Supporting Information

Synthesis of β-nitrostyrenes in the Presence of Sulfated Zirconia and Secondary Amines

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1. General methods

Commercially available reagents and solvents were used as received. Column chromatography was performed on Kiesel gel silica gel 60 (230-400 mesh). Melting points were determined using a Fisher-Johns apparatus and are uncorrected. The NMR spectra were obtained using Bruker Ascend-400 (400 MHz) and Bruker Avance DMX-500 (500 MHz) spectrometers. Chemical shifts (δ) are given in ppm and coupling constants J are given in hertz (Hz). Mass spectra (MS) were recorded on a GC-MS (Agilent Technologies 6890N, Detector 5973), in the chemical ionization mode using methane UAP grade as ionization gas. Microwave irradiation experiments were performed using a Discover System (CEM Corporation) single-mode microwave with standard sealed microwave glass vials. The nitrogen adsorption-desorption isotherm of sulfated zirconia was obtained at −196 °C on Micromeritics ASAP 2020 equipment. Powder X-ray diffraction (XRD) was performed using a Philips X’Pert Instrument with Cu-Kα radiation (45kV, 40 mA). Chemical composition of SZ was recorded by an EDX detector on a Zeiss SUPRA VP instrument. All nitrostyrenes 2a-m are known compounds.

2. Experimental procedures

Synthesis and Characterization of Sulfated Zirconia (SZ)
Sulfated zirconia was synthesized according to our previously reported method.1 Figure 1 shows XRD plot corresponding to the crystalline zirconia tetragonal as the predominantly phase which is given by reflections in 2θ = 30.18° as well as peaks in 34.616°, 35.283°, 43.002°, 50.214°, 50.770°, 59.291°, 60.187°, 62.724°, 72.894°, 74.617° and 81.768°. The nitrogen adsorption-desorption isotherm of SZ showed a profile corresponding to type IV of the IUPAC classification, typical for mesoporous materials (Figure 2). The SZ surface area, pore volume, and pore size were 90.75 m²·g⁻¹, 0.12 cm³·g⁻¹, and 52.7 Å, respectively (Table 1). SEM-EDX analysis revealed the presence of sulphur in the zirconia structure, as shown in the emission spectra of Figure 3 and elemental composition (Table 2). Once the sulfated zirconia was recycled is evident the monoclinic phase formation according with the intensification of peak in 2θ = 28° and the peak formation in 2θ = 32° (Figure 4 and 5), even though the sulphur content did not change significantly after the fourth reuse (Table 4).
a) Characterization of synthesised SZ

**Figure 1.** XRD pattern of synthesized SZ

**Figure 2.** Nitrogen adsorption-desorption isotherm of synthesised SZ

**Table 1.** Textural properties of synthesised SZ

<table>
<thead>
<tr>
<th>BET Area (m²/g)</th>
<th>Pore Volume (cm³/g)</th>
<th>Pore Size (Å)</th>
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<tbody>
<tr>
<td>90.75</td>
<td>0.12</td>
<td>52.73</td>
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**Table 2.** Composition of synthesised SZ

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight%</th>
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<tbody>
<tr>
<td>C</td>
<td>11.07</td>
</tr>
<tr>
<td>O</td>
<td>27.31</td>
</tr>
<tr>
<td>S</td>
<td>0.32</td>
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<tr>
<td>Zr</td>
<td>61.30</td>
</tr>
<tr>
<td>Total</td>
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</table>

**Figure 3.** SEM-EDX spectra of synthesised SZ
b) Characterization of recovered SZ

Figure 4. XRD pattern of recovered SZ

Figure 5. Nitrogen adsorption-desorption isotherm of recovered SZ

Table 3. Textural properties of recovered SZ

<table>
<thead>
<tr>
<th>BET Area (m²/g)</th>
<th>Pore Volume (cm³/g)</th>
<th>Pore Size (Å)</th>
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<tr>
<td>86.15</td>
<td>0.089</td>
<td>41.62</td>
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Table 4. Composition of recovered SZ

