
To a solution of imidazole (10.88 g, 0.16 mol) in 400 ml CH$_2$Cl$_2$ was added triethylamine (12.24 mL, 0.088 mol) and prolinol (4.046 g, 0.04 mol) with stirring, the reaction was cooled to 0°C, followed by the dropwise addition of thionyl chloride (3.28 mL, 0.042 mol), the mixture was allowed to warm to rt and stirred overnight, then 200 ml
water was added, the organic phase was separated and the aqueous phase was washed
with CH$_2$Cl$_2$ (40 mL × 2) twice, the combined organic phase was washed with water (40
mL), dried with Na$_2$SO$_4$ and concentrated in vacuo after filtration to give a yellow oil,
which was used without any purification in the next step.

The yellow oil was dissolved in CH$_3$CN (250 mL) under ice-water bath, RuCl$_3$ (80 mg,
0.38 mmol) and NaIO$_4$ (12.8 g, 0.06 mol) was added in portion, followed by the addition
of 200 ml water, the solution was stirred overnight under rt, quenched with 200 ml Et$_2$O,
the organic phase was collected, the aqueous phase was filtered and then transferred to a
separatory funnel, washed with Et$_2$O (40 mL × 2), the combined organic phase was
washed with 50 ml saturated aquous NaHCO$_3$, 50 ml brine, dried with Na$_2$SO$_4$, filtered,
concentrated in vacuo, the residue was purified by flash chromatography on silica gel to
afford the sulfamidate as a white solid (5.62 g, total yield of 86%). [α]$_D^{20\circ}$ = +40.1°
(C=1.0, CHCl$_3$); $^1$H NMR (300 MHz, CDCl$_3$): δ 1.74-1.83 (1H, m), 1.86-1.95 (2H, m),
2.08-2.20 (1H, m), 3.17-3.26 (1H, m), 3.57-3.65 (1H, m), 3.98-4.04 (1H, m), 4.19-4.27
(1H, m), 4.48-4.54 (1H, m); $^{13}$C NMR (CDCl$_3$, 75 MHz): δ 25.13, 31.30, 51.06,
62.44, 71.73; HRMS (EI) for C$_5$H$_9$NO$_3$S (M): calcd. 163.0303, found 163.0305.

[α]$_D^{20\circ}$=+0.6° (C=1.0, CHCl$_3$); $^1$H NMR (300 MHz, CDCl$_3$): δ 1.55
(3H, d, $J$=6.22Hz), 4.29 (1H, t, $J$=8.67Hz), 4.72 (1H, dd, $J$=5.65 Hz,
5.65Hz, 8.67 Hz), 5.07-5.14 (1H, m); $^{13}$C NMR (CDCl$_3$, 75 MHz): δ 17.60, 74.33, 80.15.
HRMS (EI) for C$_3$H$_5$O$_4$S (M-1): calcd. 136.9909, found 136.9907.
$\alpha D$ 20℃ = +48.4° (C=1.0, CHCl₃); $^1$H NMR (300 MHz, CDCl₃): δ 1.26-1.45 (2H, m), 1.51-1.64 (1H, m), 1.74-1.88 (3H, m), 2.70 (1H, td, $J = 11.87$ Hz, 2.83 Hz), 3.35-3.49 (2H, m), 4.13 (1H, dd, $J = 7.91$ Hz, 8.1 Hz, 9.79 Hz), 4.54 (1H, dd, $J = 5.27$ Hz, 5.84 Hz, 7.91 Hz); $^{13}$C NMR (CDCl₃, 75 MHz): δ 21.79, 23.32, 27.63, 43.58, 57.16, 73.75; HRMS (EI) for C₆H₁₁NO₃S (M): calcd. 177.0460, found 177.0459.

