Supporting Information

Catalyst-Free Synthesis of Trisubstituted Tetrahydrothiophenes in Water via Cascade Sulfa-Michale/Aldol (Henry) Type Reaction

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General methods

1H NMR spectra and 13C NMR spectra were respectively recorded on 300 MHz or 600 MHz and 75 MHz or 151 MHz NMR spectrometers. Chemical shifts (δ) were expressed in ppm with TMS as internal standard, and coupling constants (J) were reported in Hz. HRMS were recorded on a Varian 7.0 T FTICR-MS spectrometer. HPLC analyses were conducted on the ELITE Hypersil NH₂ 5 um on SHIMADZU and eluting with n-hexane/i-PrOH = 80/20, flow rate 1.0 mL/min, λ = 254 nm. Melting points were taken on a YuHua X-4 apparatus and were uncorrected. Routine monitoring of reaction was performed by TLC using precoated Haiyang GF254 silica gel TLC plates. All the column chromatography separations were done by using silica gel (200-300 mesh) at increased pressure. Petroleum ether used was of boiling range 60-80°C. The organic extracts were dried over anhydrous sodium sulfate. Evaporation of solvent was performed at reduced pressure. Relative configurations of the products were determined by comparison with the 1H NMR and 13C NMR analysis of known compounds. Some unknown compounds were further determined by HRMS.
The purification of product 3a with water

Fig. S1 The TLC analysis of the reaction mixture after cooling to room temperature.

Fig. S2 The TLC analysis of the product after washing with hot water twice.
Fig. S3 The $^1$H NMR of the chromatography-purified 3a and water-washing-purified 3a.
Fig. S4 The HPLC of the chromatography-purified 3a and water-washing-purified 3a.
$^1$H NMR, $^{13}$C NMR and HRMS data of the products

[(2R,3S,4S)-4-hydroxy-2-phenyltetrahydrothiophen-3-yl](phenyl)methanone (3a)$^1$

White solid; mp 105-107 °C; $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ = 7.72 (d, $J$ = 7.4 Hz, 2H), 7.49 (t, $J$ = 7.4 Hz, 3H), 7.35 (t, $J$ = 7.7 Hz, 2H), 7.24 (t, $J$ = 7.2 Hz, 2H), 7.18 (d, $J$ = 7.0 Hz, 1H), 5.21 (d, $J$ = 10.5 Hz, 1H), 4.94 (s, 1H), 4.11 (dd, $J$ = 10.5, 3.0 Hz, 1H), 3.76 – 3.54 (m, 2H), 3.16 (d, $J$ = 11.7 Hz, 1H). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ = 199.87, 139.65, 136.62, 133.74, 128.62, 128.58, 128.28, 127.99, 127.69, 77.51, 77.41, 77.09, 76.66, 62.79, 51.84, 41.09 ppm.

[(2R,3S,4S)-2-(4-chlorophenyl)-4-hydroxytetrahydrothiophen-3-yl](phenyl)methanone (3b)$^1$

White solid; mp 134-135 °C; $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ = 7.71 (d, $J$ = 7.4 Hz, 2H), 7.52 (d, $J$ = 7.4 Hz, 1H), 7.42 – 7.33 (m, 4H), 7.19 (d, $J$ = 8.4 Hz, 2H), 5.16 (d, $J$ = 10.5 Hz, 1H), 4.93 (s, 1H), 4.03 (dd, $J$ = 10.5, 3.0 Hz, 1H), 3.59 (dd, $J$ = 11.6, 3.6 Hz, 2H), 3.14 (d, $J$ = 11.6 Hz, 1H). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ = 199.26, 138.24, 136.45, 133.92, 133.29, 129.41, 128.73, 128.69, 128.23, 77.47, 77.33, 77.05, 76.62, 63.02, 50.96, 41.07 ppm.
(2R,3S,4S)-2-(3-chlorophenyl)-4-hydroxytetrahydrothiophen-3-yl)(phenyl)methanone (3c)

White solid; mp 128-130 °C; 1H NMR (300 MHz, CDCl₃) δ = 7.73 (d, J = 7.5 Hz, 2H), 7.51 (d, J = 6.5 Hz, 2H), 7.37 (t, J = 7.7 Hz, 2H), 7.29 (d, J = 3.0 Hz, 1H), 7.17 – 7.09 (m, 2H), 5.15 (d, J = 10.4 Hz, 1H), 4.94 (s, 1H), 4.04 (dd, J = 10.4, 3.0 Hz, 1H), 3.59 (dd, J = 11.7, 3.6 Hz, 2H), 3.14 (d, J = 11.7 Hz, 1H). 13C NMR (75 MHz, CDCl₃) δ = 199.01, 142.04, 136.44, 134.35, 133.90, 129.79, 128.71, 128.25, 128.05, 127.85, 126.42, 77.48, 77.39, 77.05, 76.63, 63.09, 50.92, 41.12 ppm.

