added and stirred overnight at room temperature. The solvent was removed. The residue was chromatographed on silica gel (0.4- 0.9 % methanol in CH_2Cl_2 containing 0.5 % pyridine, v/v) to obtain **25** (59 mg, 75.3 % in tow steps) as white foam. ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.38 (1H, s, H3), 7.87 (1H, s, H6), 7.45-6.85 (13H, m, aromatic), 5.77 (1H, s, H1′), 4.46 (1H, t, $J_{3',OH}$ = 4.2 Hz, $J_{2',3'}$ = 4.8 Hz, H3′), 3.81 (6H, s, 2xOC<u>H</u>$_3$), 3.36 (1H, d, J_{gem} = 10.8 Hz, H5′), 3.27 (1H, d, J_{gem} = 10.8 Hz, H5″), 2.55 (1H, s, $J_{2',3'}$ = 4.8 Hz, H2′), 1.98 (1H, m, H8′), 1.78 (3H, m, H6′, H8″ and 3′-OH), 1.67 (2H, m, H7′ and H7″), 1.39 (3H, s, thymine-C<u>H</u>$_3$), 1.37 (1H, m, H6″). ^{13}C-NMR (125MHz, $CDCl_3$): δ 164.1 (C4), 158.7 (C_{ispo}-OMe), 150.2 (C2), 144.4 (DMTr), 135.7 (C6), 135.5, 135.4 (DMTr), 130.1-127.1, 113.3 (aromatic), 109.5 (C5), 87.7 (C1′), 86.7 (DMTr-<u>C</u>), 85.7 (C4′), 67.5 (C3′), 64.8 (C5′), 55.2 (2xOMe), 45.4 (C2′), 26.7 (C6′), 20.1 (C8′), 17.6 (C7′), 12.0 (CH_3-thymine). MALDI-TOF *m/z*: $[C_{34}H_{36}N_2O_7+Na]^+$ found 607.243, calcd 607.241.

(1*R*,3*R*,4*R*,5*R*,8*S*)-8-benzyloxy-1-benzyloxymethyl-5-methyl-3-(thymin-1-yl)-2-oxa-bicyclo[3.2.1]octane and (1*R*,3*R*,4*R*,5*S*,8*S*)-8-benzyloxy-1-benzyloxymethyl-5-methyl-3-(thymin-1-yl)-2-oxa-bicyclo[3.2.1]octane (28). 27 (61 mg, 0.105 mmol) was dissolved in dry toluene (3 mL), purged with N_2 for 20 min. AIBN (9 mg, 0.032 mmol) and Bu_3SnH (42 μL, 0.158 mmol) were added to the solution and the mixture was refluxed for 4 h. The solvent was removed and the residue was purified by column chromatography on silica gel (20-35 % EtOAc in cyclohexane, v/v) to give **28** (43 mg, 87 %) as inseparable diastereomers (8′*R* : 8′*S* = 4: 5). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.25 (1.8H, broad, N<u>H</u>), 8.10 (1.8H, s, H6), 7.39-7.27 (18H, m, aromatic), 5.85 (0.8H, s, H1′$_{8'R}$), 5.67 (1H, s, H1′$_{8'S}$), 4.69-4.47 (7.2H, m, C<u>H</u>$_2$Bn), 4.20 (0.8H, d, $J_{2',3'}$ = 5.0 Hz, H3′$_{8'R}$), 4.13 (1H, d, $J_{2',3'}$= 4.5 Hz, H3′$_{8'S}$), 3.72 (1.8H, d, J_{gem} = 10.5 Hz, H5′), 3.58 (1.8H, 2×d, J_{gem} = 10.5 Hz, H5″), 2.52 (1H, app t, $J_{2',3'}$ = 4.5 Hz, $J_{2',8'}$ = 2.5 Hz, H2′$_{8'S}$), 2.33 (0.8H, d, $J_{2',3'}$ = 4.5 Hz, H2′$_{8'R}$), 2.28 (1.8H, m, H8′), 2.01 (1H, m, H7′$_{8'S}$), 1.86 (0.8H, m, H6′$_{8'R}$), 1.69 (0.8H, m, H7′$_{8'R}$), 1.47 (7.4H, m, H6′$_{8'S}$, H7″$_{8'S}$ and thymine-C<u>H</u>$_3$), 1.37-1.32 (5.6H, m, $J_{CH3,\ 8'S}$ = 7.5 Hz, H7″$_{8'R}$, H6″$_{8'R}$, H6″$_{8'S}$ and 8′*S*-CH_3), 1.14 (2.4H, d, $J_{CH3,\ 8'R}$ = 7.0 Hz, 8′*R*-C<u>H</u>$_3$). ^{13}C NMR (125 MHz, $CDCl_3$): δ 162.9, 162.8 (C4), 149.0, 148.9 (C2), 136.7, 136.5, 135.4 (C_{ipso}-Bn), 135.6, 135.5 (C6), 127.6-126.4 (aromatic), 108.1, 108.0 (C5), 88.1 (C1′$_{8'S}$), 83.8 (C4′$_{8'S}$), 83.7 (C1′$_{8'R}$), 83.6 (C4′$_{8'R}$), 73.4 (C3′$_{8'S}$), 72.6 (C3′$_{8'R}$),