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Supporting Information

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Synthese neuartiger enantiomerenreiner Kohlenhydratmimetika durch Lewis-Säure-induzierte Umlagerung von 1,3-dioxolanyl-substituierten 3,6-Dihydro-2*H*-1,2-oxazinen

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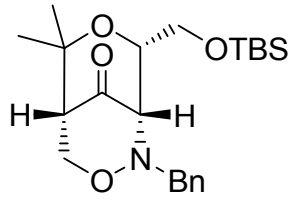
Supporting Informations: Typical Procedures and Analytical Data

(1*S*,5*R*,8*R*)-2-Benzyl-8-(*tert*-butyldimethylsiloxymethyl)-6,6-dimethyl-3,7-dioxa-2-azabicyclo[3.3.1]nonan-9-one



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Procedure:

Syn-**3** (1.00 g, 2.56 mmol) was dissolved in dichloromethane (20 ml), cooled to 0 ^{o}C and treated with *tert*-butyldimethyl silyltriflate (2.72 g, 10.2 mmol). After stirring at room temperature for 20 h, the mixture was cooled again to 0 ^{o}C and treated with triethylamine (3.92 g, 3.84 mmol). The resulting solution was stirred for 30 min at 0 ^{o}C, and then NH_4Cl solution (10 ml) was added. The two layers were separated and the aqueous layer was extracted three times with diethyl ether. The organic layer was dried ($MgSO_4$) and concentrated under vacuum to yield 1.82 g of the crude product as brownish crystals, which was filtered over silica gel (*n*-pentane:ethyl acetate = 7:1) giving the pure product **6** in 1.04 g (quant.) as colourless crystals. **Melting point:** 65-66 °C

Optical rotation: $[\alpha]_D^{22}$ = + 64.5 (c = 0.36, $CHCl_3$)

^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.08, 0.09 (2 s, 3 H each, $SiMe_2$), 0.88 (s, 9 H, *t*-Bu), 1.24, 1.42 (2 s, 3 H each, Me), 2.38 (dd, *J* = 3.0, 5.8 Hz, 1 H, 5-H), 3.39 (m_c, 1 H, 1-H), 3.82 (dd, *J* = 5.0, 7.0 Hz, 1 H, 8-CH_2), 3.96 (ddt, *J* = 1.9, 3.1, 7.0 Hz, 1 H, 8-H), 4.00 (d, *J* = 14.0 Hz, 1 H, NC$\underline{H}_2$Ph), 4.11 (t, *J* = 7.0 Hz, 1 H, 8-CH_2), 4.19 (d, *J* = 14.0 Hz, 1 H,

NC$\underline{H}_2$Ph), 4.46 (dd, J = 5.8, 12.0 Hz, 1 H, 4-H_B), 4.55 (dd, J = 3.0, 12.0 Hz, 1 H, 4-H_A), 7.26-7.41 (m, 5 H, Ph) ppm.

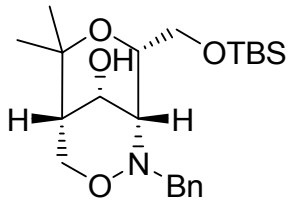
^{13}C NMR ($CDCl_3$, 125 MHz): δ = -5.3 (q, $SiMe_2$), 18.1, 23.7 (s, q, *t*-Bu), 26.7, 29.7 (2 q, Me), 58.1 (d, C-5), 60.2 (t, N$\underline{C}H_2$Ph), 61.6 (t, 8-CH_2), 68.8 (t, C-4), 70.5 (d, C-1), 76.2 (d, C-8), 78.3 (s, C-6), 127.4, 128.3, 128.7, 136.5 (3 d, s, Ph), 208.2 (s, C=O) ppm.

IR (KBr): ν = 3060-3030 cm^{-1} (=C-H), 2975-2875 (C-H), 1730 (CO), 1600 (C=C).

MS (EI, 80 eV): *m*/*z* (%) = 405 (93) $[M]^+$, 390 (6) $[M - Me]^+$, 348 (23) $[M - t\text{-}Bu]^+$, 230 (60), 136 (67), 91 (100) $[C_7H_7]^+$.