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight%</th>
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</thead>
<tbody>
<tr>
<td>C</td>
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<tr>
<td>O</td>
<td>26.71</td>
</tr>
<tr>
<td>S</td>
<td>0.37</td>
</tr>
<tr>
<td>Zr</td>
<td>49.56</td>
</tr>
<tr>
<td>Totals</td>
<td>100.00</td>
</tr>
</tbody>
</table>

Figure 6. SEM-EDX spectra of recovered SZ
Identification of the Brønsted and Lewis acids sites present in synthesised SZ

The quantification of acids sites presents in the material was determined by FT-IR (Nicolet 750 Spectrometer). The sample was passed into thin wafers (10 mg cm\(^{-2}\)) and pre-treated in a quartz cell under vacuum (10\(^{-6}\) mbar) at 450 °C during 4h. Then the sample was cooled at room temperature and expose to pyridine (1 \(\mu\)L) (\(P_{eq} = 2-3\) mbar). The pyridine excess was removed under vacuum (1 h). Finally, the spectra were recorded at 50-400 °C (Figure 7) and the concentration of Brønsted and Lewis acids sites (Table 5) were calculated by the following equation:\(^2\)

\[
n_i = \frac{A_i a_c}{\varepsilon_i \rho m}
\]

\(n_i\) is the amount of type \(i\) acid sites (\(\mu\)mmol g\(^{-1}\))

\(A_i\) is the integrated absorbance in cm\(^{-1}\)

\(a_c\) is the cross-sectional area in square centimeter of the wafer

\(\varepsilon_i\) is the integrated molar extinction coefficient in cm \(\mu\)mmol\(^{-1}\)

\(m\) is the mass of the sample

![Fig. 7 FT-IR spectra of SZ after pyridine desorption at different temperatures: a) dark blue 50 °C, purple 100 °C, turquoise 200 °C, green 300 °C and red 400 °C](image)

### Table 5. The nitrogen adsorption–desorption analysis parameters of materials

<table>
<thead>
<tr>
<th>Entry</th>
<th>Parameters</th>
<th>Synthesised SZ</th>
<th>Recovered SZ</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>(S_{BET}) (m(^2)·g(^{-1}))</td>
<td>90.75</td>
<td>86.15</td>
</tr>
<tr>
<td>2</td>
<td>Pore volume (cm(^3)·g(^{-1}))</td>
<td>0.12</td>
<td>0.089</td>
</tr>
<tr>
<td>3</td>
<td>Pore size (Å)</td>
<td>52.73</td>
<td>41.62</td>
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</table>
General procedure for the synthesis of β-nitrostyrenes 2a-m

A mixture of SZ (50 mg) and amine (0.1 mmol, 10 μL) in dry toluene (1mL) was placed in a microwave tube having a magnetic stirrer. Subsequently, aromatic aldehyde (1 mmol) and nitromethane (3 mmol, 0.15 mL) were added to the mixture, which was heated under microwave irradiation (30 W, 110 °C) during 30 minutes. Then, SZ was removed by centrifugation and washed with CH₂Cl₂ (5x5mL). The combined organic extracts were evaporated, giving the corresponding β-nitrostyrene, which was purified by column chromatography (CH₂Cl₂ or hexanes-EtOAc 1:1) and recrystallization (CH₂Cl₂-hexanes, 1:2).

3. Characterization data

(E)-1-methyl-4-(2-nitrovinyl)benzene (2a)

The compound 2a was prepared from 1a (0.1 mL, 1 mmol) and nitromethane (0.15 mL, 3mmol) according to General Procedure. Yield: 137 mg (84%); light yellow solid; mp = 99-101°C (Lit.3 mp=102°C). ¹H NMR (400 MHz, DMSO-d₆): δ = 2.36 (s, 3H, CH₃), 7.29 (d, J = 8.0 Hz, 2H, ArH), 7.74 (d, J = 8.0 Hz, 2H, ArH), 8.08 (d, J = 13.6 Hz, 1H, =CH), 8.17 (d, J = 13.6 Hz, 1H, =CH). ¹³C NMR (100.6 MHz, DMSO-d₆): δ = 21.6 (CH₃), 128.0 (Cipso), 130.28(2xArCH), 130.32 (2xArCH), 137.7 (=CH), 139.8 (=CH), 143.0 (Cipso). MS (Cl) (C₉H₉NO₂): m/z =204 [M+41]+, 192 [M+29]+, 164 [M+1]+, 149, 121.

