m, 3H), 7.20 – 7.19 (m, 2H), 6.99 – 6.97 (m, 2H), 4.57 – 4.56 (t, *J* = 6.3 Hz, 1H), 4.13 – 4.12 (d, *J* = 6.3 Hz, 2H), 3.88 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 163.1, 136.4, 131.6, 129.5, 128.9, 128.1, 128.0, 114.5, 55.9, 47.4.

M.P.: 108-109 °C.

The spectroscopic data closely match the ones previously reported in the literature.¹³

N-Benzylpropane-2-sulfonamide (41)



The title compound was synthesized according to the general procedure B stated above. **2l** (142.60 mg, 1.00 mmol), **4** (117.87 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **4l** as a white solid (196.24 mg, 0.92 mmol, 92%).

The title compound was synthesized according to the general procedure C stated above. **11** (150.30 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol)

mmol), **4** (235.73 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **4l** as a white solid (392.47 mg, 1.84 mmol, 92%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.27 – 7.19 (m, 5H), 4.82 (s, 1H), 4.21 (s, 2H), 3.01 – 2.94 (p, *J* = 6.8 Hz, 1H), 1.25 – 1.24 (d, *J* = 6.8 Hz, 6H).

¹³C NMR (151 MHz, CDCl₃) δ 137.5, 128.8, 128.0, 127.9, 53.9, 47.5, 16.6.

M.P.: 98-100 °C.

The spectroscopic data closely match the ones previously reported in the literature.¹³

N-Benzyl-2,4,6-trimethylbenzenesulfonamide (**4m**)



The title compound was synthesized according to the general procedure B stated above. **2m** (218.70 mg, 1.00 mmol), **4** (117.87 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **4m** as a white solid (252.01 mg, 0.87 mmol, 87%).

The title compound was synthesized according to the general procedure C stated above. **1m** (302.49 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01

mg, 0.10 mmol), **4** (235.73 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **4m** as a white solid (504.02 mg, 1.74 mmol, 87%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.28 – 7.24 (m, 3H), 7.19 – 7.18 (m, 2H), 6.97 (s, 2H), 4.84 (t, *J* = 6.4 Hz, 1H), 4.08 (d, *J* = 6.4 Hz, 2H), 2.65 (s, 6H), 2.33 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 142.4, 139.3, 136.52, 132.1, 128.7, 128.0, 127.9, 46.9, 23.0, 21.0.

M.P.: 98-100 °C.

N-Benzylpyridine-2-sulfonamide (**4n**)



The title compound was synthesized according to the general procedure B stated above. **2n** (177.60 mg, 1.00 mmol), **4** (117.87 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **4n** as a white solid (252.01 mg, 0.87 mmol, 61%).

The title compound was synthesized according to the general procedure C stated above. **1m** (302.49 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **4** (235.73 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **4m** as a white solid (504.02 mg, 1.74 mmol, 61%).

¹**H** NMR (600 MHz, CDCl₃) δ 8.67 – 8.66 (m, 1H), 7.99 – 7.98 (d, *J* = 7.8 Hz, 1H), 7.90 – 7.88 (t, *J* = 7.8 Hz, 1H), 7.49 – 7.47 (m, 1H), 7.27 (s, 2H), 7.26 – 7.24 (m, 3H), 5.21 (bs, 1H), 4.27 (s, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 157.7, 150.2, 138.1, 136.4, 128.8, 128.1, 127.5, 126.8, 122.4, 48.0.

M.P.: 99 °C.

N-Phenylethyl-benzenesulphonamide (**5**a)



The title compound was synthesized according to the general procedure B stated above. **2a** (176.61 mg, 1.00 mmol), **5** (133.30 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **5a** as a white solid (237.82 mg, 0.91 mmol, 91%).

The title compound was synthesized according to the general procedure C stated above. **1a** (218.33 mg, 1.00 mmol), NaOCl $*5H_2O$ (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **5** (266.60 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **5a** as a white solid (475.64 mg, 1.82 mmol, 91%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.82 – 7.80 (m, 2H), 7.59 – 7.56 (m, 1H), 7.52 – 7.49 (m, 2H), 7.29 – 7.22 (m, 2H), 7.08 – 7.07 (m, 3H), 4.33 (s, 1H), 3.26 – 3.23 (q, *J* = 6.7 Hz, 2H), 2.78 – 2.76 (t, *J* = 6.7 Hz, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 140.1, 137.7, 132.8, 129.3, 128.9, 128.8, 127.2, 127.0, 44.3, 36.0.

M.P.: 63-66 °C.

The spectroscopic data closely match the ones previously reported in the literature.¹⁴

N-Phenylethyl-4-(methyl)benzenesulphonamide (**5**b)



The title compound was synthesized according to the general procedure B stated above. **2b** (190.64 mg, 1.00 mmol), **5** (133.30 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **5b** as a white solid (247.83 mg, 0.90 mmol, 90%).

The title compound was synthesized according to the general procedure C stated above. **1b** (246.39 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **5** (266.60 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **5b** as a white solid (495.67 mg, 1.80 mmol, 90%).

¹**H** NMR (600 MHz, CDCl₃) δ 7.70 – 7.69 (d, *J* = 7.9 Hz, 2H), 7.27 – 7.25 (d, *J* = 7.9 Hz, 4H), 7.23 – 7.18 (t, *J* = 7.3 Hz, 1H), 7.07 – 7.06 (d, *J* = 7.3 Hz, 2H), 4.88 (d, *J* = 6.8 Hz, 1H), 3.19 – 3.18 (q, *J* = 6.8 Hz, 2H), 2.75 – 2.74 (t, *J* = 6.8 Hz, 2H), 2.40 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 143.4, 137.9, 137.0, 129.8, 128.8, 128.7, 127.1, 126.7, 44.3, 35.9, 21.5.

M.P.: 63-66 °C.

The spectroscopic data closely match the ones previously reported in the literature.¹⁴

N-Phenylethyl-4-(tert-buthyl)benzenesulphonamide (5c)



The title compound was synthesized according to the general procedure B stated above. **2c** (232.72 mg, 1.00 mmol), **5** (133.30 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **5c** as a white solid (279.36 mg, 0.88 mmol, 88%).

The title compound was synthesized according to the general procedure C stated above. **1c** (330.55 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **5** (266.60 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **5c** as a white solid (558.71 mg, 1.76 mmol, 88%).

¹**H** NMR (600 MHz, CDCl₃) δ 7.74 – 7.72 (d, *J* = 8.2 Hz, 2H), 7.49 – 7.48 (d, *J* = 8.2 Hz, 2H), 7.26 – 7.25 (d, *J* = 7.3 Hz, 2H), 7.24 – 7.20 (t, *J* = 7.3 Hz, 1H), 7.08 – 7.07 (m, 1H), 4.72 – 4.71 (t, *J* = 6.2 Hz, 2H), 3.23 – 3.20 (q, *J* = 6.8 Hz, 2H), 2.78 – 2.76 (t, *J* = 6.8 Hz, 2H), 1.34 (s, 9H).

¹³C NMR (151 MHz, CDCl₃) δ 156.5, 137.9, 136.9, 128.8, 128.7, 127.0, 126.8, 126.1, 44.3, 35.9, 35.2, 31.2.

M.P.: 112 °C.

The spectroscopic data closely match the ones previously reported in the literature.¹⁵

N-Phenylethyl-4-(nitro)benzenesulphonamide (**5d**)



The title compound was synthesized according to the general procedure B stated above. **2d** (221.61 mg, 1.00 mmol), **5** (133.30 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **5d** as a brown solid (303.28 mg, 0.99 mmol, 99%).