(2R,3S,4S)-2-(2-chlorophenyl)-4-hydroxytetrahydrothiophen-3-yl)(phenyl)methanone (3d+4d)

White solid; mp 125-129 °C; 1H NMR (300 MHz, CDCl₃) δ = 7.73 (dd, J = 14.0, 7.8 Hz, 3H), 7.49 (d, J = 7.3 Hz, 1H), 7.33 (dd, J = 9.7, 5.6 Hz, 2H), 7.25 (d, J = 7.1 Hz, 1H), 7.17 – 7.01 (m, 2H), 5.62 (d, J = 10.3 Hz, 1H) (major), 5.30 (d, J = 8.4 Hz, 1H) (minor), 4.99 (s, 1H), 4.18 (dt, J = 49.5, 24.7 Hz, 2H), 3.57 (dd, J = 11.6, 3.5 Hz, 1H), 3.17 (d, J = 11.5 Hz, 1H). 13C NMR (75 MHz, CDCl₃) δ = 199.84, 137.16, 134.16, 133.77, 133.48, 129.86, 129.45, 128.75, 128.52, 128.31, 127.20, 77.51, 77.34, 77.09, 76.66, 60.26, 47.91, 40.91ppm. HRMS (QFT-ESI): calcd for C₁₇H₁₅ClO₂S [M-H]⁻: 317.0409, found 317.0407.
((2R,3S,4S)-2-(4-bromophenyl)-4-hydroxytetrahydrothiophen-3-yl)(phenyl)methanone (3e)

White solid; mp 126-128 °C; ¹H NMR (300 MHz, CDCl₃) δ = 7.71 (d, J = 7.5 Hz, 2H), 7.53 (t, J = 7.4 Hz, 1H), 7.41 – 7.31 (m, 6H), 7.61 (d, J = 10.5 Hz, 1H), 4.93 (s, 1H), 4.03 (dd, J = 10.5, 3.0 Hz, 1H), 3.65 – 3.44 (m, 2H), 3.14 (d, J = 11.7 Hz, 1H).

¹³C NMR (75 MHz, CDCl₃) δ = 199.18, 138.81, 136.43, 133.93, 131.64, 129.76, 128.74, 128.24, 121.41, 77.48, 77.35, 77.05, 76.63, 63.02, 50.96, 41.09 ppm.


((2R,3S,4S)-2-(3-bromophenyl)-4-hydroxytetrahydrothiophen-3-yl)(phenyl)methanone (3f)

White solid; mp 128-130 °C; ¹H NMR (300 MHz, CDCl₃) δ = 7.74 (d, J = 7.4 Hz, 2H), 7.67 (s, 1H), 7.54 (t, J = 7.4 Hz, 1H), 7.37 (dd, J = 14.0, 6.9 Hz, 3H), 7.29 (d, J = 8.2 Hz, 1H), 7.07 (t, J = 7.8 Hz, 1H), 5.15 (d, J = 10.4 Hz, 1H), 4.96 (s, 1H), 4.05 (dd, J = 10.4, 3.0 Hz, 1H), 3.60 (dd, J = 11.7, 3.6 Hz, 2H), 3.14 (d, J = 11.6 Hz, 1H).

¹³C NMR (75 MHz, CDCl₃) δ = 198.91, 142.34, 136.42, 133.91, 130.98, 130.77, 130.08, 128.72, 128.26, 126.93, 122.55, 77.51, 77.39, 77.09, 76.66, 63.15, 50.82, 41.13 ppm. HRMS (QFT-ESI): calcd for C₁₇H₁₅BrO₂S [M-H]⁻: 360.9903, found 360.9902.
((2R,3S,4S)-4-hydroxy-2-(2-nitrophenyl)tetrahydrothiophen-3-yl)(phenyl)methanone (3g+4g)