$C_{22}H_{37}NO_4Si$ (405.6)	Calculated	C 65.15	H 8.70	N 3.45
	Found	C 65.29	H 8.44	N 3.17

(1*R*,5*S*,8*R*,9*S*)-2-Benzyl-8-(*tert*-butyldimethylsiloxymethyl)-6,6-dimethyl-3,7-dioxa-2-azabicyclo[3.3.1]nonan-9-ol



14

Procedure:

The protected bicyclic ketone **6** (1.95 g, 4.81 mmol) was dissolved in ethanol (75 ml). The solution was cooled to 0 °C and sodium borohydride (351 mg, 9.63 mmol) was added. The resulting mixture was stirred for 4 h at r.t., after removal of ethanol; water (30 ml) was added to the remaining solid and the mixture was extracted three times with dichloromethane. The organic layer was dried ($MgSO_4$) and concentrated under vacuum to yield 1.90 g (97%) of the pure product **14** as colourless crystals. **Melting point:** 134-135 °C

Optical rotation: $[\alpha]_D^{22}$ = + 56.8 (c = 0.22, $CHCl_3$)

^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.03 (s, 6 H, $SiMe_2$), 0.88 (s, 9 H, *t*-Bu), 1.30, 1.52 (2 s, 3 H each, Me), 1.59 (m_c, 1 H, 5-H), 2.00 (s_{br}, 1 H, OH), 2.85 (t, $J \approx$ 3 Hz, 1 H, 1-H), 3.74 (dd, J = 5.2, 9.2 Hz, 1 H, 8-CH_2), 4.11 (dd, J = 3.0, 9.2 Hz, 1 H, 8-CH_2), 4.07 (d,

J = 14.0 Hz, 1 H, NC$\underline{H}_2$Ph), 4.09 (m_c, 2 H, 4-H), 4.26 (td, *J* = 3.0, 5.2 Hz, 1 H, 8-H), 4.36 (d, *J* = 14.0 Hz, 1 H, NC$\underline{H}_2$Ph), 4.68 (dd, *J* = 3.0, 6.2 Hz, 1 H, 9-H), 7.21-7.39 (m, 5 H, Ph) ppm.

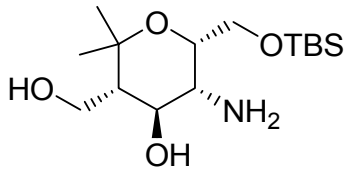
^{13}C NMR ($CDCl_3$, 125 MHz): δ = -5.3 (q, $SiMe_2$), 18.2, 25.8 (s, q, *t*-Bu), 25.9, 29.5 (2 q, Me), 42.8 (d, C-5), 57.6 (t, NC$\underline{H}_2$Ph), 57.8 (d, C-1), 62.8 (t, 8-CH_2), 65.8 (d, C-9), 66.4 (t, C-4), 69.0 (d, C-8), 73.1 (s, C-6), 127.1, 128.0, 128.3, 138.1 (3 d, s, Ph) ppm.

IR (KBr): ν = 3465 cm^{-1} (OH), 3090-2925 (=C-H), 2895-2855 (C-H), 1605 (C=C).

MS (EI, 80 eV): *m*/*z* (%) = 407 (19) $[M]^+$, 392 (3) $[M - Me]^+$, 350 (21) $[M- t\text{-Bu}]^+$, 165 (42), 91 (100) $[C_7H_7]$.

$C_{22}H_{39}NO_4Si$ (407.6)	Calculated	C 64.82	H 9.15	N 3.44
	Found	C 64.42	H 8.96	N 3.22

(3*S*,4*S*,5*R*,6*R*)-5-Amino-6-(*tert*-butyldimethylsiloxymethyl)-3-hydroxymethyl-2,2-dimethyltetrahydro-2*H*-pyran-4-ol



17

Procedure:

A stirred suspension of 0.10 g of Pd (10 %) on charcoal in 5 ml of dry methanol was saturated with hydrogen for 1 h. Then a solution of bicyclic alcohol **14** (0.10 g, 0.25 mmol) in dry methanol (5 ml) was added and the mixture was stirred for 1 d under hydrogen atmosphere at normal pressure and room temperature. Filtration through a pad of celite and removal of the solvent in vacuo yielded the desired product **17** (57 mg, 72%) as pale yellow oil.