The title compound was synthesized according to the general procedure C stated above. **1d** (308.33 mg, 1.00 mmol), NaOCl $*5H_2O$ (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **5** (266.60 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **5d** as a brown solid (606.55 mg, 1.76 mmol, 99%).

¹**H** NMR (600 MHz, CDCl₃) δ 8.28 – 8.26 (m, 2H), 7.95 – 7.93 (m, 2H), 7.25 – 7.24 (m, 3H), 7.23 – 7.18 (m, 2H), 5.14 – 5.12 (t, *J* = 6.0 Hz, 1H), 3.30 – 3.27 (q, *J* = 6.7 Hz, 2H), 2.80 – 2.78 (t, *J* = 6.9 Hz, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 150.0, 145.8, 137.4, 128.8, 128.7, 128.3, 127.0, 124.4, 44.4, 35.9.

M.P.: 95°C.

The spectroscopic data closely match the ones previously reported in the literature.¹⁶

N-Phenylethyl-4-(fluoro)benzenesulphonamide (5g)



The title compound was synthesized according to the general procedure B stated above. **2g** (232.72 mg, 1.00 mmol), **5** (133.30 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **5g** as a white solid (273.74 mg, 0.98 mmol, 98%).

The title compound was synthesized according to the general procedure C stated above. **1g** (330.55 mg, 1.00 mmol), NaOCl^{\cdot} 5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **5** (266.60 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **5c** as a white solid (547.49 mg, 1.96 mmol, 98%).

¹**H** NMR (600 MHz, CDCl₃) δ 7.82 – 7.80 (m, 2H), 7.27 – 7.26 (m, 3H), 7.16 – 7.13 (t, *J* = 8.5 Hz, 2H), 7.09 – 7.07 (m, 2H), 4.80 (s, 1H), 3.23 – 3.21 (t, *J* = 7.0 Hz, 2H), 2.78 – 2.76 (t, *J* = 7.0 Hz, 2H).

¹³**C** NMR (151 MHz, CDCl₃) δ 165.1 (d, $J_{C-F} = 135.9$ Hz), 137.7, 136.1, 129.8 (d, $J_{C-F} = 271.8$ Hz), 128.9, 128.8, 126.9, 116.5 (d, $J_{C-F} = 15.1$ Hz), 44.3, 35.9.

M.P.: 81-83 °C.

The spectroscopic data closely match the ones previously reported in the literature.¹⁴

N-Phenylethyl-4-(chloro)benzenesulphonamide (**5h**)



The title compound was synthesized according to the general procedure B stated above. **2h** (211.06 mg, 1.00 mmol), **5** (133.30 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **5h** as a white solid (275.10 mg, 0.93 mmol, 93%).

The title compound was synthesized according to the general procedure C stated above. **1h** (287.22 mg, 1.00 mmol), NaOCl $*5H_2O$ (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **5** (266.60 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **5h** as a white solid (550.20 mg, 1.86 mmol, 93%).

¹**H** NMR (600 MHz, CDCl₃) δ 7.72 – 7.71 (m, 2H), 7.42 – 7.40 (m, 2H), 7.24 – 7.20 (m, 2H), 7.19 – 7.18 (m, 1H), 7.07 – 7.05 (m, 2H), 5.14 – 5.13 (t, *J* = 6.3 Hz, 1H), 3.21 – 3.17 (q, *J* = 6.7 Hz, 2H), 2.76 – 2.74 (t, *J* = 6.9 Hz, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 139.0, 138.4, 137.9, 129.4, 128.7, 128.5, 126.8, 44.4, 44.3 35.8.

M.P.: 101 °C

CI

The spectroscopic data closely match the ones previously reported in the literature.¹⁴

N-Phenylethyl-4-(trifluoromethyl)benzenesulphonamide (5i)



The title compound was synthesized according to the general procedure B stated above. **2h** (244.61 mg, 1.00 mmol), **5** (133.30 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **5h** as a white solid (312.87 mg, 0.95 mmol, 95%).

The title compound was synthesized according to the general procedure C stated above. **1h** (354.33 mg, 1.00 mmol), NaOCl^{\cdot} 5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **5** (266.60 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **5h** as a white solid (625.75 mg, 1.90 mmol, 95%).

¹**H** NMR (600 MHz, CDCl₃) δ 7.91 – 7.89 (m, 2H), 7.74 – 7.73 (d, *J* = 8.3 Hz, 2H), 7.27 – 7.21 (m, 2H), 7.08 – 7.06 (m, 3H), 4.70 – 4.69 (t, *J* = 5.9 Hz, 1H), 3.29 – 3.25 (q, *J* = 6.5 Hz, 2H), 2.79 – 2.78 (t, *J* = 6.9 Hz, 2H).

¹³**C NMR** (151 MHz, CDCl₃) δ 143.6, 137.3, 134.3 (q, $J_{C-F} = 30.2$ Hz), 128.8 (d, $J_{C-F} = 15.1$ Hz), 127.5, 126.4, 126.3, 126.2, 123.3 (q, $J_{C-F} = 135.9$ Hz), 44.3, 35.8.

M.P.: 93-94 °C

The spectroscopic data closely match the ones previously reported in the literature.¹⁷

N-Phenylethyl-4-(methoxy)benzenesulphonamide (5j)



The title compound was synthesized according to the general procedure B stated above. **2j** (206.64 mg, 1.00 mmol), **5** (133.30 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **5j** as a white solid (262.23 mg, 0.90 mmol, 90%).

The title compound was synthesized according to the general procedure C stated above. **1j** (278.38 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **5** (266.60 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **5j** as a white solid (524.47 mg, 1.80 mmol, 90%).

¹**H** NMR (600 MHz, CDCl₃) δ 7.76 – 7.73 (m, 2H), 7.26 – 7.25 (m, 2H), 7.24 – 7.18 (m, 1H), 7.08 – 7.07 (m, 2H), 6.94 – 6.93 (m, 2H), 4.79 – 4.77 (t, *J* = 6.2 Hz, 1H), 3.84 (s, 3H), 3.19 – 3.16 (t, *J* = 7.1 Hz, 2H), 2.76 – 2.74 (t, *J* = 7.1 Hz, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 162.9, 137.9, 131.3, 129.2, 128.8, 128.7, 126.7, 114.3, 55.7, 44.3, 35.8.

M.P.: 52-53 °C

The spectroscopic data closely match the ones previously reported in the literature.¹⁸

N-(Phenyl)benzenesulfonamide (6a)



The title compound was synthesized according to the general procedure B stated above. **2a** (176.61 mg, 1.00 mmol), **6** (102.44 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **6a** as a white solid (198.30 mg, 0.86 mmol, 86%).

The title compound was synthesized according to the general procedure C stated above. **1a** (218.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **6** (204.89 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **6a** as a

white solid (396.59 mg, 1.72 mmol, 86%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.74 – 7.72 (m, 2H), 7.44 – 7.43 (m, 2H), 7.42 – 7.40 (m, 2H), 7.33 – 7.30 (m, 2H), 7.14 – 7.10 (m, 2H), 7.03 – 6.98 (m, 1H).

¹³C NMR (151 MHz, CDCl₃) δ 139.0, 136.6, 133.1, 129.3, 129.1, 127.3, 125.4, 121.7.

M.P.: 110 °C

The spectroscopic data closely match the ones previously reported in the literature.¹⁹

N-Phenyl-4-(methyl)benzenesulfonamide (6b)



The title compound was synthesized according to the general procedure B stated above. **2b** (190.64 mg, 1.00 mmol), **6** (102.44 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **6b** as a white solid (205.27 mg, 0.83 mmol, 83%).