(E)-1-methoxy-3-(2-nitrovinyl)benzene (2b)

The compound 2b was prepared from 1b (136.15 mg, 1 mmol) and nitromethane (0.15 mL, 3mmol) according to General Procedure. Yield: 150 mg (84%); light yellow solid; mp = 91-95°C (Lit.3 mp=93-94°C). ¹H NMR (500 MHz, DMSO-d₆): δ = 3.81 (s, 3H, OCH₃), 7.08-7.11 (m, 1H, ArH), 7.37-7.42 (m, 2H, ArH), 7.45-7.47 (m, 1H, ArH), 8.09 (d, J = 13.6 Hz, 1H, =CH), 8.26 (d, J = 13.6 Hz, 1H, =CH). ¹³C NMR (125.7 MHz, DMSO-d₆): δ = 55.8 (OCH₃), 114.5 (ArCH), 118.8(ArCH), 123.1 (ArCH), 130.6 (ArCH), 132.1 (Cipso), 138.8 (=CH), 139.6 (=CH), 160.1 (Cipso). MS (Cl) (C₉H₉NO₃): m/z = 220 [M+41]+, 208 [M+29]+, 180 [M+1]+, 165, 152, 137.
(E)-1-methoxy-4-(2-nitrovinyl)benzene (2c)

The compound 2c was prepared from 1c (136.15 mg, 1 mmol) and nitromethane (0.15 mL, 3mmol) according to General Procedure. Yield: 131 mg (73%); light yellow solid; mp = 84-86°C (Lit. mp = 86-87°C). ¹H NMR (400 MHz, DMSO-d₆): δ = 3.83 (s, 3H, OCH₃), 7.05 (d, J = 8.8 Hz, 2H, ArH), 7.83 (d, J = 8.8 Hz, 2H, ArH), 8.09 (d, J = 13.5 Hz, 1H, =CH), 8.13 (d, J = 13.5 Hz, 1H, =CH). ¹³C NMR (100.6 MHz, DMSO-d₆): δ = 56.0 (OCH₃), 115.3 (2xArCH), 123.2 (Cipso), 132.4 (2xArCH), 136.2 (=CH), 139.8 (=CH), 163.0 (Cipso). MS (Cl) (C₉H₉NO₃): m/z = 220 [M+41]⁺, 208 [M+29]⁺, 180 [M+1]⁺, 165, 150, 137.

(E)-1,2-dimethoxy-4-(2-nitrovinyl)benzene (2d)

The compound 2d was prepared from 1d (166.18 mg, 1 mmol) and nitromethane (0.15 mL, 3mmol) according to General Procedure. Yield: 130 mg (62%); yellow solid; mp = 140-142°C (Lit. mp = 140-141°C). ¹H NMR (400 MHz, DMSO-d₆): δ = 3.82 (s, 3H, OCH₃), 3.83 (s, 3H, OCH₃), 7.06 (d, J = 8.3 Hz, 1H, ArH), 7.43 (dd, J = 1.8, 8.3 Hz, 1H, ArH), 7.49 (d, J = 1.8 Hz, 1H, ArH), 8.07 (d, J = 13.5 Hz, 1H, =CH), 8.21 (d, J = 13.5 Hz, 1H, =CH). ¹³C NMR (100.6 MHz, DMSO-d₆): δ = 56.2 (OCH₃), 56.3 (OCH₃), 111.8 (ArCH), 112.2 (ArCH), 123.4 (Cipso), 126.1 (ArCH), 136.4 (=CH), 140.3 (=CH), 149.7 (Cipso), 153.0 (Cipso). MS (Cl) (C₁₀H₁₁NO₄): m/z = 250 [M+41]⁺, 238 [M+29]⁺, 210 [M+1]⁺, 194, 178, 167.