Optical rotation: $[\alpha]_D^{22}$ = + 14.1 (c = 0.62, $CHCl_3$)

^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.00 (s, 6 H, $SiMe_2$), 0.81 (s, 9 H, *t*-Bu), 1.03, 1.22 (2 s, 3 H each, Me), 1.72 (t, *J* = 8.7 Hz, 1 H, 3-H), 2.90 (t, *J* = 5.0 Hz, 1 H, 5-H), 3.36 (s_{br}, 1 H, OH), 3.54 (m_c, 1 H, 3-CH_2), 3.58 (m_c, 1 H, 6-CH_2), 3.63 (dd, *J* = 5.0, 8.7 Hz, 1 H, 4-H), 3.65 (m_c, 1 H, 6-CH_2), 3.68 (m_c, 1 H, 3-CH_2), 3.85 (m_c, 1 H, 6-H) ppm.

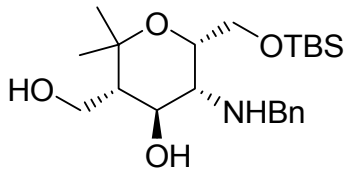
^{13}C NMR ($CDCl_3$, 125 MHz): δ = -5.5 (q, $SiMe_2$), 18.0, 26.0 (s, q, *t*-Bu), 24.1, 26.1 (2 q, Me), 48.0 (d, C-3), 57.0 (d, C-5), 62.2 (t, 3-CH_2), 63.1 (t, 6-CH_2), 69.0 (d, C-6), 74.7 (s, C-2), 75.5 (d, C-4) ppm.

IR (KBr): ν = 3365 cm^{-1} (NH_2), 3300 (OH), 2955-2855 (C-H).

MS (EI, 80 eV): *m/z* (%) = 319 (6) $[M]^+$, 304 (10) $[M - CH_3]^+$, 262 (100) (M - *t*-Bu)$^+$, 203 (11) $[M - C_6H_{17}Si]^+$, 186 (34) $[M - C_6H_{18}OSi]^+$.

$C_{15}H_{33}NO_4Si$ (319.5)	Calculated	C 56.39	H 10.41	N 4.38
	Found	C 56.05	H 10.36	N 4.32

(3*S*,4*S*,5*R*,6*R*)-5-Benzylamino-6-(*tert*-butyldimethylsiloxymethyl)-3-hydroxymethyl-2,2-dimethyltetrahydro-2*H*-pyran-4-ol



20

Procedure:

Diiodoethane (0.23 g, 0.81 mmol) and Samarium (0.13 g, 0.89 mmol) were transferred onto a dried flask under argon. THF (10 ml) was added under argon. After the solution turned blue, the mixture was stirred for 2 h. The bicyclic alcohol **14** (100 mg, 0.25 mmol) was added and the reaction mixture was stirred for 4 h at room temperature, then quenched with satd. $NaHCO_3$ solution (9 ml). The solution was decanted from the residue, and the solvent removed in vacuo to yield 102 mg (qunat.) of the crude product **20** as pale yellow oil.

Optical rotation: $[\alpha]_D^{22}$ = + 11.1 (c = 0.65, $CHCl_3$)

^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.02, 0.04 (2 s, 3 H each, $SiMe_2$), 1.20, 1.33 (2 s, 3 H each, Me), 0.84 (s, 9 H, *t*-Bu), 1.61 (dt, *J* = 1.6, 5.3 Hz, 1 H, 3-H), 2.78 (dd, *J* = 4.0, 4.5 Hz, 1 H, 5-H), 3.61 (dd, *J* = 1.6, 11.3 Hz, 1 H, 3-CH_2), 3.79 (m_c, 1 H, 3-CH_2), 3.78, 3.79 (2 d, *J* = 14.1 Hz, 1 H each, NC$\underline{H_2}$Ph), 3.80 (dd, *J* = 5.3, 12.5 Hz, 1 H, 6-CH_2), 3.91 (d, *J* = 5.3, 12.5 Hz, 1 H, 6-CH_2), 4.02 (m_c, 1 H, 4-H), 4.10 (dt, *J* = 4.0, 5.3 Hz, 1 H, 6-H), 7.23-7.40 (m, 5 H, Ph) ppm.