The title compound was synthesized according to the general procedure C stated above. **1b** (246.39 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **6** (204.89 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford (410.54 mg, 1.66 mg, 1.66 mg)

6b as a white solid (410.54 mg, 1.66 mmol, 83%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.71 – 7.70 (d, *J* = 7.9 Hz, 2H), 7.55 (t, *J* = 8.1 Hz, 1H), 7.22 – 7.19 (d, *J* = 7.9 Hz, 4H), 7.12 – 7.06 (m, 3H), 2.35 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 143.9, 136.8, 136.1, 129.7, 129.3, 127.4, 125.2, 121.4, 21.6.

M.P.: 103 °C

The spectroscopic data closely match the ones previously reported in the literature.²⁰

N-Phenyl-4-(*tert*-buthyl)benzenesulfonamide (6c)



The title compound was synthesized according to the general procedure B stated above. **2c** (190.64 mg, 1.00 mmol), **6** (102.44 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **6c** as a white solid (231.51 mg, 0.80 mmol, 80%).

The title compound was synthesized according to the general procedure C stated above. **1c** (246.39 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **6** (204.89 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **6c** as a white solid (460.02 mg, 1.60 mmol, 80%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.77 – 7.76 (m, 2H), 7.47 – 7.42 (m, 3H), 7.24 – 7.21 (m, 2H), 7.14 – 7.07 (m, 2H), 1.29 (s, 9H).

¹³C NMR (151 MHz, CDCl₃) δ 156.9, 136.9, 136.2, 129.4, 127.2, 126.2, 125.1, 121.3, 35.2, 31.1.

M.P.: 118-119 °C

The spectroscopic data closely match the ones previously reported in the literature.²¹

N-Phenyl-4-(nitro)benzenesulfonamide (**6d**)



The title compound was synthesized according to the general procedure B stated above. **2d** (221.61 mg, 1.00 mmol), **6** (102.44 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **6d** as a brown solid (256.02 mg, 0.92 mmol, 92%).

The title compound was synthesized according to the general procedure C stated above. **1d** (308.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **6** (204.89 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used,

to afford **6d** as a brown solid (512.04 mg, 1.84 mmol, 92%).

¹**H NMR** (600 MHz, CDCl₃) δ 8.29 – 8.26 (m, 2H), 7.94 – 7.92 (m, 2H), 7.29 – 7.26 (m, 2H), 7.20 – 7.18 (m, 1H), 7.09 – 7.07 (m, 2H), 6.80 (s, 1H).

¹³C NMR (151 MHz, CDCl₃) δ 150.4, 144.8, 135.4, 129.8, 128.7, 126.7, 124.4, 122.6.

M.P.: 135-136 °C

The spectroscopic data closely match the ones previously reported in the literature.²²

N-Phenyl 4-(fluoro)benzenesulfonamide (6g)



The title compound was synthesized according to the general procedure B stated above. **2g** (194.60 mg, 1.00 mmol), **6** (102.44 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **6g** as a white solid (226.15 mg, 0.90 mmol, 90%).

The title compound was synthesized according to the general procedure C stated above. **1c** (254.31 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **6** (204.89 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford

6g as a white solid (452.30 mg, 1.80 mmol, 90%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.81 – 7.78 (m, 2H), 7.26 – 7.23 (m, 2H), 7.14 – 7.07 (m, 5H), 7.03 (s, 1H)

¹³**C** NMR (151 MHz, CDCl₃) δ 165.1 (d, $J_{C-F} = 252.5$ Hz), 136.3, 135.0 (d, $J_{C-F} = 328.9$ Hz), 130.3, 129.9, 125.6, 121.8, 116.2 (d, $J_{C-F} = 22.9$ Hz).

M.P.: 109-111 °C

The spectroscopic data closely match the ones previously reported in the literature.²³

N-Phenyl-4-(chloro)benzenesulfonamide (6h)



The title compound was synthesized according to the general procedure B stated above. **2h** (211.06 mg, 1.00 mmol), **6** (102.44 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **6h** as a white solid (232.93 mg, 0.87 mmol, 87%).

The title compound was synthesized according to the general procedure C stated above. **1h** (287.22 mg, 1.00 mmol), NaOC1*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **6** (204.89 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to

afford **6h** as a white solid (465.85 mg, 1.74 mmol, 87%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.72 – 7.71 (m, 2H), 7.40 – 7.38 (m, 2H), 7.26 – 7.23 (m, 2H), 7.14 (m, 1H), 7.13 – 7.08 (m, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 139.7, 137.5, 136.2, 129.6, 129.5, 128.8, 125.9, 122.0.

M.P.: 103-105 °C

The spectroscopic data closely match the ones previously reported in the literature.²³

N-Phenyl-4-(trifluoromethyl)benzenesulfonamide (6i)



The title compound was synthesized according to the general procedure B stated above. **2i** (211.06 mg, 1.00 mmol), **6** (102.44 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **6i** as a white solid (268.14 mg, 0.89 mmol, 89%).

The title compound was synthesized according to the general procedure C stated above. **1i** (287.22 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **6** (204.89 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used,

to afford **6i** as a white solid (536.28 mg, 1.78 mmol, 89%).

¹**H** NMR (600 MHz, CDCl₃) δ 7.90 – 7.89 (d, *J* = 8.2 Hz, 2H), 7.71 – 7.70 (d, *J* = 8.2 Hz, 2H), 7.29 – 7.25 (m, 2H), 7.18 – 7.15 (m, 1H), 7.10 – 7.07 (m, 2H), 6.95 (s, 1H).

¹³**C NMR** (151 MHz, CDCl₃) δ 142.7, 135.8, 133.5 (q, J_{C-F} = 32.9 Hz), 127.8, 126.3 (q, J_{C-F} = 3.8 Hz), 124.2, 122.4, 122.3 (d, J_{C-F} = 272.1 Hz), 121.5.

M.P.: 121-123 °C

The spectroscopic data closely match the ones previously reported in the literature.²³

N-Phenyl-4-(methoxy)benzenesulfonamide (6j)



The title compound was synthesized according to the general procedure B stated above. **2j** (206.64 mg, 1.00 mmol), **6** (102.44 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **6j** as a white solid (208.02 mg, 0.79 mmol, 79%).

The title compound was synthesized according to the general procedure C stated above. **1j** (278.38 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **6** (204.89 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were

used, to afford 6j as a white solid (416.03 mg, 1.58 mmol, 79%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.73 – 7.71 (m, 2H), 7.26 – 7.21 (m, 2H), 7.11 – 7.07 (m, 3H), 6.94 – 6.93 (m, 1H), 6.89 – 6.87 (m, 2H), 3.82 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 163.1, 136.8, 130.5, 129.5, 129.3, 125.1, 121.4, 114.2, 55.6.

M.P.: 108-109 °C

The spectroscopic data closely match the ones previously reported in the literature.²³

N-phenyl-methanesulfonamide (6k)



The title compound was synthesized according to the general procedure B stated above. 2k (114.54 mg, 1.00 mmol), **6** (102.44 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **6k** as a white solid (107.86 mg, 0.63 mmol, 63%).

The title compound was synthesized according to the general procedure C stated above. **1k** (94.19 mg, 1.00 mmol), NaOCl 5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **6**

(204.89 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **6k** as a white solid (215.73 mg, 1.26 mmol, 63%).

 1 H NMR (600 MHz, CDCl₃) δ 7.37 – 7.36 (m, 2H), 7.35 – 7.34 (m, 2H), 7.33 (s, 1H), 3.02 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 136.9, 129.9, 125.6, 120.9, 39.4.