(E)-1,2,3-trimethoxy-5-(2-nitrovinyl)benzene (2e)

The compound 2e was prepared from 1e (196.20 mg, 1 mmol) and nitromethane (0.15 mL, 3mmol) according to General Procedure. Yield: 174 mg (73%), yellow solid; mp = 114-116°C (Lit. mp=120°C). ¹H NMR (400 MHz, DMSO-d₆): δ = 3.73 (s, 3H, OCH₃), 3.83 (s, 6H, OCH₃), 7.24 (s, 2H, ArH), 8.06 (d, J = 13.5 Hz, 1H, =CH), 8.28 (d, J = 13.5, 1H, =CH). ¹³C NMR(100.6 MHz, DMSO-d₆): δ = 56.7 (2xOCH₃), 60.7 (OCH₃), 108.2 (2xArCH), 126.1 (Cipso), 137.9 (=CH), 140.1 (=CH), 149.7 (Cipso), 153.0 (Cipso). MS (Cl) (C₁₀H₁₁NO₄): m/z = 250 [M+41]⁺, 238 [M+29]⁺, 210 [M+1]⁺, 194, 178, 167.
141.5 (C<sub>ipso</sub>), 153.6 (2xC<sub>ipso</sub>). MS (Cl) (C<sub>11</sub>H<sub>13</sub>NO<sub>5</sub>): m/z = 280 [M+41]<sup>+</sup>, 268 [M+29]<sup>+</sup>, 240 [M+1]<sup>+</sup>, 224.

**(E)-5-bromo-6-(2-nitrovinyl)benzo[<i>d</i>][1,3]dioxole (2f)**

The compound 2f was prepared from 1f (229.03 mg, 1 mmol) and nitromethane (0.15 mL, 3 mmol) according to General Procedure. Yield: 176 mg (65%); light yellow solid; mp = 156-158°C. 1H NMR (400 MHz, DMSO-<i>d</i><sub>6</sub>): δ = 6.19 (s, 2H, OCH<sub>2</sub>O), 7.41 (s, 1 H, ArH), 7.67 (s, 1 H, ArH), 8.19 (s, 2H, =CH). 13C NMR (100.6 MHz, DMSO-<i>d</i><sub>6</sub>): δ = 103.6 (OCH<sub>2</sub>O), 108.0 (ArCH), 113.6 (ArCH), 120.1 (C<sub>ipso</sub>), 123.1 (C<sub>ipso</sub>), 137.2 (=CH), 138.9 (=CH), 148.6 (C<sub>ipso</sub>), 152.2 (C<sub>ipso</sub>). MS (Cl) (C<sub>9</sub>H<sub>6</sub>BrNO<sub>4</sub>): m/z = 314, 312 [M+41]<sup>+</sup>, 302, 300 [M+29]<sup>+</sup>, 274, 272 [M+1]<sup>+</sup>, 259, 257, 244, 242, 231, 229, 228, 226, 193, 176, 162.

**(E)-1-(2-nitrovinyl)benzene (2g)**

The compound 2g was prepared from 1g (0.1 mL, 1 mmol) and nitromethane (0.15 mL, 3 mmol) according to General Procedure. Yield: 104 mg (70%); light yellow solid; mp = 55-57°C (Lit. mp = 56°C). 1H NMR (500 MHz, DMSO-<i>d</i><sub>6</sub>): δ = 7.46-7.50 (m, 2H, ArH), 7.52-7.56 (m, 1H, ArH), 7.84-7.88 (m, 2H, ArH), 8.13 (d, <i>J</i> = 13.6 Hz, 1H, =CH), 8.22 (d, <i>J</i> = 13.6 Hz, 1H, =CH). 13C NMR (125.7 MHz, DMSO-<i>d</i><sub>6</sub>): δ = 129.6 (2xArCH), 130.2 (2xArCH), 130.8 (C<sub>ipso</sub>), 132.5 (ArCH), 138.5 (=CH), 139.7 (=CH). MS (Cl) (C<sub>8</sub>H<sub>7</sub>NO<sub>2</sub>): m/z = 190 [M+41]<sup>+</sup>, 178 [M+29]<sup>+</sup>, 150 [M+1]<sup>+</sup>, 135, 107.

