

72.5, 72.4, 71.5, 70.8 ( $\underline{\text{CH}_2\text{Bn}}$ ), 69.6 ( $\text{C}5'_{8\text{S}}$ ), 69.5 ( $\text{C}5'_{8\text{R}}$ ), 47.5 ( $\text{C}2'_{8\text{R}}$ ), 45.5 ( $\text{C}2'_{8\text{S}}$ ), 29.4 ( $\text{C}8'_{8\text{S}}$ ), 26.0 ( $\text{C}6'_{8\text{R}}$ ), 25.6 ( $\text{C}7'_{8\text{R}}$ ), 24.6 ( $\text{C}8'_{8\text{R}}$ ), 23.4 ( $\text{C}7'_{8\text{S}}$ ), 22.9 ( $\text{C}6'_{8\text{S}}$ ), 18.3 ( $8'\text{S}-\text{CH}_3$ ), 17.9 ( $8'\text{R}-\text{CH}_3$ ), 10.8 (thymine- $\underline{\text{CH}_3}$ ). MALDI-TOF  $m/z$ : [ $\text{C}_{28}\text{H}_{32}\text{N}_2\text{O}_5+\text{Na}$ ] $^+$  found 499.221, calcd 499.221.

**(1R,3R,4R,5R,8S)-1-(4,4'-Dimethoxytrityloxymethyl)-8-hydroxyl-5-methyl-3-(thymine-1-yl)-2-oxa-bicyclo[3.2.1]octane and (1R,3R,4R,5S,8S)-1-(4,4'-Dimethoxytrityloxymethyl)-8-hydroxyl-5-methyl-3-(thymine-1-yl)-2-oxa-bicyclo[3.2.1]octane (29).** To a solution of **28** (43 mg, 0.09 mmol) in dry methanol (2 mL) was added 20% Pd(OH) $_2$ /C (90 mg) and ammonium formate (117 mg, 1.8 mmol). The mixture was refluxed for 4 h, then filtered through a pad of Celite and the organic solvent was evaporated to dryness. The residue was coevaporated with dry pyridine twice and dissolved in the same solvent (2 mL). 4,4'-Dimethoxytrityl chloride (96 mg, 0.27 mmol) was added and the resulting mixture was stirred overnight at room temperature. After the solvent was removed, the residue was diluted with  $\text{CH}_2\text{Cl}_2$ , washed with saturated  $\text{NaHCO}_3$ . The organic layer was dried over  $\text{MgSO}_4$ , evaporated and the residue was applied to column chromatography on silica gel (0.5-1 % methanol in  $\text{CH}_2\text{Cl}_2$  containing 1% pyridine, v/v) to give **29** (43 mg, 80 % in two steps) as inseparable diastereomers.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.41 (2.2H, broad, H3), 7.91 (1H, s,  $\text{H}6_{8\text{S}}$ ), 7.86 (1.5H, s,  $\text{H}6_{8\text{R}}$ ), 7.46-6.86 (32.5H, m, aromatic), 5.80 (1H, s,  $\text{H}1'_{8\text{R}}$ ), 5.69 (1.5H, s,  $\text{H}1'_{8\text{S}}$ ), 4.51 (1.5H, d,  $J_{2',3'} = 4.5$  Hz,  $\text{H}3'_{8\text{S}}$ ), 4.47 (1H, d,  $J_{2',3'} = 5.0$  Hz,  $\text{H}3'_{8\text{R}}$ ), 3.81 (15H, s,  $2 \times \text{OCH}_3$ ), 3.41 (1H, d,  $J_{\text{gem}} = 10.5$  Hz,  $\text{H}5'_{8\text{R}}$ ), 3.40 (1.5H, d,  $J_{\text{gem}} = 11.0$  Hz,  $\text{H}5'_{8\text{S}}$ ), 3.29 (1H, d,  $J_{\text{gem}} = 10.5$  Hz,  $\text{H}5''_{8\text{R}}$ ), 3.27 (1.5H, d,  $J_{\text{gem}} = 11.0$  Hz,  $\text{H}5''_{8\text{S}}$ ), 2.47 (1.5H, app t,  $J_{2',3'} = 4.5$  Hz,  $J_{2',8'} = 2.5$  Hz,  $\text{H}2'_{8\text{S}}$ ), 2.31 (1H, d,  $J_{2',3'} = 5.0$  Hz,  $\text{H}2'_{8\text{R}}$ ), 2.29 (2.5H, m,  $\text{H}8'$ ), 1.97 (3H, m,  $\text{H}7'_{8\text{S}}$  and  $\text{H}6'_{8\text{S}}$ ), 1.79 (1H, m,  $\text{H}6'_{8\text{R}}$ ), 1.67 (1H, m,  $\text{H}7'_{8\text{R}}$ ), 1.47 (1.5H, m,  $\text{H}7''_{8\text{S}}$ ), 1.41 (4H, s,  $8'\text{R}$ -thymine- $\underline{\text{CH}_3}$  and  $\text{H}7''_{8\text{R}}$ ), 1.39 (4.5H, s,  $8'\text{S}$ -thymine- $\underline{\text{CH}_3}$ ), 1.36 (1H, m,  $\text{H}6''_{8\text{R}}$ ), 1.29 (6H, m,  $J_{\text{CH}_3, 8'} = 7.5$  Hz,  $\text{H}6''_{8\text{S}}$  and  $8'\text{S}-\underline{\text{CH}_3}$ ), 1.15 (3H, d,  $J_{\text{CH}_3, 8'} = 7.0$  Hz,  $8'\text{R}-\underline{\text{CH}_3}$ ).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  162.9 (C4), 157.7 ( $\text{C}_{\text{isop-OMe}}$ ), 149.0 ( $\text{C}2'_{8\text{S}}$ ), 148.8 ( $\text{C}2'_{8\text{R}}$ ), 143.3, 134.9, 134.8 (DMTr), 134.4, 134.3 (C6), 129.1-124.3, 112.6, 112.3 (aromatic), 108.5, 108.3 (C5), 88.2 ( $\text{C}1'_{8\text{S}}$ ), 85.8 (DMTr- $\text{C}8'\text{R}$ ), 85.6 ( $\text{C}4'_{8\text{S}}$ ), 84.2 ( $\text{C}1'_{8\text{R}}$  and  $\text{C}4'_{8\text{R}}$ ), 83.8 ( $\text{C}4'_{8\text{S}}$ ), 68.3 ( $\text{C}3'_{8\text{R}}$ ), 63.6 ( $\text{C}5'_{8\text{R}}$ ), 63.5