M.P.: 98-100 °C

The spectroscopic data closely match the ones previously reported in the literature.²⁴

N-(4-Hydroxyphenyl)-benzenesulfonamide (**7a**)



The title compound was synthesized according to the general procedure B stated above. **2a** (176.61 mg, 1.00 mmol), **7** (120.04 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **7a** as a brown solid (221.86 mg, 0.89 mmol, 89%).

The title compound was synthesized according to the general procedure C stated above. **1a** (218.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **7** (240.09 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **7a** as a brown solid (443.72 mg, 1.78 mmol, 89%).

¹**H NMR** (600 MHz, DMSO) δ 9.71 (s, 1H), 9.30 (s, 1H), 7.66 – 7.64 (m, 2H), 7.60 – 7.57 (m, 1H), 7.53 – 7.50 (m, 2H), 6.83 – 6.81 (m, 2H), 6.60 – 6.58 (m, 2H).

¹³C NMR (151 MHz, DMSO) δ 155.6, 139.9, 133.5, 129.8, 129.1, 127.5, 125.2, 116.4.

M.P.: 155-156 °C

The spectroscopic data closely match the ones previously reported in the literature.²⁵

N-(4-Hydroxyphenyl)-4-(methyl)benzenesulfonamide (**7b**)



The title compound was synthesized according to the general procedure B stated above. **2b** (190.64 mg, 1.00 mmol), **7** (120.04 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **7b** as a brown solid (229.08 mg, 0.87 mmol, 87%).

The title compound was synthesized according to the general procedure C stated above. **1b** (218.33 mg, 1.00 mmol), NaOCl $*5H_2O$ (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **7** (240.09 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **7b** as a brown solid (458.16 mg, 1.74 mmol, 87%).

¹**H NMR** (600 MHz, DMSO) δ 9.65 (s, 1H), 7.56 – 7.54 (m, 2H), 7.30 – 7.28 (m, 2H), 6.87 – 6.85 (m, 2H), 6.63 – 6.60 (m, 2H), 2.30 (s, 3H).

¹³C NMR (151 MHz, DMSO) δ 154.9, 142.9, 136.8, 129.5, 128.7, 126.8, 124.0, 115.6, 21.0.

M.P.: 148-150 °C

The spectroscopic data closely match the ones previously reported in the literature.²⁶

N-(4-Hydroxyphenyl)-4-(*tert*-buthyl)benzenesulfonamide (**7c**)



The title compound was synthesized according to the general procedure B stated above. **2c** (232.72 mg, 1.00 mmol), **7** (120.04 mg, 1.1 mmol), and MgO (161.20 mg, 4.00 mmol) were used to afford **7c** as a brown solid (253.47 mg, 0.83 mmol, 83%).

The title compound was synthesized according to the general procedure C stated above. **1c** (330.55 mg, 1.00 mmol), NaOCl[·] $5H_2O$ (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **7** (240.09 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **7c** as a brown solid (506.95 mg, 1.66 mmol, 83%).

¹**H NMR** (600 MHz, DMSO) δ 9.72 (s, 1H), 7.62 – 7.60 (m, 2H), 7.54 – 7.52 (m, 2H), 6.88 – 6.87 (m, 2H), 6.62 – 6.60 (m, 2H), 1.25 (s, 9H).

¹³C NMR (151 MHz, DMSO) δ 172.0, 155.5, 154.7, 137.0, 128.7, 126.6, 126.2, 115.6, 34.8, 30.8.

M.P.: 159-161 °C

The spectroscopic data closely match the ones previously reported in the literature.²⁶

N-(4-Hydroxyphenyl)-4-(nitro)benzenesulfonamide (**7d**)



The title compound was synthesized according to the general procedure B stated above. **2d** (221.61 mg, 1.00 mmol), **7** (120.04 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **7d** as a brown solid (276.62 mg, 0.94 mmol, 94%).

The title compound was synthesized according to the general procedure C stated above. **1d** (308.33 mg, 1.00 mmol), NaOCl^{\cdot} 5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **7** (240.09 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **7d** as a brown solid (553.25 mg, 1.88 mmol, 94%).

¹**H NMR** (600 MHz, DMSO) δ 10.04 (s, 1H), 9.38 (s, 1H), 8.36 – 8.34 (m, 2H), 7.89 – 7.87 (m, 2H), 6.84 – 6.82 (m, 2H), 6.63 – 6.60 (m, 2H).

¹³C NMR (151 MHz, DMSO) δ 155.6, 150.0, 145.2, 128.6, 127.7, 125.1, 124.7, 116.0.

M.P.: 186 °C

The spectroscopic data closely match the ones previously reported in the literature.²⁷

N-(4-Hydroxyphenyl)-4-(fluoro)benzenesulfonamide (**7g**)



The title compound was synthesized according to the general procedure B stated above. **2g** (194.60 mg, 1.00 mmol), **7** (120.04 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **7g** as a brown solid (245.89 mg, 0.92 mmol, 92%).

The title compound was synthesized according to the general procedure C stated above. **1g** (254.31 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **7** (240.09 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **7g** as a brown solid (491.78 mg, 1.84 mmol, 92%).

¹**H NMR** (600 MHz, DMSO) δ 9.72 (s, 1H), 9.33 (s, 1H), 7.70 – 7.68 (m, 2H), 7.38 – 7.35 (m, 2H), 6.83 – 6.81 (m, 2H), 6.62 – 6.59 (m, 2H).

¹³**C NMR** (151 MHz, DMSO) δ 164.0 (d, $J_{C-F} = 135.9$ Hz), 155.1, 135.9, 130.2 (d, $J_{C-F} = 30.2$ Hz), 128.7, 124.4, 116.7 (d, $J_{C-F} = 15.1$ Hz), 116.0.

M.P.: 171-172 °C

The spectroscopic data closely match the ones previously reported in the literature.²⁸

N-(4-Hydroxyphenyl)-4-(chloro)benzenesulfonamide (7h)



The title compound was synthesized according to the general procedure B stated above. **2h** (211.06 mg, 1.00 mmol), **7** (120.04 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **7h** as a brown solid (258.19 mg, 0.91 mmol, 91%).

The title compound was synthesized according to the general procedure C stated above. **1h** (287.22 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **7** (240.09 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **7h** as a brown solid (516.39 mg, 1.82 mmol, 91%).

¹**H NMR** (600 MHz, DMSO) δ 10.50 (s, 1H), 7.79 – 7.71 (m, 2H), 7.70 – 7.63 (m, 2H), 7.05 – 7.04 (m, 2H), 6.95 – 6.93 (m, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 135.1, 133.6, 129.9, 129.6, 129.5, 128.7, 123.4, 122.9.

M.P.: 178 °C

The spectroscopic data closely match the ones previously reported in the literature.²⁹

N-(4-Hydroxyphenyl)-4-(trifluoromethyl)benzenesulfonamide (7i)



The title compound was synthesized according to the general procedure B stated above. **2i** (244.61 mg, 1.00 mmol), **7** (120.04 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **7i** as a brown solid (298.24 mg, 0.94 mmol, 94%).

The title compound was synthesized according to the general procedure C stated above. **1i** (354.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **7** (240.09 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **7i** as a brown solid (596.49 mg, 1.88 mmol, 94%). ¹**H NMR** (600 MHz, DMSO) δ 9.96 (s, 1H), 9.38 (s, 1H), 7.93 – 7.92 (d, J = 8.3 Hz, 2H), 7.86 – 7.84 (d, J = 8.3 Hz, 2H), 6.85 – 6.83 (m, 2H), 6.63 – 6.61 (m, 2H).