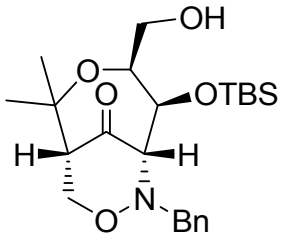
^{13}C NMR ($CDCl_3$, 125 MHz): δ = -5.5, -3.6 (2 q, $SiMe_2$), 18.2, 25.6 (s, q, *t*-Bu), 25.7, 25.8 (2 q, Me), 49.5 (d, C-3), 52.4 (t, 6-CH_2), 60.3 (d, C-5), 63.6 (t, N$\underline{C}H_2$Ph), 63.9 (t, 3-CH_2), 68.9 (d, C-6), 74.0 (d, C-4), 74.1 (s, C-2), 127.4, 128.3, 128.6, 139.1 (3 d, s Ph) ppm.

IR (KBr): ν = 3360 cm^{-1}(NH), 3305 (OH), 2950-2855 (=CH), 2850-2835 (C-H) 1605 (C=C).

MS (EI, 80 eV): *m/z* (%) = 409 (4) $[M]^+$, 394 (5) $[M - CH_3]^+$, 352 (16) [M - *t*-Bu]$^+$, 293 (13) (M - $C_6H_{17}Si]^+$, 276 (12) $[M - C_6H_{18}OSi]^+$, 106 (78) $[C_7H_8N]^+$, 91 (100) $[C_7H_7]^+$.

HRMS ($C_{22}H_{39}NO_4Si$): Calc. 409.26483, Found 409.26574

(1*R*,4*R*,5*S*,6*S*)-7-Benzyl-5-(*tert*-butyldimethylsilyloxy)-4-hydroxymethyl-2,2-dimethyl-3,8-dioxa-7-aza-bicyclo[4.3.1]decan-10-one



Procedure:

To a solution of *syn*-1,2-oxazine **35** (200 mg, 0.37 mmol) in acetonitrile (3 ml) at -30 °C was added $SnCl_4$ (137 µl, 1.12 mmol), and the resulting solution was stirred at this temperature till it warm up to room temperature (6 h). It was then quenched with H_2O (ca. 3 ml). The two layers were separated and the aqueous layer was extracted several times with dichloromethane. The organic layer was dried ($MgSO_4$) and concentrated under vacuum to yield 208 mg of crude product as pale yellow oil. Column chromatography (silica gel, EtOAc:*n*-pentane = 1:5) gave the pure product 89 mg (55%) as colourless highly viscous oil. Starting material was recovered in 20%.

Optical rotation: $[\alpha]_D^{22}$ = + 19.8 (c = 0.48, $CHCl_3$)

^{1}H NMR ($CDCl_3$, 500 MHz): δ = -0.10, - 0.07 (2 s, 3 H each, $SiMe_2$), 0.85 (s, 9 H, *t*-Bu), 1.36, 1.37 (2 s, 3 H each, Me), 1.76 (dd, *J* = 2.0, 9.2 Hz, 1 H, OH), 2.56 (m_c, 1 H, 1-H), 3.35 (dd, *J* = 1.0, 2.4 Hz, 1 H, 5-H), 3.54 (ddd, *J* = 4.5, 9.2, 10.7 Hz, 1 H, 4-CH_2), 3.66 (ddd, *J* = 2.0, 8.4, 10.7 Hz, 1 H, 4-CH_2), 3.95 (d, *J* = 13.6 Hz, 1 H, NC$\underline{H}_2$Ph), 4.09-

4.16 (m, 3 H, 6-H, 9-H), 4.14 (d, *J* = 13.6 Hz, 1 H, NC$\underline{H}_2$Ph), 4.56 (ddd, *J* = 1.0, 4.5, 8.4 Hz, 1 H, 4-H), 7.25-7.34 (m, 5 H, Ph) ppm.

