

## Novel Pd(II)-catalysed *N,O*-bicyclisation as an efficient route to 6-oxa-2-aza-bicyclo[3.2.1]octane skeleton

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### *1*-(Benzyloxycarbonylamino)-hex-5-en-3-ol (**5**)

TLC:  $R_f = 0.58$  (hexanes/AcOEt = 2/3);

$^1\text{H NMR}$ :  $\delta_{\text{H}}$  (300 MHz,  $\text{CDCl}_3$ ) 1.54 (*dddd*, 1H,  $J_{2,3} = 4.3$ ,  $J_{2,1'} = J_{2,1} = 5.5$  Hz, H-2), 1.69 (*dddd*, 1H,  $J_{2',3} = 3.3$ ,  $J_{2',1} = 5.6$ ,  $J_{2',1'} = 8.8$ ,  $J_{2,2'} = 11.9$  Hz, H-2'), 2.17-2.27 (*m*, 2H,  $J_{4,5} = 1.2$ ,  $J_{4,3} = J_{4',5} = 7.4$ ,  $J_{4,4'} = 11.6$  Hz, H-4, H-4'); 2.28-2.76 (*br s*, 1H, exchange with  $\text{D}_2\text{O}$ , OH), 3.22 (*dt*, 1H,  $J_{1,2'} = J_{1,2} = 5.7$ ,  $J_{1,1'} = 11.3$  Hz, H-1), 3.44-3.51 (*m*, 1H,  $J_{1',2} = 5.4$ ,  $J_{1',2'} = 8.5$  Hz, H-1'), 3.71 (*dddd*, 1H,  $J_{3,4'} = 0.7$ ,  $J_{3,2'} = 3.1$ ,  $J_{3,2} = 4.9$ ,  $J_{3,4} = 7.4$  Hz, H-3), 5.10 (*m*, 3H, exchange with  $\text{D}_2\text{O}$ , NH,  $\text{CH}_2\text{Ph}$ ), 5.13 (*dt*, 2H,  $J_{6,6'} = 1.7$ ,  $J_{6,5} = 3.1$ ,  $J_{5,6'} = 9.6$  Hz, H-6, H-6'), 5.79 (*dddd*, 1H,  $J_{4,5} = 1.3$ ,  $J_{5,6} = 3.2$ ,  $J_{5,4'} = 7.3$ ,  $J_{5,6'} = 9.6$  Hz, H-5), 7.30-7.39 (*m*, 5H, Ph).

$^{13}\text{C NMR}$ :  $\delta_{\text{C}}$  (75 MHz,  $\text{CDCl}_3$ ) 36.6 (*t*, C-2), 38.1 (*t*, C-1), 41.9 (*t*, C-4), 66.7 (*t*,  $\text{CH}_2\text{Ph}$ ), 68.5 (*d*, C-5), 118.1 (*t*, C-6), 128.0, 128.1, 128.5 (all *d*, all CH-Ph), 134.5 (*d*, C-5), 136.4 (*s*,  $\text{C}_q\text{-Ph}$ ), 157.0 (*s*, C=O);

IR:  $\nu_{\text{max}}$  (film on KBr)/ $\text{cm}^{-1}$  1263 (*s*), 1531 (*s*), 1698 (*s*, C=O), 3342, 3411 (all *s*, NH, OH);

MS (MALDI):  $m/z$  249  $[\text{M}]^+$ , 271  $[\text{M}+\text{Na}-\text{H}]^+$ ;

Elemental analysis: Found C 67.21, H 7.71, N 5.58;  $\text{C}_{14}\text{H}_{19}\text{NO}_3$  requires C 67.45, H 7.68, N 5.62 %.

### *N*-(Benzyloxycarbonyl)-6-oxa-2-aza-bicyclo[3.2.1]octane (**6**)

TLC:  $R_f = 0.42$  (hexanes/AcOEt = 1/1);  $R_f = 0.45$  ( $\text{CHCl}_3/\text{PrOH} = 40/1$ );

$^1\text{H NMR}$ :  $\delta_{\text{H}}$  (300 MHz,  $\text{CDCl}_3$ , 50°C) 1.58 (*dddd*, 1H,  $J_{4,5} = 1.2$ ,  $J_{4,3'} = 6.9$ ,  $J_{4,3} = 10.9$ ,  $J_{4,4'} = 12.3$  Hz, H-4), 1.71 (*dtd*, 1H,  $J_{4',5} = 4.5$ ,  $J_{4',3} = 5.3$  Hz, H-4'), 1.76 (*d*, 1H,  $J_{8,8'} = 11.4$  Hz, H-8), 1.86 (*dd*, 1H,  $J_{8',5} = 6.2$ ,  $J_{8,8'} = 11.4$  Hz, H-8'), 3.29 (*ddd*, 1H,  $J_{3,4'} = 5.6$ ,  $J_{3,4} = 11.1$ ,  $J_{3,3'} = 13.5$  Hz, H-3), 3.87 (*dd*, 1H,  $J_{7,1} = 3.8$ ,  $J_{7,7'} = 9.1$  Hz, H-7), 3.94-4.01 (*m*, 2H,  $J_{7',1} = 0.9$ ,  $J_{7,7} = 9.1$ ,  $J_{3,3'} = 13.7$  Hz, H-7', H-3'), 4.52 (*dtd*, 1H,  $J_{5,4} = 1.2$ ,  $J_{5,4'} = 4.4$ ,  $J_{5,8'} = 5.9$  Hz, H-5), 4.80-4.88 (*br s*, 1H, H-1), 5.12 (*s*, 2H,  $\text{CH}_2\text{Ph}$ ), 7.29-7.41 (*m*, 5H, Ph).

# Supplementary Material (ESI) for Chemical Communications  
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$^{13}\text{C}$  NMR:  $\delta_{\text{C}}$  (75 MHz,  $\text{CDCl}_3$ ,  $50^\circ\text{C}$ ) 31.4 (*t*, C-4), 37.2 (*t*, C-8), 38.3 (*t*, C-3), 54.1 (*d*, C-1), 67.2 (*t*,  $\text{CH}_2\text{Ph}$ ), 72.0 (*t*, C-7), 74.3 (*d*, C-5), 128.0, 128.1, 128.6 (all *d*, all CH-Ph), 136.9 (*s*,  $\text{C}_q\text{-Ph}$ ), 154.7 (*s*, C=O);

IR:  $\nu_{\text{max}}$  (film on KBr)/ $\text{cm}^{-1}$  1075 (*s*), 1103 (*s*), 1220 (*s*), 1311 (*s*), 1423 (*s*), 1698 (*s*, C=O), 3342, 3411 (all *s*, NH, OH);

MS (MALDI):  $m/z$  247  $[\text{M}]^+$ , 269  $[\text{M}+\text{Na}-\text{H}]^+$ ;

Elemental analysis: Found C 68.02, H 6.90, N 5.71;  $\text{C}_{14}\text{H}_{17}\text{NO}_3$  requires C 68.00, H 6.93, N 5.66%.