

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 ($8'\text{S-CH}_3$), 17.9 ($8'\text{R-CH}_3$), 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 CH_2Cl_2 , washed with saturated NaHCO_3 . The organic layer was dried over MgSO_4 , evaporated and the residue was applied to column chromatography on silica gel (0.5-1 % methanol in CH_2Cl_2 containing 1% pyridine, v/v) to give **29** (43 mg, 80 % in two steps) as inseparable diastereomers. ^1H NMR (500 MHz, CDCl_3): δ 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, $8'\text{R-thymine-CH}_3$ and $\text{H}7''_{8\text{R}}$), 1.39 (4.5H, s, $8'\text{S-thymine-CH}_3$), 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 $8'\text{S-CH}_3$), 1.15 (3H, d, $J_{\text{CH}_3, 8'} = 7.0$ Hz, $8'\text{R-CH}_3$). ^{13}C NMR (125 MHz, CDCl_3): δ 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