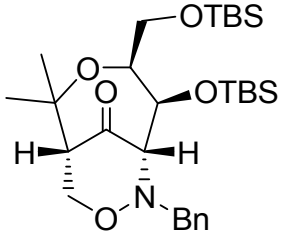
^{13}C NMR ($CDCl_3$, 125 MHz): δ = -4.9, -4.7 (2 q, $SiMe_2$), 17.9, 25.6 (s, q, *t*-Bu), 23.3, 31.6 (2 q, Me), 57.8 (d, C-1), 59.1 (t, N$\underline{C}H_2$Ph), 63.3 (t, 4-C), 67.9 (t, C-9), 68.8 (d, C-6), 74.5 (d, C-4), 75.2 (s, C-2), 75.6 (d, C-5), 127.7, 128.5, 128.6, 136.2 (3 d, s, Ph), 199.9 (s, CO) ppm.

IR (KBr): ν = 3460 cm^{-1} (OH), 3090-3030 (=C-H), 2980-2855 (C-H), 1720 (C=O), 1250 (C-O).

MS (EI, 80 eV): *m*/*z* (%) = 435 (12) $[M]^+$, 378 (7) $[M - t\text{-Bu}]^+$, 321 (9) $[M + H - TBS]^+$, 91 (100) $[C_7H_7]^+$.

HRMS ($C_{23}H_{37}NO_5Si$): Calc. 435.24411, Found 435.24522

(1*R*,4*R*,5*S*,6*S*)-7-Benzyl-5-(*tert*-butyldimethylsilyloxy)-4-(*tert*-butyldimethylsilyloxy)-methyl-2,2-dimethyl-3,8-dioxa-7-aza-bicyclo[4.3.1]decan-10-one



36

Procedure:

To a solution of bicyclic 1,2-oxazinone (200 mg, 0.46 mmol) in dichloromethane (4 ml) was added triethylamine (0.20 ml, 1.38 mmol). The solution was cooled to 0 °C and treated with *tert*-butyldimethylsilyl triflate (0.20 g, 0.69 mmol). The resulting solution was stirred for 1 h at 0 °C, and then satd. NH_4Cl solution (3 ml) was added. The two layers were separated and the aqueous layer was extracted several times with diethyl ether. The organic layer was dried ($MgSO_4$) and concentrated under vacuum to yield 286 mg of the crude product as yellowish crystals, which was chromatographed on silica gel (hexane:EtOAc = 7:1) giving the pure product **36** in 245 mg (97%) as colourless crystals.

Melting point: 88-90 °C

Optical rotation: $[\alpha]_D^{22}$ = + 9.20 (c = 1.0, $CHCl_3$)

^{1}H NMR ($CDCl_3$, 500 MHz): δ = - 0.05, 0.01, 0.08, 0.10 (4 s, 3 H each, $SiMe_2$), 0.86, 0.90 (2 s, 9 H each, *t*-Bu), 1.32, 1.34 (2 s, 3 H each, Me), 2.51 (s_{br}, 1 H, 1-H), 3.42 (d, *J* = 2.5 Hz, 1 H, 5-H), 3.49 (dd, *J* = 6.4, 10.0 Hz, 1 H, 4-CH_2), 3.62 (dd, *J* = 7.6, 10.0 Hz, 1 H, 4-CH_2), 3.92 (d, *J* = 13.7 Hz, 1 H, NC$\underline{H}_2$Ph), 4.08 (dd, *J* = 4.5, 12.6 Hz, 1 H, 9-H_B), 4.10 (m_c, 1 H, 9-H_A), 4.17 (d, *J* = 13.7 Hz, 1 H, NC$\underline{H}_2$Ph), 4.34 (s_{br}, 1 H, 6-H), 4.47 (dd, *J* = 6.4, 7.6 Hz, 1 H, 4-H), 7.30-7.34 (m, 5 H, Ph) ppm.