¹³**C NMR** (151 MHz, DMSO) δ 155.3, 143.4, (q, $J_{C-F} = 30.2$ Hz), 132.0, 127.8, 127.7, 126.4, 126.3, 124.4 (q, $J_{C-F} = 135.9$ Hz), 115.7.

M.P.: 181-182 °C

N-(4-Hydroxyphenyl)-4-(methoxy)benzenesulfonamide (7j)



The title compound was synthesized according to the general procedure B stated above. **2j** (206.64 mg, 1.00 mmol), **7** (120.04 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **7j** as a brown solid (223.45 mg, 0.80 mmol, 80%).

The title compound was synthesized according to the general procedure C stated above. **1j** (278.38 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **7** (240.09 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **7j** as a brown solid (446.90 mg, 1.60 mmol, 80%).

¹**H NMR** (600 MHz, DMSO): δ 9.56 (s, 1H), 7.58 – 7.57 (m, 2H), 7.03 – 7.02 (m, 2H), 6.84 – 6.82 (m, 2H), 6.60 – 6.59 (m, 2H), 3.79 (s, 3H).

¹³C NMR (151 MHz, DMSO): δ 172.0, 162.2, 154.7, 131.2, 128.7, 123.9, 115.5, 114.1, 55.6.

M.P.: 185 °C

MeO

The spectroscopic data closely match the ones previously reported in the literature.²⁸

N-phenylsulfonylpiperidine (8a)



The title compound was synthesized according to the general procedure B stated above. **2a** (176.61 mg, 1.00 mmol), **8** (93.67 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **8a** as a white solid (220.80 mg, 0.98 mmol, 98%).

The title compound was synthesized according to the general procedure C stated above. **1a** (218.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **8** (187.34 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **7a** as a white solid (441.61 mg, 1.96 mmol, 98%).

¹**H** NMR (600 MHz, CDCl₃) δ 7.76 – 7.74 (m, 2H), 7.60 – 7.57 (t, *J* = 7.4 Hz, 1H), 7.53 – 7.51 (t, *J* = 7.5 Hz, 2H), 2.99 – 2.97 (t, *J* = 5.7 Hz, 4H), 1.65 – 1.61 (p, *J* = 5.7 Hz, 4H), 1.43 – 1.39 (m, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 136.5, 132.7, 129.0, 127.8, 47.1, 25.3, 23.6.

M.P.: 95-97 °C

The spectroscopic data closely match the ones previously reported in the literature.²⁷

N-(4-(Methyl)phenylsulfonyl) piperidine (8b)



The title compound was synthesized according to the general procedure B stated above. **2b** (190.64 mg, 1.00 mmol), **8** (93.67 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **8b** as a white solid (229.76 mg, 0.96 mmol, 96%). The title compound was synthesized according to the general procedure C stated above. **1b** (246.39 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **8** (187.34 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **8b** as a white solid (469.51 mg, 1.96 mmol, 96%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.64 – 7.63 (m, 2H), 7.32 – 7.31 (m, 2H), 2.98 – 2.96 (m, 4H), 2.43 (s, 3H), 1.65 – 1.62 (m, 4H), 1.42 – 1.40 (qd, *J* = 7.1, 4.7 Hz, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 143.4, 133.5, 129.7, 127.9, 47.1, 25.3, 23.7, 21.7.

M.P.: 97-99 °C

The spectroscopic data closely match the ones previously reported in the literature.^{6,20}

N-(4-(*tert*-Butyl)phenylsulfonyl) piperidine (8c)



The title compound was synthesized according to the general procedure B stated above. **2c** (232.72 mg, 1.00 mmol), **8** (93.67 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **8c** as a white solid (261.71 mg, 0.93 mmol, 93%).

The title compound was synthesized according to the general procedure C stated above. **1c** (330.55 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **8** (187.34 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol)

were used, to afford 8c as a white solid (523.43 mg, 1.86 mmol, 93%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.68 – 7.66 (m, 2H), 7.52 – 7.51 (m, 2H), 3.00 – 2.98 (m, 4H), 1.66 – 1.63 (m, 4H), 1.44 – 1.40 (m, 2H), 1.34 (s, 9H).

¹³C NMR (151 MHz, CDCl₃) δ 156.3, 133.5, 127.7, 126.0, 47.1, 35.3, 31.3, 25.4, 23.7.

M.P.: 129 – 130 °C

The spectroscopic data closely match the ones previously reported in the literature.³⁰

N -(4-(Nitro)phenylsulfonyl) piperidine (**8d**)



The title compound was synthesized according to the general procedure B stated above. **2d** (232.72 mg, 1.00 mmol), **8** (93.67 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **8d** as a pale-yellow solid. (261.71 mg, 0.99 mmol, 99%).

The title compound was synthesized according to the general procedure C stated above. **1d** (330.55 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **8** (187.34 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **8d** as a pale-yellow solid (523.43 mg, 1.98 mmol, 99%).

¹**H NMR** (600 MHz, CDCl₃) δ 8.38 – 8.37 (m, 2H), 7.95 – 7.93 (m, 2H), 3.07 – 3.05 (m, 4H), 1.68 – 1.64 (m, 4H), 1.48 – 1.44 (m, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 162.0, 142.9, 128.9, 124.4, 47.1, 25.3, 23.5.

M.P.: 168-170 °C

The spectroscopic data closely match the ones previously reported in the literature.^{27,30}

N -(4-(Fluoro)phenylsulfonyl) piperidine (8g)



The title compound was synthesized according to the general procedure B stated above. **2g** (194.60 mg, 1.00 mmol), **8** (93.67 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **8g** as a white solid. (218.97 mg, 0.90 mmol, 90%).

The title compound was synthesized according to the general procedure C stated above. **1g** (254.31 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01

mg, 0.10 mmol), **8** (187.34 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **8g** as a white solid (437.94 mg, 1.80 mmol, 90%).

¹**H** NMR (600 MHz, CDCl₃) δ 7.78 – 7.76 (m, 2H), 7.22 – 7.19 (m, 2H), 3.00 – 2.98 (m, 4H), 1.67 – 1.63 (p, J = 5.8 Hz, 4H), 1.45 – 1.41 (m, 2H).

¹³**C NMR** (151 MHz, CDCl₃) δ 165.3 (d, J_{C-F} = 135.9 Hz), 130.5, 130.4, 116.2 (d, J_{C-F} = 15.1 Hz), 47.1, 25.3, 23.6.

M.P.: 76–77 °C

The spectroscopic data closely match the ones previously reported in the literature.³¹

N -(4-(Chloro)phenylsulfonyl) piperidine (8h)



The title compound was synthesized according to the general procedure B stated above. **2h** (211.06 mg, 1.00 mmol), **8** (93.67 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **8h** as a white solid. (225.37 mg, 0.87 mmol, 87%).

The title compound was synthesized according to the general procedure C stated above. **1h** (287.22 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄

(12.01 mg, 0.10 mmol), **8** (187.34 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **8h** as a white solid (450.73 mg, 1.74 mmol, 87%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.71 – 7.68 (m, 2H), 7.51 – 7.49 (m, 2H), 3.00 – 2.98 (t, *J* = 5.5 Hz, 4H), 1.67 – 1.63 (q, *J* = 5.5 Hz, 4H), 1.46 – 1.42 (m, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 139.1, 135.0, 129.3, 129.1, 46.9, 25.2, 23.5.