**(E)-1-bromo-2-(2-nitrovinyl)benzene (2h)**

The compound 2h was prepared from 1h (0.11 mL, 1 mmol) and nitromethane (0.15 mL, 3 mmol) according to General Procedure. Yield: 192 mg (84%); light yellow solid; mp = 82-84°C (Lit. mp = 86°C). 1H NMR (500 MHz, DMSO-<i>d</i><sub>6</sub>): δ = 7.44-7.51 (m, 2H, ArH), 7.77-7.80 (m, 1H, ArH), 8.00-8.03 (m, 1H, ArH), 8.22 (d, <i>J</i> = 13.5 Hz, 1H, =CH), 8.25 (d, <i>J</i> = 13.5 Hz, 1H, =CH). 13C NMR (125.7 MHz, DMSO-<i>d</i><sub>6</sub>): δ = 125.7 (C<sub>ipso</sub>), 128.4 (ArCH), 129.5 (ArCH), 129.7 (C<sub>ipso</sub>), 133.5 (2xArCH), 136.4 (=CH), 140.1 (=CH). MS (Cl) (C<sub>9</sub>H<sub>6</sub>BrNO<sub>2</sub>): m/z = 270, 268 [M+41]<sup>+</sup>, 258, 256 [M+29]<sup>+</sup>, 230, 228 [M+1]<sup>+</sup>, 215, 213, 202, 200, 187, 185, 149, 132.
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(E)-1-bromo-4-(2-nitrovinyl)benzene (2i)

The compound 2i was prepared from 1i (185.02 mg, 1 mmol) and nitromethane (0.15 mL, 3 mmol) according to General Procedure. Yield: 169 mg (74%); yellow solid; mp = 141-143°C (Lit. mp = 148-150°C). $^1$H NMR (400 MHz, DMSO-d$_6$): δ = 7.70 (d, J = 8.6 Hz, 2H, ArH), 7.82 (d, J = 8.6 Hz, 2H, ArH), 8.12 (d, J = 13.6 Hz, 1H, =CH), 8.26 (d, J = 13.6 Hz, 1H, =CH). $^{13}$C NMR (100.6 MHz, DMSO-d$_6$): δ = 126.2 (C$_{ipso}$), 130.1 (C$_{ipso}$), 132.1 (2xArCH), 132.7 (2xArCH), 138.5 (=CH), 139.1 (=CH). MS (Cl) (C$_8$H$_6$BrNO$_2$): m/z = 270, 268 [M+41]$^+$, 258, 256 [M+29]$^+$, 230, 228 [M+1]$^+$, 215, 213, 187, 185, 150.

(E)-1-chloro-4-(2-nitrovinyl)benzene (2j)

The compound 2j was prepared from 1j (140.57 mg, 1 mmol) and nitromethane (0.15 mL, 3 mmol) according to General Procedure. Yield: 150 mg (82%); yellow solid; mp = 111-113°C (Lit. mp = 113-114°C). $^1$H NMR (500 MHz, DMSO-d$_6$): δ = 7.55 (d, J = 8.5 Hz, 2H, ArH), 7.89 (d, J = 8.4 Hz, 2H, ArH), 8.13 (d, J = 13.6 Hz, 1H, =CH), 8.26 (d, J = 13.6 Hz, 1H, =CH). $^{13}$C NMR (125.7 MHz, DMSO-d$_6$): δ = 129.7 (2xArCH), 129.8 (C$_{ipso}$), 131.9 (2xArCH), 137.2 (C$_{ipso}$), 138.4 (=CH), 139.0 (=CH). MS (Cl) (C$_8$H$_6$ClNO$_2$): m/z = 226, 224 [M+41]$^+$, 214, 212 [M+29]$^+$, 186, 184 [M+1]$^+$, 143, 141.

(E)-1-nitro-2-(2-nitrovinyl)benzene (2k)

