

at room temperature. The reaction was neutralized with 1N HCl solution at 0°C and extracted with DCM twice. The combined organic layers were dried over MgSO₄ and concentrated. The residue was chromatographed on silica gel (15-21% acetone in cyclohexane, v/v) to obtain **34** (411 mg, 91.5 %) as white foam. ¹H NMR (500 MHz, CDCl₃): δ 8.52 (1H, bs, H3), 7.91 (1H, d, $J_{6, \text{CH}_3} = 1.0$ Hz, H6), 7.35-7.25 (10H, m, aromatic), 6.06 (1H, s, H1'), 4.75 (1H, d, $J_{\text{gem}} = 11.5$ Hz, $\underline{\text{CH}_2\text{Bn}}$), 4.57 (1H, d, $J_{\text{gem}} = 11.5$ Hz, $\underline{\text{CH}_2\text{Bn}}$), 4.53 (1H, d, $J_{\text{gem}} = 11.5$ Hz, $\underline{\text{CH}_2\text{Bn}}$), 4.51 (1H, d, $J_{\text{gem}} = 11.5$ Hz, $\underline{\text{CH}_2\text{Bn}}$), 4.32 (1H, d, $J_{2', 3'} = 3.0$ Hz, H2'), 4.02 (2H, dd, $J = 2.5$ Hz and 7.0 Hz, H7' and H7''), 3.93 (1H, d, $J_{2', 3'} = 3.0$ Hz, H3'), 3.76 (1H, d, $J_{\text{gem}} = 11.0$ Hz, H5'), 3.60 (1H, d, $J_{\text{gem}} = 11.0$ Hz, H5''), 2.28 (1H, m, H6'), 1.40 (3H, d, $J_{6, \text{CH}_3} = 1.0$ Hz, thymine- $\underline{\text{CH}_3}$), 1.35 (1H, d, $J = 12.5$ Hz, H6''). ¹³C NMR (125 MHz, CDCl₃): δ 163.8 (C4), 149.9 (C2), 137.4, 137.2 (*C_{ipso}*-Bn), 135.6 (C6), 129.1-127.7 (aromatic), 110.0 (C5), 85.9 (C1'), 83.9 (C4'), 75.8 (C2'), 73.6, 71.8 ($\underline{\text{CH}_2\text{Bn}}$), 71.4 (C3'), 69.9 (C5'), 60.7 (C7'), 27.8 (C6'), 11.9 (thymine- $\underline{\text{CH}_3}$).

5'-O-(4,4'-Dimethoxytrityl)-2'-O,4'-C-ethylene-thymidine (36).

To a solution of **34** (208 mg, 0.448 mmol) in dry methanol (20 mL) was added 20% Pd(OH)₂/C (224 mg) and ammonium formate (565 mg, 8.95 mmol) and reflux for 2.5h. The suspension was filtered through a pad of Celite and filtrate was evaporated. The residue was co-evaporated twice with dry pyridine and dissolved in the same (6 mL). 4, 4'-Dimethoxytrityl chloride (321 mg, 0.948 mmol) was added and stirred overnight at room temperature. The solvent was removed. The residue was chromatographed on silica gel (0-0.3 % methanol in CH₂Cl₂ containing 1% pyridine, v/v) to obtain **36** (163 mg, 62.1 % in tow steps) as white foam. ¹H NMR (500 MHz, DMSO-*d*₆): δ 11.39 (1H, s, H3), 7.68 (1H, s, H6), 7.43-6.90 (13H, m, aromatic), 5.89 (1H, s, H1'), 5.58 (1H, d, $J_{3', \text{OH}} = 5.5$ Hz, 3'-OH), 4.12 (1H, dd, $J_{3', \text{OH}} = 5.5$ Hz, $J_{2', 3'} = 3.0$ Hz, H3'), 4.06 (1H, d, $J_{2', 3'} = 3.0$ Hz, H2'), 3.81 (2H, m, H7' and H7''), 3.74 (6H, s, 2xO $\underline{\text{CH}_3}$), 3.20 (1H, d, $J_{\text{gem}} = 11.0$ Hz, H5'), 3.17 (1H, d, $J_{\text{gem}} = 11.0$ Hz, H5''), 1.96 (1H, m, H6'), 1.34 (1H, m, H7''), 1.17 (3H, s, thymine- $\underline{\text{CH}_3}$).