M.P.: 92-93 °C

The spectroscopic data closely match the ones previously reported in the literature.³¹

N-(4-(Trifluoromethyl)phenylsulfonyl) piperidine (8i)



The title compound was synthesized according to the general procedure B stated above. **2i** (244.61 mg, 1.00 mmol), **8** (93.67 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **8i** as a white solid. (269.84 mg, 0.92 mmol, 92%).

The title compound was synthesized according to the general procedure C stated above. **1i** (354.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄

(12.01 mg, 0.10 mmol), **8** (187.34 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **8i** as a white solid (539.67 mg, 1.84 mmol, 92%).

¹**H** NMR (600 MHz, CDCl₃) δ 7.89 – 7.88 (d, *J* = 8.3 Hz, 2H), 7.81 – 7.79 (d, *J* = 8.3 Hz, 2H), 3.03 – 3.01 (t, *J* = 5.5 Hz, 4H), 1.68 – 1.64 (p, *J* = 5.8 Hz, 4H), 1.47 – 1.43 (m, 2H).

¹³**C NMR** (151 MHz, CDCl₃) δ 152.2, 140.3, 134.3 (q J_{C-F} = 32.9 Hz), 126.2 (q J_{C-F} = 15.1 Hz), 122.4, 46.9, 25.2, 23.4.

M.P.: 95–96 °C

The spectroscopic data closely match the ones previously reported in the literature.³⁰

N-(4-(Methoxy)phenylsulfonyl) piperidine (**8j**)



The title compound was synthesized according to the general procedure B stated above. **2j** (206.64 mg, 1.00 mmol), **8** (93.67 mg, 1.1 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **8j** as a white solid. (217.03 mg, 0.85 mmol, 85%).

The title compound was synthesized according to the general procedure C stated above. **1j** (278.38 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol),

NaHSO₄ (12.01 mg, 0.10 mmol), **8** (187.34 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **8j** as a white solid (434.06 mg, 1.7 mmol, 85%).

¹**H NMR** (600 MHz, CDCl₃) δ 7.64 – 7.62 (m, 2H), 6.95 – 6.94 (m, 2H), 3.82 (s, 3H), 2.91 – 2.89 (m, 4H), 1.60 – 1.56 (m, 4H), 1.37 – 1.34 (m, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 162.9, 129.7, 127.8, 114.1, 55.6, 46.9, 25.1, 23.5.

M.P.: 105-108 °C

The spectroscopic data closely match the ones previously reported in the literature.^{10,31}

N-(5-Methylisoxazol-3-yl)-4-(nitro)benzenesulfonamide (9d)



The title compound was synthesized according to the general procedure B stated above. **2d** (221.61 mg, 1.00 mmol), **9** (107.86 mg, 1.10 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **9d** as an orange solid. (212.45 mg, 0.75 mmol, 75%).

The title compound was synthesized according to the general procedure C stated above. **1d** (308.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **9** (215.72 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **9d** as an orange solid (424.89 mg, 1.50 mmol, 75%).

¹**H NMR** (600 MHz, DMSO) δ 8.44 – 8.43 (m, 2H), 8.13 – 8.11 (m, 2H), 2.31 (s, 3H).

¹³C NMR (151 MHz, DMSO) δ 171.2, 157.5, 150.6, 145.1, 128.9, 125.3, 96.0, 12.5.

M.P.: 194-195 °C

The spectroscopic data closely match the ones previously reported in the literature.³²

N-(3,4-dimethylisoxazol-5-yl)-4-(nitro)benzenesulfonamide (**10d**)



The title compound was synthesized according to the general procedure B stated above. **2d** (221.61 mg, 1.00 mmol), **10** (107.86 mg, 1.10 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **10d** as an orange solid. (196.21 mg, 0.66 mmol, 66%).

The title compound was synthesized according to the general procedure C stated above. **1d** (308.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **10** (215.72 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **10d** as an orange solid (392.42 mg, 1.32 mmol, 66%).

¹**H** NMR (600 MHz, CDCl₃) δ 8.33 – 8.32 (d, *J* = 8.7 Hz, 2H), 8.02 – 8.01 (d, *J* = 8.7 Hz, 2H), 4.15 (s, 1H), 2.19 (s, 3H), 1.93 (s, 3H).

¹³C NMR (151 MHz, DMSO) δ 164.6, 162.3, 154.7, 150.5, 145.8, 128.7, 124.6, 11.0, 6.8.

M.P.: 205-207 °C

The spectroscopic data closely match the ones previously reported in the literature.³³

N-Butyl-4-nitrobenzenesulfonamide (11d)



The title compound was synthesized according to the general procedure B stated above. **2d** (221.61 mg, 1.00 mmol), **11** (80.45 mg, 1.10 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **11d** as a white solid. (100.62 mg, 0.39

mmol, 39%).

The title compound was synthesized according to the general procedure C stated above. **1d** (308.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **11** (160.91 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **11d** as a white solid (201.24 mg, 0.78 mmol, 39%).

¹**H** NMR (600 MHz, CDCl₃) δ 8.36 (d, J = 8.3 Hz, 2H), 8.05 (d, J = 8.3 Hz, 2H), 4.55 (bs, 1H), 3.03 (q, J = 6.9 Hz, 2H), 1.47 (t, J = 7.5 Hz, 2H), 1.31 (p, J = 7.5 Hz, 2H), 0.89 – 0.85 (m, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 150.2, 146.2, 128.4, 124.5, 43.3, 31.8, 19.8, 13.6.

M.P.: 75 °C

Methyl ((4-nitrophenyl)sulfonyl)phenylalaninate (12d)



The title compound was synthesized according to the general procedure B stated above. **2d** (221.61 mg, 1.00 mmol), **12** (237.25 mg, 1.10 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **11d** as a white solid. (236.84 mg, 0.65 mmol,

The title compound was synthesized according to the general procedure C stated above. **1d** (308.33 mg, 1.00 mmol), NaOCl $5H_2O$ (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **12** (474.50 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **12d** as a white solid (473.68 mg, 1.30 mmol, 65%).

¹**H** NMR (600 MHz, CDCl₃) δ 8.28 – 8.22 (m, 2H), 7.90 – 7.84 (m, 2H), 7.35 – 7.31 (m, 1H), 7.26 (m, 2H), 7.10 (m, 2H), 5.59 (d, *J* = 9.3 Hz, 1H), 4.30 (q, *J* = 7.0 Hz, 1H), 3.66 (s, 3H), 3.19 – 2.98 (m, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 171.3, 150.1, 145.7, 128.9, 128.7, 128.3, 127.6, 125.2, 124.3, 57.3, 52.9, 39.3. M.P.: 153-155°C

Methyl ((4-nitrophenyl)sulfonyl)tyrosinate (13d)



The title compound was synthesized according to the general procedure B stated above. **2d** (221.61 mg, 1.00 mmol), **13** (254.85 mg, 1.10 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **13d** as a white solid. (167.36 mg, 0.44 mmol, 44%).

The title compound was synthesized according to the general procedure C stated above. **1d** (308.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **13** (509.70 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **13d** as a white solid (334.73 mg, 0.88 mmol, 44%).

¹**H NMR** (600 MHz, CDCl₃) δ 8.38 – 8.32 (m, 2H), 8.05 – 7.97 (m, 2H), 7.17 – 7.06 (m, 2H), 6.96 – 6.88 (m, 2H), 4.18 (bs, 1H), 3.74 (s, 3H), 3.15 – 2.99 (m, 2H), 2.96 – 2.79 (m, 2H).

¹³**C NMR** (151 MHz, CDCl₃) δ 174.9, 155.6, 134.9, 131.1, 130.8, 130.1, 129.9, 128.3, 128.2, 56.9, 55.4, 38.6.