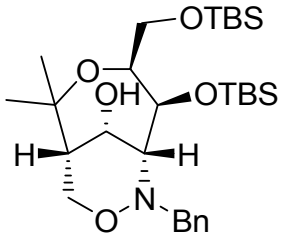
^{13}C NMR ($CDCl_3$, 125 MHz): δ = -5.4, -5.3, -5.0, -4.8 (4 q, $SiMe_2$), 18.1, 18.2, 25.7, 25.9 (2 s, 2 q, *t*-Bu), 22.7, 31.5 (2 q, Me), 57.9 (d, C-1), 58.3 (t, N$\underline{C}H_2$Ph), 62.9 (t, 4-C), 67.6 (d, C-6), 68.4 (t, C-9), 74.2 (s, C-2), 74.4 (d, C-4), 74.9 (d, C-5), 127.6, 128.3, 128.5, 136.6 (3 d, s, Ph), 200.1 (s, CO) ppm.

IR (KBr): ν = 3090-3030 cm^{-1} (=C-H), 2955-2855 (C-H), 1725 (C=O), 1250 (C-O).

MS (EI, 80 eV): *m*/*z* (%) = 549 (9) $[M]^+$, 492 (9) [M - *t*-Bu]$^+$, 417 (5) [M - TBSOH]$^+$, 91 (100) $[C_7H_7]^+$, 73 (55) $[SiMe_3]^+$.

HRMS ($C_{29}H_{51}NO_5Si_2$): Calc. 549.33057, Found 549.33211

(1*S*,4*S*,5*S*,6*S*,10*S*)-7-Benzyl-5-(*tert*-butyldimethylsilyloxy)-4-(*tert*-butyldimethylsilyloxy) methyl-2,2-dimethyl-3,8-dioxa-7-aza-bicyclo[4.3.1]decan-10-ol



Procedure:

Bicyclic 1,2-oxazinone **36** (200 mg, 0.36 mmol) was dissolved in ethanol (7 ml). The solution was cooled to 0 °C and sodium borohydride (27 mg, 0.73 mmol) solution in ethanol (6 ml) was added dropwise to the reaction mixture. The resulting solution was stirred for 4 h at 0 ^oC, after removal of ethanol water (7 ml) was added to the remaining crystals and extracted five times with dichloromethane. The organic layer was dried ($MgSO_4$) and concentrated under vacuum to yield 186 mg (93%) of the pure product as colourless oil.

Optical rotation: $[\alpha]_D^{22}$ = + 40.6 (c = 0.50, $CHCl_3$)

^{1}H NMR ($CDCl_3$, 500 MHz): δ = - 0.03, 0.08, 0.09, 0.11 (4 s, 3 H each, $SiMe_2$), 0.90, 0.96 (2 s, 9 H each, *t*-Bu), 1.27, 1.48 (2 s, 3 H each, Me), 2.12 (dd, *J* = 3.1, 5.6 Hz, 1 H, 1-H), 3.34 (dt, *J* = 2.6, 8.4 Hz, 1 H, 5-H), 3.50 (dd, *J* = 6.1, 9.8 Hz, 1 H, 4-CH_2), 3.65 (dd, *J* = 8.4, 9.8 Hz, 1 H, 4-CH_2), 3.75 (dd, *J* = 3.1, 12.7 Hz, 1 H, 9-H_B), 3.84 (d, *J* = 14.2 Hz, 1 H, NC$\underline{H}_2$Ph), 3.92 (d, *J* = 12.7 Hz, 1 H, 9-H_A), 4.03 (d, *J* = 11.4 Hz, 1 H, OH), 4.16 (d, *J* = 14.2 Hz, 1 H, NC$\underline{H}_2$Ph), 4.35 (dd, *J* = 6.1, 8.4 Hz, 1 H, 4-H), 4.40 (dddd, *J* = 0.5, 5.3, 5.6, 11.4 Hz, 1 H, 10-H), 4.58 (m_c, 1 H, 6-H), 7.31-7.34 (m, 5 H, Ph) ppm.