$\alpha D$ 20℃ = +33.6° (C=1.0, CHCl₃); $^1$H NMR (300 MHz, CDCl₃): δ 0.96 (6H, dd, $J = 4.90$ Hz, 4.90 Hz, 6.97 Hz), 1.96-2.08 (1H, m), 2.77 (3H, s), 3.30-3.36 (1H, m), 4.23 (1H, dd, $J = 7.35$ Hz, 7.91 Hz, 8.67 Hz), 4.44 (1H, dd, $J = 7.35$ Hz, 7.35 Hz, 8.48 Hz); $^{13}$C NMR (CDCl₃, 75 MHz): δ 21.79, 23.32, 27.63, 43.58, 57.16, 73.75; HRMS (EI) for C₆H₁₃NO₃S (M): calcd. 179.0616, found 179.0615.

$\alpha D$ 20℃ = +67.7° (C=1.0, CHCl₃); $^1$H NMR (300 MHz, CDCl₃): δ 0.92 (6H, t, $J = 6.4$ Hz), 1.38-1.46 (1H, m), 1.54-1.67 (2H, m), 2.72 (3H, s), 3.46-3.55 (1H, m), 4.10 (1H, t, $J = 8.28$ Hz), 4.55 (1H, dd, $J = 6.60$ Hz, 6.78 Hz, 8.29 Hz); $^{13}$C NMR (CDCl₃, 75 MHz): δ 22.04, 23.33, 24.38, 32.22, 40.36, 59.21, 71.92; HRMS (EI) for C₇H₁₅NO₃S (M): calcd. 193.0773, found 193.0772.

$\alpha D$ 20℃ = +25.1° (C=1.0, CHCl₃); $^1$H NMR (300 MHz, CDCl₃): δ 2.72-2.84 (4H, m), 3.16 (1H, dd, $J = 5.66$ Hz, 5.84 Hz, 13.57 Hz), 3.65-3.76 (1H, m), 4.19 (1H, dd, $J = 7.5$ 3 Hz, 7.72 Hz, 8.67 Hz), 4.35 (1H, dd, $J = 6.6$ Hz, 6.6 Hz, 8.66 Hz), 7.15-7.21 (2H, m), 7.27-7.37 (3H, m); $^{13}$C NMR (CDCl₃, 75 MHz): δ
32.69, 37.93, 61.72, 71.81, 127.53, 129.04, 129.07, 134.65; HRMS (EI) for C_{10}H_{13}NO_{3}S (M): calcd. 227.0616, found 227.0613.

\[ \alpha \]_D^{20\circ} = +3.7° (C=1.0, CHCl_3); \ H NMR (300 MHz, CDCl_3):  \delta 
1.50 (3H, d, J = 6.41 Hz), 1.55 (9H, s), 4.19 (1H, dd, J = 3.02 Hz, 9.04 Hz), 4.36-4.48 (1H, m), 4.66 (1H, dd, J = 6.02 Hz, 9.05 Hz); \ ^{13}C \ NMR (CDCl_3, 75 MHz):  \delta 18.31, 27.94, 53.80, 71.31, 85.36, 148.47; HRMS (EI) for C_{8}H_{15}NO_{5}S (M): calcd. 237.0671, found 237.0673.

\[ \alpha \]_D^{20\circ} = +14.5° (C=1.0, CHCl_3); \ H NMR (300 MHz, CDCl_3):  \delta 
0.96 (6H, dd, J = 6.59 Hz, 6.59 Hz, 9.23 Hz), 1.54 (9H, s), 1.57-1.84 (3H, m), 4.27-4.36 (2H, m), 4.63 (1H, dd, J = 6.03 Hz, 9.23 Hz); \ ^{13}C \ NMR (CDCl_3, 75 MHz):  \delta 21.66, 23.23, 24.68, 27.92, 40.96, 56.30, 70.03, 85.32, 148.61; HRMS (EI) for C_{11}H_{21}NO_{5}S (M): calcd. 279.1140, found 279.1143.
NMR Spectra:
NN
C₄H₉
N
H
N₃
O
O

21b

NN
C₄H₉
N
H
N₃
O
O

21b