M.P.: 169 °C

N-(2-Hydroxyethyl)-4-nitrobenzenesulfonamide (14d)

Q
S
HNThe title compound was synthesized according to the general procedure B stated above.2d (221.61 mg, 1.00 mmol), 14 (67.19 mg, 1.10 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford 14d as a white solid. (128.05 mg, 0.52 mmol, 52%).

The title compound was synthesized according to the general procedure C stated above. **1d** (308.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **14** (134.38 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **14d** as a white solid (256,09 mg, 1.04 mmol, 52%).

¹**H** NMR (600 MHz, CDCl₃) δ 8.37 (d, *J* = 8.3 Hz, 2H), 8.07 (d, *J* = 8.3 Hz, 2H), 5.11 (bs, 1H), 3.74 (t, *J* = 5.2 Hz, 2H), 3.19 (q, *J* = 5.2 Hz, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 156.6, 146.0, 128.5, 124.6, 61.3, 45.3.

M.P.: 124 °C

N-(2-mercaptoethyl)-4-nitrobenzenesulfonamide (15d)



The title compound was synthesized according to the general procedure B stated above. **2d** (221.61 mg, 1.00 mmol), **15** (84,87 mg, 1.10 mmol) and MgO (161.20 mg, 4.00 mmol) were used, to afford **15d** as a white solid. (170.50 mg, 0.65 mmol, 65%).

The title compound was synthesized according to the general procedure C stated above. **1d** (308.33 mg, 1.00 mmol), NaOCl*5H₂O (987.12 mg, 6.00 mmol), NaHSO₄ (12.01 mg, 0.10 mmol), **15** (169.73 mg, 2.2 mmol), MgO (161.20 mg, 4.00 mmol) were used, to afford **15d** as a white solid (341.00 mg, 1.30 mmol, 65%).

¹**H NMR** (600 MHz, CDCl₃) δ 8.44 – 8.32 (m, 2H), 8.13 – 8.04 (m, 2H), 3.34 (m, 2H), 3.03 (m, 1H), 2.83 – 2.73 (m, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 150.4, 145.8, 128.5, 124.7, 41.9, 38.3.

M.P.: 131-133 °C

6. Green Metrics Calculations

Calculation of Green Chemistry Metrics (this mechanochemical method)



It is worth pointing out that we considered in our E-Factor calculations the amount of solvent used for the recovery of the product. If this value is neglected (due to the reusability of the solvent after distillation under reduced pressure), the E-Factor of our procedure should be:

Environmental Factor = $\frac{Mass of tota}{Mass of tota}$			=						1510.00)	-=2.98
		Mass of desired	l product	349.69						2.90
Ecoscale calculate	or									
Reagents 🗵										
Link										
ide	entifier*	name	MF*	MW 218.3314	density 1.353	purity*	ml	g 0.218331	mmoles	equiv.
2 + -	Hypochlorite salts		H1006CINa	164.51857		100%	0	0.987111	6	6
3 + -	Sodium bisulfate		HNaO4S	120.05531	2.1	100%	0.005717	0.012006	0.1	0.1
Products 🗵			11111045	120.00001			10.005717	10.012000		
Troducts E	identifier*: name:		MF	*: M\	V: g:	mn	noles: g	theor: yi	ield:	
	Benzenes	ulfonyl chloride	C6	5H5CIO2S	76.6175 0.	353235 2	0	.176618	199.9994	
Conditions 🗵										
Reagents	Name Phenyl disulfide	mmoles 2.02	eq. Bp	Hazard	Price					
	Hypochlorite salts		6 182							
	Sodium bisulfate		0.1 3600							
Yield	99	0.2	0.1 5000		-0.5					
Price / availability	39				-0.5					
Safety					-5					
Technical setup			Selected items	7						
	Common set-up Instruments for controlled add Unconventional activation tech		Common set-up		0					
Temperature / time	Possible items	indee .	Selected items							
	Room temperature, < 1h Room temperature, < 24h Heating, < 1h		Room temperature	e, < 1h ⊸ ↓	0					
Workup and purification	Possible items None	*	Selected items Adding solvent							
	Cooling to room temperature Adding solvent	•	· taanig oolvent 2		0					
EcoScale					91.5					

Calculation of Green Chemistry Metrics (solution method)³⁴



http://ecoscale.cheminfo.org/calculator


Calculation of Green Chemistry Metrics (this mechanochemical method)

It is worth pointing out that we considered in our E-Factor calculations the amount of solvent used for the recovery of the product, and both the solvent and the acid mass for the purification washes. If the value for all of this is neglected (due to the reusability of the solvent after distillation under reduced pressure), the E-Factor of our procedure should be:

Linviron	mental Factor	Mass of desir	red product	-		244.	84			1.00
coscale calculat	or									
Reagents 🗵										
✓ Link	entifier [*]	name	MF [*]	мw	density	purity*	ml	q	mmoles	equiv
1 + -	Benzenesulfony	chloride	C6H5CIO2S	176.6175	1.384	100%	0.127614	0.176618	1	1
2 + -	Benzylamine		C7H9N	107.15516	0.98	100%	0.120277	0.117871	1.1	1.1
3 + -	Magnesium oxid	e	MgO	40.3044	3.58	100%	0.045033	0.161218	4	4
Products 🗵										
Reagents	Name Benzenesulfonyl chloride Benzylamine Magnesium oxide	mmole: 2.83 3.11 11.32	eq. Bp 1 251 1.1 182 4 3600	Hazard G	Þ					
	99]			-0.5					
Price / availability Safety					0					
Technical setup	Possible items Common set-up Instruments for controlled ad Unconventional activation te	dition of chemicals	Selected items Common set-up		0					
Temperature / time		Selected items Room temperature, < 24h		-1						
orkup and purification	None	*	Selected items Adding solvent		3					
	Cooling to room temperature Adding solvent	~	Liquid - liquid extract	ion or washing						