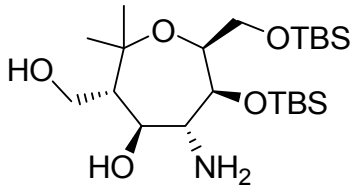
^{13}C NMR ($CDCl_3$, 125 MHz): δ = -5.4, -5.2, -5.0, -4.7 (4 q, $SiMe_2$), 18.1, 18.2, 25.8, 25.9 (2 s, 2 q, *t*-Bu), 24.0, 34.3 (2 q, Me), 47.4 (d, C-1), 58.4 (t, N$\underline{C}H_2$Ph), 62.6 (t, 4-C), 64.6 (d, C-5), 69.5 (t, C-9), 69.6 (d, C-6), 73.6 (d, C-4), 75.0 (d, C-10), 77.8 (s, C-2), 127.3, 127.9, 128.4, 137.5 (3 d, s, Ph) ppm.

IR (KBr): ν = 3525 cm^{-1} (OH), 3085-3030 (=C-H), 2955-2855 (C-H), 1255 (CO).

MS (EI, 80 eV): *m*/*z* (%) = 551 (3) $[M]^+$, 494 (20) [M - *t*-Bu]$^+$, 419 (12) [M - TBSOH]$^+$, 91 (100) $[C_7H_7]^+$, 73 (64) $[SiMe_3]^+$.

HRMS ($C_{29}H_{53}NO_5Si_2$): Calc. 551.34625, Found 551.34733

(3*S*,4*S*,5*R*,6*S,7S*)-5-Amino-6-(*tert*-butyldimethylsilyloxy)-7-(*tert*-butyldimethylsilyloxy-methyl)-3-hydroxymethyl-2,2-dimethyloxepan-4-ol



37

Procedure:

A stirred suspension of Pd on charcoal (0.17 g) in dry methanol (7 ml) was saturated with hydrogen for 1 h. Then a solution of bicyclic alcohol (0.17 g, 0.31 mmol) in dry methanol (3 ml) was added and the mixture was stirred overnight under hydrogen atmosphere at normal pressure at room temperature. Filtration through a pad of celite and removal of the solvent in vacuo yielded the desired product **37** (101 mg, 70%) as colourless crystals.

Melting point: 78-80 °C. **Optical rotation:** $[\alpha]_D^{22}$ = - 25.7 (c = 0.48, $CHCl_3$)

^{1}H NMR (MeOD, 500 MHz): δ = 0.04, 0.06, 0.16, 0.21 (4 s, 3 H each, $SiMe_2$), 0.94, 0.95 (2 s, 9 H, *t*-Bu), 1.24, 1.34 (2 s, 3 H each, Me), 1.96 (ddd, *J* = 3.0, 6.3, 9.3 Hz, 1 H, 3-H), 3.34 (dd, *J* = 5.5, 9.0 Hz, 1 H, 5-H), 3.55 (m_c, 1 H, 3-CH_2), 3.69 (dd, *J* = 6.4, 11.3 Hz, 1 H, 7-CH_2), 3.76-3.82 (m, 3 H, 3-CH_2, 7-CH_2, 6-H), 4.02 (m_c, 1 H, 7-H), 4.04 (dd, *J* = 3.0, 5.5 Hz, 1 H, 4-H).

^{13}C NMR ($CDCl_3$, 125 MHz): δ = - 5.1, -5.0, -4.2, -4.0 (4 q, $SiMe_2$), 19.0, 21.2, 26.3, 26.4 (2 s, 2 q, *t*-Bu), 26.2, 31.6 (2 q, Me), 56.9 (d, C-3), 63.0 (t, 3-CH_2), 63.1 (d, C-5), 63.3 (t, 7-CH_2), 71.1 (d, C-7), 72.5 (d, C-4), 73.3 (d, C-6), 77.0 (s, C-2).

IR (KBr): ν = 3380 cm^{-1} (OH), 2955-2860 (C-H), 1255 (C-O).

MS (EI, 80 eV): *m*/*z* (%) = 464 (12) $[M]^+$, 434 (5) $[M - CHO]^+$, 73 (100) $[SiMe_3]^+$, 57 (11) $[t\text{-Bu}]^+$.

$C_{22}H_{29}NO_5Si_2$ (463.8)	Calculated	C 56.97	H 10.65	N 3.02
	Found	C 56.15	H 10.55	N 2.88