Yellow solid; mp 135-138 °C; \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta = 7.98\) (dd, \(J = 140.4, 7.9\) Hz, 1H), 7.79 – 7.71 (m, 2H), 7.64 – 7.42 (m, 3H), 7.35 – 7.24 (m, 3H), 5.62 (d, \(J = 10.2\) Hz, 1H) (minor), 5.32 – 5.22 (m, 1H) (major), 5.04 (s, 1H), 4.95 – 4.77 (m, 1H), 4.34 (ddd, \(J = 31.9, 8.6, 4.4\) Hz, 1H), 4.17 – 3.99 (m, 1H), 3.61 (dd, \(J = 11.7, 3.6\) Hz, 1H), 3.29 – 3.10 (m, 2H). \(^{13}\)C NMR (151 MHz, CDCl\(_3\)) \(\delta = 198.73, 198.48, 150.34, 149.56, 136.60, 136.55, 135.93, 134.68, 133.71, 133.52, 133.10, 132.64, 130.88, 130.79, 128.64, 128.56, 128.53, 128.41, 128.40, 128.29, 124.48, 124.16, 77.45, 77.31, 77.10, 76.89, 63.99, 62.34, 62.29, 60.46, 45.76, 45.71, 41.30, 39.12 ppm. HRMS (QFT-ESI): calcd for C\(_{17}\)H\(_{15}\)NO\(_4\)S [M-H]: 328.0649, found 328.0643.

((2R,3S,4S)-4-hydroxy-2-(4-nitrophenyl)tetrahydrothiophen-3-yl)(phenyl)methanone (3h)

Yellow solid; mp 136-140 °C; \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta = 8.13 – 8.03\) (m, 2H), 7.73 (d, \(J = 7.3\) Hz, 2H), 7.65 (d, \(J = 8.8\) Hz, 2H), 7.55 (dd, \(J = 15.8, 8.4\) Hz, 1H), 7.38 (t, \(J = 7.8\) Hz, 2H), 5.30 (d, \(J = 10.5\) Hz, 1H), 5.00 (d, \(J = 3.3\) Hz, 1H), 4.06 (dd, \(J = 10.5, 3.1\) Hz, 1H), 3.65 (dd, \(J = 11.7, 3.6\) Hz, 1H), 3.21 (dd, \(J = 24.1, 8.0\) Hz, 2H). \(^{13}\)C NMR (151 MHz, CDCl\(_3\)) \(\delta = 216.51, 77.28, 213.94, 198.47, 150.34, 149.56, 136.60, 136.55, 135.93, 134.68, 133.71, 133.52, 133.10, 132.64, 130.88, 130.79, 128.64, 128.56, 128.53, 128.41, 128.40, 128.29, 124.48, 124.16, 77.45, 77.31, 77.10, 76.89, 63.99, 62.34, 62.29, 60.46, 45.76, 45.71, 41.30, 39.12 ppm.
((2R,3S,4S)-4-hydroxy-2-p-tolyltetrahydrothiophen-3-yl)(phenyl)methanone (3i)

White solid; mp 100-103 °C; ¹'H NMR (300 MHz, CDCl₃) δ = 7.74 (d, J = 7.6 Hz, 2H), 7.51 (d, J = 7.4 Hz, 1H), 7.36 (dt, J = 12.6, 6.0 Hz, 4H), 7.05 (d, J = 7.8 Hz, 2H), 5.20 (d, J = 10.4 Hz, 1H), 4.94 (s, 1H), 4.12 (dd, J = 10.5, 3.0 Hz, 1H), 3.76 – 3.53 (m, 2H), 3.15 (d, J = 11.6 Hz, 1H), 2.27 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ = 199.82, 137.37, 136.64, 136.51, 133.70, 129.25, 128.62, 128.32, 127.86, 77.54, 77.40, 77.12, 76.69, 62.79, 51.50, 41.05, 21.02 ppm.

((2R,3S,4S)-4-hydroxy-2-(4-methoxyphenyl)tetrahydrothiophen-3-yl)(phenyl)methanone (3j)

White solid; mp 115-117 °C; ¹'H NMR (300 MHz, CDCl₃) δ = 7.72 (d, J = 7.6 Hz, 2H), 7.50 (d, J = 7.4 Hz, 1H), 7.40 – 7.33 (m, 4H), 6.76 (d, J = 8.6 Hz, 2H), 5.17 (d, J = 10.5 Hz, 1H), 4.92 (s, 1H), 4.08 (dd, J = 10.6, 3.0 Hz, 1H), 3.72 (s, 3H), 3.58 (dd, J = 11.6, 3.7 Hz, 1H), 3.13 (d, J = 11.9
\[ \text{H}_3\text{C} \quad \text{O} \quad \text{S} \quad \text{H} \quad \text{O} \quad \text{C} \]

\[ (2R,3S,4S)-4\text{-hydroxy-2-phenyltetrahydrothiophen-3-yl})(p\text{-tolyl})\text{methanone (3k)} \]