	0 S Ph ⊂CI + 2	Ph [^] NH ₂	Pyridine Conversion >> 99.9	→ ^O S N 9% Ph H Ph				
	Used: 194.27	of reagent	M.W. 79.10 Used: 4900 m (5 mL)	M.W. 247.31 Prod.: 239.89	15 m 546.90 mg (HCl) 5 i	rification L HCI (1 N)) + 14453.10 mg (H; mL brine CI + 5000 mg (H ₂ O)		
	Atom Econor	$my = \frac{Mass \text{ of } d}{Total M}$	esired useful proc ass of all reactant	$\frac{huct}{ts} \times 100 = \frac{1}{1}$	247.31 76.61 + 107.16 + 1	79.10 × 100 =	68.2%	
Envi	ironmental Facto	$r = \frac{Mass of to}{Mass of des}$	$\frac{\text{otal waste}}{\text{ired product}} = \frac{1}{2}$	17.66 + 107.16 + 4	900 + 546.90 + 14 239.89	4453.10 + 1785 +	$\frac{5000}{2} = 111.76$	6
R Ecoscale calculat	eaction Mass Ef	ficiency = ac	tual mass of desir mass of react	$\frac{\text{red product}}{\text{ants}} \times 10$	$0 = \frac{239}{194.27 + 21}$	$\frac{9.89}{4.32+4900} \times 1$	00 = 4.5 %	
Reagents 🗵			*					
Link	entifier [*]	name chloride	MF [*]	MW de	nsity purity [*]	ml	g mmoles 76618 1	equiv.
☑ Link id	entifier [*]					0.127614 0.1		equiv.
Link id 1	entifier*		C6H5CIO2S	176.6175 1.384	100%	0.127614 0.1	76618 1 1431 2	1
Link id 1 ± 2 ± ± 5 Products ©	entifier* Benzenesulfonyl Benzylamine Pyridine identifier*: name: N-(pheny		C6H5CIO2S C7H9N C5H5N	176.6175 1.384 107.15516 0.98	100% 100% 100%	0.127614 0.1 0.218684 0.2 5 4.8 moles: 9 theor:	76618 1 1431 2 9 61.8193862 yield:	2
Link 1 + - 2 + - 3 + -	entifier" Benzenesulfonyl Benzylamine Pyridine identifier": name: N-(pheny Name	chloride Imethyl)benzenesulfona mmole	C6H5CIO2S C7H9N C5H5N MF*: amide C133 as eq. Bp	176.6175 1.384 107.15516 0.98 79.1014 0.976 H13NO2S 247.31172 Hazard Price	100% 100% 100% 9: m	0.127614 0.1 0.218684 0.2 5 4.8 moles: 9 theor:	76618 1 1431 2 9 61.8193862 yield:	2
Conditions	entifier" Benzenesulfonyl Benzylamine Pyridine identifier": name: N-(pheny Name Benzenesulfonyl chloride	chloride Imethyl)benzenesulfona mmole 2.83	C6H5CIO2S C7H9N C5H5N mF#: amide C13 cs eq. Bp 1 251	176.6175 1.384 107.15516 0.98 79.1014 0.976 H13NO2S 247.31172 Hazard Price	100% 100% 100% 9: m	0.127614 0.1 0.218684 0.2 5 4.8 moles: 9 theor:	76618 1 1431 2 9 61.8193862 yield:	2
Conditions	entifier" Benzenesulfonyl Benzylamine Pyridine identifier": name: N-(pheny Name	chloride Imethyl)benzenesulfona mmole	C6H5CIO2S C7H9N C5H5N MF*: amide C133 as eq. Bp	176.6175 1.384 107.15516 0.98 79.1014 0.976 H13NO2S 247.31172 Hazard Price	100% 100% 100% 9: m	0.127614 0.1 0.218684 0.2 5 4.8 moles: 9 theor:	76618 1 1431 2 9 61.8193862 yield:	2
Conditions	entifier" Benzenesulfonyl Benzylamine Pyridine identifier": name: N-(pheny N-(pheny Benzenesulfonyl chloride Benzylamine Pyridine	chloride Imethyl)benzenesulfona 2.83 5.66	C6H5CIO2S C7H9N C5H5N MF*: amide C13i es eq. Bp 1 251 2 182	176.6175 1.384 107.15516 0.98 79.1014 0.976 H13NO2S 247.31172 Hazard Price	100% 100% 100% 9: m	0.127614 0.1 0.218684 0.2 5 4.8 moles: 9 theor:	76618 1 1431 2 9 61.8193862 yield:	2
Link I Link I Link I Link Conditions Conditions Reagents Yield Price / availability	entifier" Benzenesulfonyl Pyridine identifier": name: N-(pheny Rame Benzenesulfonyl chloride Benzylamine Pyridine 97	chloride Imethyl)benzenesulfona 2.83 5.66	C6H5CIO2S C7H9N C5H5N MF*: amide C13i es eq. Bp 1 251 2 182	176.6175 1.384 107.15516 0.98 79.1014 0.976 H13NO2S 247.31172 Hazard Price	0: 0.494623	0.127614 0.1 0.218684 0.2 5 4.8 moles: 9 theor:	76618 1 1431 2 9 61.8193862 yield:	2
Link I Link I Link I Link Conditions Conditions Reagents Yield Price / availability Safety	entifier" Benzenesulfonyl Benzylamine Pyridine identifier": name: N-(pheny Benzenesulfonyl chloride Benzylamine Pyridine 97	chloride Imethyl)benzenesulfona 2.83 5.66	C6H5CIO2S C7H9N C5H5N C5H5N C13I c13I c13I c13I c13I c13I c13I c13I c	176.6175 1.384 107.15516 0.98 79.1014 0.976 H13NO2S 247.31172 Hazard Price	0: 0.494623 2. 1.5	0.127614 0.1 0.218684 0.2 5 4.8 moles: 9 theor:	76618 1 1431 2 9 61.8193862 yield:	2
Link id Link id Link Conditions Reagents Yield Price / availability Safety Technical setup	entifier* Benzenesulfonyl Benzylamine Pyridine Pyridine Name Benzenesulfonyl chloride Benzylamine Pyridine 97 Possible items Common set-up Instruments for controlled add Unconventional activation tec	chloride Imethyl)benzenesulfona 2.83 5.66 175	C6H5CIO2S C7H9N C5H5N amide C133 c1 251 2 182 61.81 115 Selected items Common set-up	176.6175 1.384 107.15516 0.98 79.1014 0.976 H13NO2S 247.31172 Hazard Price	0.494623 0 0.494623 0 0.494623 0 0	0.127614 0.1 0.218684 0.2 5 4.8 moles: 9 theor:	76618 1 1431 2 9 61.8193862 yield:	2
Link I Link I Link I Link Conditions Conditions Reagents Yield Price / availability Safety	entifier* Benzenesulfonyl Benzylamine Pyridine Pyridine Name Benzenesulfonyl chloride Benzylamine Pyridine 97 Possible items Common set-up Instruments for controlled add Unconventional activation tec	chloride Imethyl)benzenesulfona 2.83 5.66 175	C6H5CIO2S C7H9N C5H5N mide C133 cs eq. Bp 1 251 2 182 61.81 115 Selected items	176.6175 1.384 107.15516 0.98 79.1014 0.976 MW: H13NO2S 247.31172 Hazard Price Image: State	0.494623 2	0.127614 0.1 0.218684 0.2 5 4.8 moles: 9 theor:	76618 1 1431 2 9 61.8193862 yield:	2
Link id Link id Link Conditions Reagents Yield Price / availability Safety Technical setup	entifier* Benzenesulfonyl Benzylamine Pyridine Name Benzenesulfonyl chloride Benzylamine Pyridine 97 Possible items Common set-up Instruments for controlled add Unconventional activation tec Possible items Heating, > 1h Cooling to 0°C Cooling, < 0°C	chloride Imethyl)benzenesulfon 2.83 5.66 175 dition of chemicals	C6H5CIO2S C7H9N C5H5N C5H5N C131 c131 c131 c131 c131 c131 c131 c131	176.6175 1.384 107.15516 0.98 79.1014 0.976 H13NO2S 247.31172 Hazard Price ● ● ●	[100%] [100%] [100%] [100%] [0.494623] [2] [0.494623] [2] [0] [-5] [0] [0]	0.127614 0.1 0.218684 0.2 5 4.8 moles: 9 theor:	76618 1 1431 2 9 61.8193862 yield:	2

<u>Please note</u>: Since not enough information are provided on the amount of sodium sulfate, silica and solvents used for the separation, they have been neglected in calculations for the purification process. Since neither the amount of hydrochloric acid solution was given, we considered a minimum 5 mL of HCl 1 N (3x5 mL = 15 mL) and a minimum of 5 mL of brine (solubility of NaCl in water: 358 g/L).

7. Spectra















S45





S47





























170 160 150 140 130 120 110 100 90 f1 (ppm) -10






























120 110 f1 (ppm) -1 180 170 160 150 140 130 40 230 220 210 200 190







110 100 f1 (ppm) -; -10













S82



130 120 110 100 90 f1 (ppm) -10

























140 130 120 110 100 f1 (ppm) -2 -10









170 160 150 140 130 120 110 100 90 f1 (ppm) -10































8. References

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