The compound 2k was prepared from 1k (151.12 mg, 1 mmol) and nitromethane (0.15 mL, 3 mmol) according to General Procedure. Yield: 78 mg (40%); orange solid; mp = 103-105°C (Lit. mp = 103-105°C). $^1$H NMR (500 MHz, DMSO-d$_6$): δ = 7.78 (td, J = 1.4, 7.9 Hz, 1H, ArH), 7.86 (td, J = 0.8, 7.5 Hz, 1H, ArH), 7.97 (dd, J = 1.4, 7.7 Hz, 1H, ArH), 8.12 (d, J = 13.5, 1H, =CH), 8.20 (dd, J = 1.2, 8.1, 1H, ArH), 8.43 (d, J = 13.5 Hz, 1H, =CH). $^{13}$C NMR (125.7 MHz, DMSO-d$_6$): δ = 125.7 (ArCH), 126.2 (C$_{ipso}$), 130.7 (ArCH), 132.7 (ArCH), 134.7 (ArCH), 135.9 (=CH), 140.9 (=CH), 148.9 (C$_{ipso}$). MS (Cl) (C$_8$H$_6$N$_2$O$_4$): m/z = 235 [M+41]$^+$, 223 [M+29]$^+$, 195 [M+1]$^+$, 148, 134, 120, 92.

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(E)-1-nitro-4-(2-nitrovinyl)benzene (2l)

The compound 2l was prepared from 1l (151.12 mg, 1 mmol) and nitromethane (0.15 mL, 3 mmol) according to General Procedure. Yield: 138 mg (71%); orange solid; mp = 196-198°C (Lit. mp = 189-192°C). $^1$H NMR (500 MHz, DMSO-d$_6$): $\delta$ = 8.11 (d, $J = 8.9$ Hz, 2H, ArH), 8.23 (d, $J = 13.7$ Hz, 1H, =CH), 8.29 (d, $J = 8.8$ Hz, 2H, ArH), 8.36 (d, $J = 13.7$ Hz, 1H, =CH). $^{13}$C NMR (125.7 MHz, DMSO-d$_6$): $\delta$ = 124.5 (2xArCH), 131.3 (2xArCH), 137.0 (=CH), 137.3 (C$_{ipso}$), 141.4 (=CH), 149.3 (C$_{ipso}$). MS (CI) (C$_8$H$_6$N$_2$O$_4$): $m/z$ = 235 [M+41]$^+$, 223 [M+29]$^+$, 195 [M+1]$^+$, 165, 152.

(E)-2-(2-nitrovinyl)furan (2m)

The compound 2m was prepared from 1m (0.08 mL, 1 mmol) and nitromethane (0.15 mL, 3 mmol) according to General Procedure. Yield: 122 mg (88%); light yellow solid; mp = 71-73°C (Lit. mp = 72°C). $^1$H NMR (400 MHz, DMSO-d$_6$): $\delta$ = 6.76 (dd, $J = 1.8$, 3.5 Hz, 1H, ArH), 7.29 (d, $J = 3.5$ Hz, 1H, ArH), 7.75 (d, $J = 13.3$ Hz, 1H, =CH), 8.01 (d, $J = 1.3$ Hz, 1H, ArH), 8.02 (d, $J = 13.3$ Hz, 1H, =CH). $^{13}$C NMR (100.6 MHz, DMSO-d$_6$): $\delta$ = 114.2 (ArCH), 121.6 (ArCH), 126.6 (=CH), 134.9 (=CH), 146.9 (C$_{ipso}$), 148.6 (ArCH). MS (Cl) (C$_6$H$_5$NO$_3$): $m/z$ = 180 [M+41]$^+$, 168 [M+29]$^+$, 140 [M+1]$^+$, 97.

4. $^1$H and $^{13}$C NMR spectra for compounds 2a-2m
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$^1$H NMR and $^{13}$C NMR for compound 2a

$^1$H NMR and $^{13}$C NMR for compound 2b
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$^{1}H$ NMR and $^{13}C$ NMR for compound 2c
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\( ^1H \) NMR and \( ^{13}C \) NMR for compound 2d
$^1$H NMR and $^{13}$C NMR for compound 2e
$^1$H NMR and $^{13}$C NMR for compound 2f
Supporting Information

\[ \text{\textsuperscript{1}H NMR and \textsuperscript{13}C NMR for compound 2g} \]
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$^1$H NMR and $^{13}$C NMR for compound 2h
$^1$H NMR and $^{13}$C NMR for compound 2i
Supporting Information

$^1$H NMR and $^{13}$C NMR for compound 2j

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$^1$H NMR and $^{13}$C NMR for compound 2k
$^1$H NMR and $^{13}$C NMR for compound 2I
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$^1$H NMR and $^{13}$C NMR for compound 2m
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