White solid; mp 125-127 °C; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta = 7.61 (d, J = 8.1 \text{ Hz}, 2H), 7.47 (d, J = 7.1 \text{ Hz}, 2H), 7.23 (dd, J = 13.8, 6.3 \text{ Hz}, 3H), 7.14 (d, J = 8.0 \text{ Hz}, 2H), 5.18 (d, J = 10.5 \text{ Hz}, 1H), 4.92 (s, 1H), 4.08 (dd, J = 10.5, 2.9 \text{ Hz}, 1H), 3.91 – 3.49 (m, 2H), 3.17 (d, J = 11.6 \text{ Hz}, 1H), 2.36 (s, 3H). \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta = 199.76, 144.89, 139.60, 134.13, 129.30, 129.17, 128.79, 128.62, 128.55, 128.46, 127.97, 127.86, 127.66, 78.79, 77.48, 77.43, 77.06, 76.63, 64.30, 62.31, 52.15, 51.77, 41.08, 21.63, 21.60 \text{ ppm. HRMS (QFT-ESI): calcd for C}_{18}H_{18}O_2S [M-H]: 297.0955, found 297.0953.}

\[ \text{Cl} \quad \text{O} \quad \text{S} \quad \text{H} \quad \text{O} \quad \text{C} \]

\[ (4\text{-chlorophenyl})(2R,3S,4S)-4\text{-hydroxy-2-phenyltetrahydrothiophen-3-yl})\text{methanone (3l)}^1 \]

White solid; mp 135-137 °C; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta = 7.63 (d, J = 8.6 \text{ Hz}, 2H), 7.45 (d, J = 6.9 \text{ Hz}, 2H), 7.36 – 7.13 (m, 5H), 5.17 (d, J = 10.4 \text{ Hz}, 1H), 4.94 (s, 1H), 4.03 (dd, J = 10.4, 3.0 \text{ Hz}, 1H), 3.72 (s, 1H), 3.59 (dd, J = 11.6, 3.6 \text{ Hz}, 1H), 3.15 (d, J = 11.6 \text{ Hz}, 1H). \(^{13}\)C NMR (75
MHz, CDCl$_3$ $\delta = 198.59, 140.25, 139.54, 134.90, 129.68, 128.91, 127.97, 127.82, 77.55,$
77.38, 77.13, 76.71, 62.89, 51.76, 41.07 ppm.

(4-chlorophenyl)((2R,3S,4S)-2-(4-chlorophenyl)-4-hydroxytetrahydrothiophen-3-yl)methanone (3m)

White solid; mp 143-146°C; $^1$H NMR (300 MHz, CDCl$_3$) $\delta = 7.69 – 7.61$ (m, 2H), 7.38 (dd, $J = 13.0, 8.5$ Hz, 4H), 7.22 (d, $J = 8.4$ Hz, 2H), 5.15 (d, $J = 10.5$ Hz, 1H), 4.93 (s, 1H), 3.97 (dd, $J = 10.5, 3.0$ Hz, 1H), 3.66 – 3.39 (m, 2H), 3.16 (d, $J = 11.8$ Hz, 1H). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta =$ 198.07, 140.55, 138.10, 134.74, 133.46, 129.61, 129.33, 129.06, 128.78, 77.43, 77.30, 77.01, 76.59, 63.06, 50.92, 41.08 ppm. HRMS (QFT-ESI): calcd for C$_{17}$H$_{14}$Cl$_2$O$_2$S [M-H]$^-$: 351.0019, found 351.0016.

((2R,3S,4S)-2-(2,4-dichlorophenyl)-4-hydroxytetrahydrothiophen-3-yl)(phenyl)methanone (3n+4n)

White solid; mp 139-142°C; $^1$H NMR (300 MHz, CDCl$_3$) $\delta = 7.69$ (t, $J = 8.9$ Hz, 3H), 7.52 (d, $J = 7.4$ Hz, 1H), 7.35 (t, $J = 7.7$ Hz, 2H), 7.30 – 7.08 (m, 2H), 5.56 (d, $J = 10.3$ Hz, 1H) (major), 5.24
(d, J = 8.2 Hz, 1H) (minor), 4.99 (s, 1H), 4.16 (dd, J = 10.2, 3.0 Hz, 2H), 3.56 (dd, J = 11.6, 3.5 Hz, 1H), 3.16 (d, J = 11.6 Hz, 1H). $^{13}$C NMR (75 MHz, CDCl$_3$) δ = 199.37, 136.30, 133.91, 130.41, 129.52, 129.19, 128.60, 128.53, 128.28, 127.70, 127.49, 77.49, 77.22, 77.06, 76.64, 60.47, 47.31, 40.91 ppm. HRMS (QFT-ESI): calcd for C$_{17}$H$_{14}$Cl$_2$O$_2$S [M-H]: 351.0019, found 351.0017.

![Chemical Structure](image1)

**((2R,3S,4S)-2-(2,6-dichlorophenyl)-4-hydroxytetrahydrothiophen-3-yl)(phenyl)methanone (3o+4o)**

White solid; mp 142-145 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ = 7.75 (d, J = 7.6 Hz, 2H), 7.49 (t, J = 7.4 Hz, 1H), 7.34 (t, J = 7.7 Hz, 2H), 7.29 – 7.16 (m, 2H), 7.03 (t, J = 8.0 Hz, 1H), 6.10 (d, J = 10.6 Hz, 1H), 5.62 (d, J = 10.5 Hz, 1H), 5.03 (s, 1H), 4.95 (dd, J = 10.7, 2.8 Hz, 1H), 3.65 (dd, J = 11.6, 2.8 Hz, 2H), 3.20 (d, J = 11.6 Hz, 1H). $^{13}$C NMR (75 MHz, CDCl$_3$) δ = 199.91, 136.65, 133.72, 133.61, 133.38, 129.00, 128.90, 128.52, 128.32, 128.24, 128.15, 77.75, 77.48, 77.06, 76.63, 55.75, 45.48, 41.96 ppm. HRMS (QFT-ESI): calcd for C$_{17}$H$_{14}$Cl$_2$O$_2$S [M+Na]$^+$: 374.9984, found 374.9977.

![Chemical Structure](image2)

**((3R,3aR,7aS)-3-hydroxyhexahydrobenzo[b]thiophen-4(2H)-one (6a)**

(3R,3aR,7aS)-3-hydroxyhexahydrobenzo[b]thiophen-4(2H)-one (6a)
White solid; mp 70-72 °C; $^1$H NMR (300 MHz, CDCl$_3$) $\delta = 4.98$ (s, 1H), 4.33 – 4.07 (m, 1H), 3.19 (dd, $J = 11.6, 4.6$ Hz, 1H), 2.88 (dd, $J = 5.8, 2.6$ Hz, 1H), 2.79 (dd, $J = 11.5, 1.7$ Hz, 1H), 2.59 (s, 1H), 2.46 – 2.07 (m, 5H), 2.02 – 1.84 (m, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta = 209.71, 77.46, 77.03, 76.61, 74.72, 61.79, 46.85, 41.08, 38.46, 27.00, 21.75$ ppm.

![Chemical structure](image)

(3R,3aR,6aS)-3-hydroxytetrahydro-2H-cyclopenta[b]thiophen-4(5H)-one (6b)$^2$

White solid; mp 68-70 °C; $^1$H NMR (600 MHz, CDCl$_3$) $\delta = 4.81$ (s, 1H), 4.30 (t, $J = 5.9$ Hz, 1H), 3.05 (dd, $J = 12.1, 4.0$ Hz, 1H), 2.96 (d, $J = 6.7$ Hz, 1H), 2.87 (d, $J = 12.0$ Hz, 1H), 2.59 – 2.54 (m, 1H), 2.37 – 2.27 (m, 3H), 2.21 (dd, $J = 15.6, 5.9$ Hz, 1H). $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta = 216.51, 77.28, 77.07, 76.86, 64.97, 47.27, 41.32, 38.46, 35.49, 27.68$ ppm.

![Chemical structure](image)

(3R,3aR,6S,7aS)-3-hydroxy-3a-methyl-6-(prop-1-en-2-yl)hexahydrobenzo[b]thiophen-4(2H)-one (6c)$^2$

Colorless oil; $^1$H NMR (300 MHz, CDCl$_3$) $\delta = 4.77$ (dd, $J = 9.6, 5.7$ Hz, 3H), 3.78 (s, 1H), 3.09 (dd, $J = 11.7, 3.9$ Hz, 2H), 2.75 – 2.55 (m, 2H), 2.47 – 2.29 (m, 2H), 2.00 (dd, $J = 11.0, 3.4$ Hz, 2H), 1.74 (s, 3H), 1.30 (s, 3H). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta = 213.36, 146.85, 110.13, 78.26, 61.22, 50.22, 43.15, 39.67, 36.97, 29.05, 20.80, 17.81$ ppm.
1-((2R,3S,4S)-4-hydroxy-2-(2,6,6-trimethylcyclohex-1-enyl)tetrahydrothiophen-3-yl)ethanone (6d)

White solid; mp 98-102 °C; $^1$H NMR (300 MHz, CDCl$_3$) $\delta = 4.77$ (s, 1H), 4.45 (d, $J = 11.8$ Hz, 1H), 4.21 (s, 1H), 3.58 (dd, $J = 11.8$, 2.6 Hz, 1H), 3.26 (dd, $J = 11.6$, 3.3 Hz, 1H), 2.99 (d, $J = 11.7$ Hz, 1H), 2.19 (s, 3H), 2.01 (s, 1H), 1.98 (s, 3H), 1.65 – 1.33 (m, 5H), 1.04 (s, 3H), 0.84 (s, 3H).

$^{13}$C NMR (75 MHz, CDCl$_3$) $\delta = 212.62, 134.79, 133.66, 76.07, 63.35, 59.92, 45.80, 39.77, 39.53, 34.97, 32.73, 29.08, 26.84, 21.24, 19.12$ ppm.

(4R,5S)-5-(2-methoxyphenyl)-4-nitrotetrahydrothiophen-3-ol (9a+10a)

Yellow oil; $^1$H NMR (300 MHz, CDCl$_3$) $\delta = 7.48$ (d, $J = 7.5$ Hz, 1H), 7.37 – 7.26 (m, 1H), 6.98 (t, $J = 7.4$ Hz, 1H), 6.90 (d, $J = 8.2$ Hz, 1H), 5.67 (s, 1H), 5.29 (d, $J = 8.3$ Hz, 1H), 5.13 (t, $J = 7.6$ Hz, 1H), 5.04 – 4.90 (m, 1H), 3.84 (s, 3H), 3.26 (dd, $J = 11.1$, 6.1 Hz, 1H), 3.14 (dd, $J = 12.9$, 5.6 Hz, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta = 157.17, 130.12, 129.77, 129.26, 128.38, 125.32, 120.93, 120.61, 111.07, 110.50, 96.16, 77.83, 77.46, 77.04, 76.61, 76.24, 57.67, 55.51, 55.20, 44.98, 38.45, 36.96, 34.97$ ppm.
(4R,5R)-4-nitro-5-(thiophen-2-yl)tetrahydrothiophen-3-ol (9b+10b)\(^3,4\)

Yellow oil; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta = 7.29\) (d, \(J = 5.1\) Hz, 1H), 7.09 (dd, \(J = 11.6, 3.2\) Hz, 1H), 7.02 – 6.90 (m, 1H), 5.60 – 5.39 (m, 1H) (minor), 5.34 – 5.17 (m, 1H) (major), 5.06 – 4.79 (m, 2H), 3.90 – 2.95 (m, 5H). \(^1\)C NMR (75 MHz, CDCl\(_3\)) \(\delta = 34.1, 36.0, 43.9, 44.8, 75.4, 76.9, 96.1, 98.5, 126.1, 126.4, 126.8, 127.2, 140.8, 140.9\) ppm.

(4R,5R)-5-(furan-2-yl)-4-nitrotetrahydrothiophen-3-ol (9c+10c)\(^3,4\)

Yellow oil; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta = 7.46\) (s, 1H), 7.41 – 7.24 (m, 2H), 6.65 – 6.19 (m, 6H), 5.28 (s, 1H), 5.17 (s, 1H), 5.03 (d, \(J = 14.1\) Hz, 4H), 4.93 – 4.84 (m, 2H), 4.78 (d, \(J = 1.7\) Hz, 2H), 3.85 (dd, \(J = 11.7, 6.7\) Hz, 3H), 3.57 – 3.33 (m, 4H), 3.21 – 3.07 (m, 3H), 3.06 – 2.91 (m, 5H). \(^1\)C NMR (75 MHz, CDCl\(_3\)) \(\delta = 149.01, 148.61, 143.52, 143.27, 143.23, 110.94, 110.82, 110.71, 109.84, 109.11, 79.37, 77.98, 77.09, 76.66, 40.50, 37.21, 35.93, 33.91\) ppm.
$^1$H NMR, $^{13}$C NMR and HRMS Spectra Copies of the products

3a
3b
3e
3g+4g
3h
3m
3n+4n
References:


