(*tert*-Butyldimethylsilyloxy)methyl Chloride: Synthesis and Use as *N*-protecting Group in Pyrimidinones

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A new reagent, (*tert*-butyldimethylsilyloxy)methyl chloride (**3**) has been synthesized by sulfuryl chloride cleavage of (*tert*-butyldimethylsilyloxy)methyl ethyl sulfide (**2**). The latter was prepared from *tert*-butyldimethylsilyl chloride and ethylthiomethanol. The chloromethyl ether **3** has been used for the protection of the NH functionality in 5-halo-2(1*H*)-pyrimidinones and for the protection of the N-1 in N-3 alkylation of thymine.

In our studies on nucleophilic addition to 2-pyrimidinones[1] we needed a blocking group for the NH functionality. We also were interested in developing a protecting group for N-1 in uracils in order to effect selective alkylation on N-3. Relatively few reagents have been used for the protection of the NH functionality in pyrimidinones,[2] and none of these were suitable for our purpose. We therefore turned to organosilicon compounds, which have been widely used as protecting groups in organic synthesis. The *tert*-butyldimethylsilyl group (TBDMS) has proven to be very useful for the protection of alcohols.[3] The α-halo ether functionality is incorporated in many protecting reagents because of its reactivity.[4] We felt that combining the properties of the TBDMS group and the reactivity of α-halo ethers would give a reactive reagent for ready introduction of the (*tert*-butyldimethylsilyloxy)methyl protecting group. The protecting group should have good stability properties and be removable under mild and selective reaction conditions.

In previous papers we have published methods for the synthesis of α-chloro ethers by cleavage of *O*/*S*-acetals with sulfuryl chloride.[5] Accordingly, our concept for the synthesis of (*tert*-butyldimethylsilyloxy)methyl chloride (**3**) was to cleave a TBDMS-protected hemithioacetal with sulfuryl chloride (Scheme 1).

The hemithioacetal **1**[6] was made by hydroxymethylation of ethanethiol with paraformaldehyde employing base catalysis (Scheme 1). Crude **1** was sufficiently pure to be used directly in the silylating step. The reaction of **1** with TBDMS–Cl gave the TBDMS-protected *O*/*S*-acetal **2**. By carrying out the silylation in dichloromethane in the presence of triethylamine and 4-(*N*,*N*-dimethylamino)pyridine (DMAP)[7] instead of DMF and imidazole,[8] a smaller excess of the rather expensive TBDMS–Cl was required. The *O*/*S*-acetal **2** was easily and chemoselectively cleaved by sulfuryl chloride in dichloromethane at 0 °C to give the chloromethyl reagent **3**.

The co-product from the cleavage reaction is ethanesulfenyl chloride (**4**) (b.p. 6–8 °C/13 mm Hg),[9] which was conveniently removed together with the solvent. The chloromethyl reagent **3** could be distilled, but was best used as the crude product due to partial decomposition during distillation.

Reaction of **3** with *O*-silylated or *O*-stannylated pyrimidinones gave selective formation of the *N*-alkylated isomer. In the case of 5-halo-2-pyrimidinones (**5**), however, a better yield of the *N*-alkylated product was obtained using basic conditions (NEt_3,CH_2Cl_2), although some *O*-alkylated isomer (15–20 %) was formed. The *O*-

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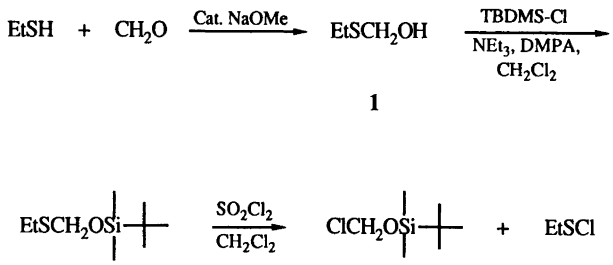
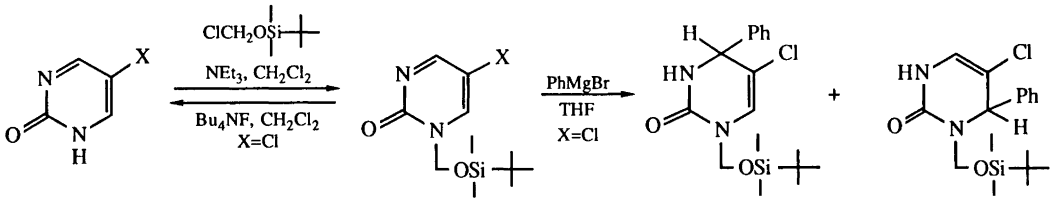


Scheme 1.

isomer was separated from the *N*-isomer simply by trituration with pentane, which dissolved the former.[10]

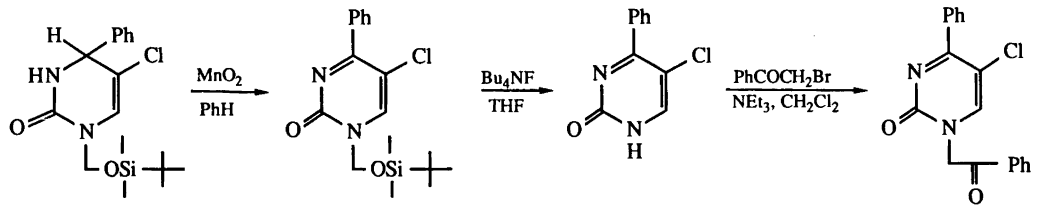
In order to remove the protecting group the pyrimidinone **6a** was treated with Bu_4NF on SiO_2[11] in dichloromethane. After 4 h at ambient temperature the pyrimidinone **5a** was regenerated in 74 % yield.

Alkyl and aryl groups can be introduced into the 4-position of N-1 substituted 2-pyrimidinones by means of organometallic reagents.[12] This procedure implies that the N-1 substituent must be inert to the organometallic reagent. In cases where the organometallic reagent would interfere with the N-1 substituent, the organometallic reaction must precede N-1 alkylation, in which case a suitable protecting group is required. Compound **6** can be regarded as protected in this manner. Treatment of **6** with PhMgBr in THF at room temperature gave a ca. 1:1 mixture of the regioisomers **7a** and **7b** without any significant deprotection (Scheme 2). The dihydropyrimidinone **7a** was oxidized by MnO_2[12] in benzene to the aromatic compound **8**. Removal of the protecting



5
a : X=Cl
b : X=Br

6 **7a** **7b**



7a **8** **9** **10**

Scheme 2.

group with Bu_4NF in THF gave the pyrimidinone **9**, which could be alkylated with phenacyl bromide to give the N-1 alkyloxo pyrimidinone **10**.

Our new protecting group has also been applied in uracil chemistry. Reaction of the silylated thymine **11**[13] with the chloromethyl ether **3** gave the N-1 alkylated thymine **12** (Scheme 3). The latter was further alkylated at N-3. Our best conditions for the alkylation involved the use of NaH in DMF (a number of other methods gave less satisfactory results).

The silyloxymethyl substituent in the dialkylated product **13** was removed by Bu_4NF in THF to give the N-3 alkylated thymine **14**. We thus believe that the (*tert*-butyldimethylsilyloxy) methyl group is a potentially useful protecting group for N-1 in the syntheses of N-3 alkylated uracils.

Experimental

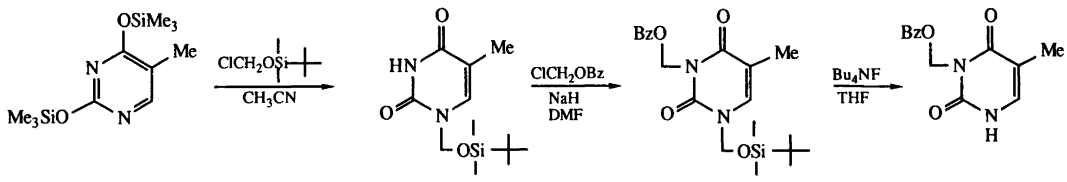
The ^1H NMR spectra were recorded at 60 MHz or 300 MHz, and ^{13}C NMR spectra at 75 MHz. The mass spectra under electron impact conditions were recorded at 70 eV ionizing energy. Isobutane was used for chemical ionizing mass spectrometry (C.I.); the spectra are presented as *m/z* (% rel. int.).

Ethylthiomethanol (**1**).[6] A mixture of ethanethiol (14.8 ml, 0.2 mol), paraformaldehyde (6.0 g, 0.2 mol) and a 30 % solution of sodium methoxide in methanol (0.06 ml) was heated at 40 °C for 30 min and cooled; yield 17.3 g (94 %). ^1H NMR ($CDCl_3$): δ 1.32 (t, CH_3, *J* 7 Hz), 2.43 (OH), 2.73 (q, CH_2S, *J* 7 Hz), 4.75 (d, CH_2OH, *J* 6 Hz). MS: 92 (9, *M*), 77 (2), 75 (2), 64 (6), 62 (100), 58 (16), 47 (73).

*(*tert*-Butyldimethylsilyloxy)methyl ethyl sulfide* (**2**). *tert*-Butyldimethylchlorosilane (8.31 g, 55 mmol), 4-(*N,N*-dimethylamino)pyridine (244 mg, 2 mmol) and triethylamine (8.35 ml, 60 mmol) were added to a solution of ethylthiomethanol (4.60 g, 50 mmol) in dry dichloromethane (50 ml). The mixture was stirred at ambient temperature under nitrogen for 4 h, diluted with dichloromethane and washed successively with water (×2) and saturated aqueous ammonium chloride (×2). The dried ($MgSO_4$) solution was evaporated; yield 8.72 g (84 %). ^1H NMR ($CDCl_3$): δ 0.12 ($SiCH_3$), 0.91 (Bu^t), 1.30 (t, CH_3, *J* 7 Hz), 2.67 (q, CH_2S, *J* 7 Hz), 4.81 (CH_2O). ^{13}C NMR ($CDCl_3$): δ −5.6 ($SiCH_3$), 15.0 (CH_3), 18.2 (C in Bu^t), 24.6 (CH_2S), 25.8 (CH_3 in Bu^t), 66.0 (CH_2O). MS(C.I.): 207 (10, *M*), 191 (3), 149 (62), 145 (100), 133 (64), 119 (15), 115 (9), 89 (35), 81 (11), 75 (45).

*(*tert*-Butyldimethylsilyloxy)methyl chloride* (**3**). Sulfuryl chloride (0.08 ml, 1.0 mmol) in dry dichloromethane (1 ml) was added dropwise at 0 °C to a solution of (*tert*-butyldimethylsilyloxy)-methyl ethyl sulfide (207 mg, 1.0 mmol) in dry dichloromethane (2 ml). The mixture was stirred under nitrogen for 30 min at 0 °C and for 10 min at ambient temperature before the solvent and ethanesulfenyl chloride were evaporated off at reduced pressure; yield 175 mg (97 %). Anal. $C_7H_{17}ClOSi$: C,H. ^1H NMR ($CDCl_3$): δ 0.20 ($SiCH_3$), 0.91 (Bu^t), 5.61 (CH_2). ^{13}C NMR ($CDCl_3$): δ −5.3 ($SiCH_3$), 19.0 (C in Bu^t), 25.4 (CH_3 in Bu^t), 76.3 (CH_2). MS(C.I.): 145 (54), 135 (12), 123 (18), 115 (100), 109 (17), 95 (17), 93 (48), 89 (96), 73 (51).

*1-(*tert*-Butyldimethylsilyloxy)methyl-5-chloro-2(1H)-pyrimidinone* (**6a**). Triethylamine (0.28 ml, 2 mmol) was added to a suspension of 5-chloro-2(1*H*)-pyrimidinone (262 mg, 2 mmol) in dry dichloromethane (4 ml). The solution was cooled to −78 °C. A solution of (*tert*-butyldi-



11 **12** **13** **14**

Scheme 3.

methylsilyloxy)methyl chloride (360 mg, 2 mmol) in dry dichloromethane was added dropwise during 30 min. The mixture was stirred for 23 h under nitrogen while reaching ambient temperature, diluted with dichloromethane, washed with saturated aqueous sodium chloride (×2), dried ($MgSO_4$) and evaporated. Pentane was added to the residue, and the solid was collected and washed with ether; yield 294 mg (53%). Anal. $C_{11}H_{19}ClN_2O_2Si$: C,H. ^1H NMR ($CDCl_3$): δ 0.18 (CH_3), 0.94 (Bu^t), 5.39 (CH_2), 7.94 and 8.56 (d, H-4 and H-6, respectively, *J* 3 Hz). ^{13}C NMR ($CDCl_3$): δ −5.4 (CH_3), 18.1 (C in Bu^t), 25.6 (CH_3 in Bu^t), 73.5 (CH_2), 111.2 (C-5), 141.4 (C-6), 153.7 (C-2), 165.4 (C-4). MS (C.I.): 277/275 (5/15, *M*+1), 259 (2), 245 (21), 229 (12), 219 (14), 217 (11), 187 (25), 145 (44), 115 (22), 89 (100).

*5-Bromo-1-(*tert*-butyldimethylsilyloxy)methyl-2(1H)-pyrimidinone* (**6b**). **6b** was prepared by the same method as **6a** above, but the reaction was carried out at 0°C; yield 43%. Anal. $C_{11}H_{19}BrN_2O_2Si$: C,H. ^1H NMR ($CDCl_3$): δ 0.18 (CH_3), 0.95 (Bu^t), 5.40 (CH_2), 8.02 and 8.60 (d, H-4 and H-6, respectively, *J* 3 Hz). ^{13}C NMR ($CDCl_3$): δ −5.4 (CH_3), 18.0 (C in Bu^t), 25.4 (CH_3 in Bu^t), 73.4 (CH_2), 112.3 (C-5), 143.7 (C-6), 153.3 (C-2), 166.5 (C-4). MS(C.I.): 321/319 (63/61, *M*+1), 289 (17), 273 (2), 261 (18), 241 (6), 231 (39), 153 (3), 145 (58), 89 (100).

5-Chloro-2(1H)-pyrimidinone (**5a**). Tetrabutylammonium fluoride on silica gel (909 mg, 1.0 mmol, 1.1 mmol per g) was added to a solution of 1-(*tert*-butyldimethylsilyloxy)methyl-5-chloro-2(1*H*)-pyrimidinone (136 mg, 0.5 mmol) in dry dichloromethane (2 ml). The mixture was stirred at ambient temperature under nitrogen for 4 h before the solvent was evaporated off. The residue was stirred for 2 h in 1.5 ml of 1 M NaOH and the mixture filtered. The filtrate was adjusted to pH 5 with 1 M HCl and kept in a refrigerator overnight. The solid was collected and washed with ether; yield 48 mg (74%). The product was identical with an authentic sample.

*1-(*tert*-Butyldimethylsilyloxy)methyl-5-chloro-3,4-dihydro-4-phenyl-2(1H)-pyrimidinone* (**7a**) and 1-(tert-*butyldimethylsilyloxy)methyl-5-chloro-3,6-dihydro-6-phenyl-2(1H)-pyrimidinone* (**7b**). A 1 M solution of phenylmagnesium bromide (2.1 ml) in THF was added dropwise at 0°C to a solution of 1-(tert-butyldimethylsilyloxy)-methyl-5-chloro-2(1*H*)-pyrimidinone (413 mg, 1.5 mmol) in dry THF (5 ml). The mixture was stirred at ambient temperature under nitrogen for 20 h and diluted with dichloromethane. The solution was washed with saturated aqueous ammonium chloride (×2) and saturated aqueous sodium hydrogen carbonate (×2), dried ($MgSO_4$) and evaporated. The isomers were separated by flash chromatography using ethyl acetate/hexane (2:5);

7a: (Rf 0.34); yield 172 mg (33%). Anal. $C_{17}H_{25}ClN_2O_2Si$: C,H. ^1H NMR ($CDCl_3$): δ 0.12 (CH_3), 0.91 (Bu^t), 4.99 and 5.05 (d, CH_2, *J* 10 Hz), 5.03 (d, H-4, *J* 2 Hz), 5.89 (NH), 6.41 (H-6), 7.3–7.4 (Ph). ^{13}C NMR ($CDCl_3$): δ −5.3 (CH_3), 17.9 (C in Bu^t), 25.6 (CH_3 in Bu^t), 61.4 (C-4), 70.2 (CH_2), 108.9 (C-5), 124.8 (C-6), 127.0, 128.6 and 128.7 (CH in Ph), 140.5 (C in Ph), 151.2 (C-2). MS: 337 (3), 295 (100), 265 (9), 222 (24), 149 (27), 129 (33), 115 (20), 93 (27), 89 (15), 75 (93).

7b: (Rf 0.29); yield 118 mg (22%). Anal. $C_{17}H_{25}ClN_2O_2Si$: C,H. ^1H NMR ($CDCl_3$): δ 0.02 (CH_3), 0.88 (Bu^t), 4.19 and 5.49 (d, CH_2, *J* 10 Hz), 5.18 (H-6), 6.25 (d, H-4, *J* 2 Hz), 7.3–7.4 (Ph), 7.88 (NH). ^{13}C NMR ($CDCl_3$): δ −5.2 (CH_3), 18.0 (C in Bu^t), 25.6 (CH_3 in Bu^t), 62.3 (C-6), 68.3 (CH_2), 108.2 (C-5), 121.3 (C-4), 127.6, 128.6 and 128.9 (CH in Ph), 137.8. MS: 337 (3), 295 (100), 265 (29), 222 (9), 192 (17), 187 (6), 149 (28), 120 (11), 115 (15), 100 (27), 75 (90).

*1-(*tert*-Butyldimethylsilyloxy)methyl-5-chloro-4-phenyl-2(1H)-pyrimidinone* (**8**). Activated manganese dioxide[13] (2.0 g) was added to a solution of 1-(*tert*-butyldimethylsilyloxy)methyl-5-chloro-3,4-dihydro-4-phenyl-2(1*H*)-pyrimidinone (186 mg, 0.53 mmol) in benzene (20 ml). The mixture was stirred at ambient temperature under nitrogen for 25 h before the solid was filtered off and washed with benzene. The solution was evaporated and the product was purified by flash chromatography using ethyl acetate/hexane (1:1); yield 165 mg (89%). Anal. $C_{17}H_{23}ClN_2O_2Si$: C,H. ^1H NMR ($CDCl_3$): δ 0.21 (CH_3), 0.96 (Bu^t), 5.47 (CH_2), 7.4–7.9 (Ph), 8.05 (H-6). ^{13}C NMR ($CDCl_3$): δ −5.4 (CH_3), 18.0 (C

in Bu^t), 25.5 (CH_3 in Bu^t), 73.1 (CH_2), 112.5 (C-5), 127.9, 129.3 and 130.9 (CH in Ph), 135.3 (C in Ph), 142.8 (C-6), 153.5 (C-2), 170.9 (C-4) MS(C.I.): 353/351 (33/100, *M*+1), 321 (5), 317 (2), 293 (10), 275 (3), 263 (10), 221 (18), 207 (6), 133 (6), 89 (14).

5-Chloro-4-phenyl-2(1H)-pyrimidinone (**9**). A 0.50 M solution of tetrabutylammonium fluoride in dry THF (1 ml) was added to 1-(*tert*-butyldimethylsilyloxy)methyl-5-chloro-4-phenyl-2(1*H*)-pyrimidinone (117 mg, 0.33 mmol). The mixture was stirred at ambient temperature under nitrogen for 30 min; water was added, and the mixture was adjusted to pH 4 with acetic acid and extracted with chloroform (×4). The chloroform solution was washed with saturated aqueous sodium chloride, dried ($MgSO_4$) and evaporated. The crude product was purified by flash chromatography using triethylamine/methanol/chloroform (1:10:50); yield 48 mg (70%). Anal. $C_{10}H_7ClN_2O$: C,H. ^{1}H NMR (DMSO-d_6): δ 7.5–7.7 (Ph), 8.49 (H-6). ^{13}C NMR (DMSO-d_6): δ 112.8 (C-5), 128.2, 128.8 and 130.4 (CH in Ph), 134.8 (C in Ph), 155.2 (C-6), 158.6 (C-2), 165.4 (C-4). MS(C.I.): 209/207 (33/100, *M*+1), 178 (1), 173 (4), 171 (2), 144 (1), 102 (23), 100 (5), 86 (5).

5-Chloro-1-phenacyl-4-phenyl-2(1H)-pyrimidinone (**10**). Phenacyl bromide (78 mg, 0.4 mmol) in dry dichloromethane (1 ml) was added dropwise during 15 min at 0 °C to a mixture of triethylamine (0.05 ml, 0.4 mmol) and 5-chloro-4-phenyl-2(1*H*)-pyrimidinone (80 mg, 0.4 mmol) in dry dichloromethane (2 ml). The mixture was stirred at ambient temperature under nitrogen for 3 h, diluted with dichloromethane and washed with saturated aqueous sodium chloride (×2). The dried ($MgSO_4$) solution was evaporated and the crude product was purified by flash chromatography using methanol/chloroform (1:15); yield 69 mg (55%). Anal. $C_{18}H_{13}ClN_2O_2$: C,H. ^{1}H NMR ($CDCl_3$,CD_3OD): δ 5.52 (CH_2), 7.5–8.0 (Ph), 8.04 (H-6). ^{13}C NMR ($CDCl_3$,CD_3OD): δ 55.2 (CH_2), 110.3 (C-5), 127.8, 128.7, 128.9, 130.9 and 134.2 (CH in Ph), 133.8 and 134.9 (C in Ph), 148.1 (C-6), 155.8 (C-2), 171.6 (C-4), 191.1 (CO). MS(C.I.): 327/325 (39/100, *M*+1), 248 (1), 221 (1), 207 (12), 189 (1), 121 (41), 105 (11), 85 (2), 71 (2).

*1-(*tert*-Butyldimethylsilyloxy)methylthymine* (**12**). A solution of thymine (2.50 g, 19.8 mmol) and ammonium sulfate (20 mg) in hexamethyldisilazane (100 ml) was heated at reflux under nitrogen for 4 h, the solution evaporated and the residue dissolved in dry acetonitrile (50 ml). A solution of (*tert*-butyldimethylsilyloxy)methyl chloride (2.38 g, 13.2 mmol) in dry acetonitrile (20 ml) was added dropwise at 0 °C under nitrogen. The mixture was stirred for 20 h while reaching ambient temperature; the solvent was then evaporated and the product isolated by flash chromatography using ethyl acetate/hexane (3:4); yield 1.91 g (54%). M.p. 155–157 °C. Anal. $C_{12}H_{22}N_2O_3Si$: C,H. ^{1}H NMR ($CDCl_3$): δ 0.12 ($SiCH_3$), 0.88 (Bu^t), 1.93 (d, CH_3, *J* 1 Hz), 5.24 (CH_2), 7.15 (q, H-6, *J* 1 Hz), 8.77 (NH). ^{13}C NMR ($CDCl_3$): δ −5.4 ($SiCH_3$), 12.2 (CH_3), 17.9 (C in Bu^t), 25.5 (CH_3 in Bu^t), 70.8 (CH_2), 111.1 (C-5), 138.6 (C-6), 150.3 (C-2), 164.1 (C-4). MS (C.I.): 271 (100, *M*+1), 255 (4), 241 (4), 225 (2), 213 (65), 183 (41), 139 (60), 133 (3), 113 (9), 89 (16).

*3-Benzyloxymethyl-1-(*tert*-butyldimethylsilyloxy)-methylthymine* (**13**). Benzyl chloromethyl ether (0.08 ml, 0.6 mmol) was added to a solution of 1-(*tert*-butyldimethylsilyloxy)methylthymine (135 mg, 0.5 mmol) and an 80% oily suspension of sodium hydride (18 mg, 0.6 mmol) in dry DMF (3 ml). The mixture was stirred at ambient temperature under nitrogen for 4 h before the solvent was distilled off under reduced pressure. The residue was dissolved in hexane, washed with saturated aqueous sodium chloride (×4), dried ($MgSO_4$) and evaporated. The crude product was purified by flash chromatography using ethyl acetate/hexane (2:5); yield 94 mg (48%). ^{1}H NMR ($CDCl_3$): δ 0.15 ($SiCH_3$), 0.19 (Bu^t), 1.95 (d, CH_3, *J* 1 Hz), 4.63 (*N* 3-CH_2), 5.26 (*N* 1-CH_2), 5.51 (OCH_2), 7.13 (q, H-6, *J* 1 Hz), 7.3–7.5 (Ph). ^{13}C NMR ($CDCl_3$): δ −5.4 ($SiCH_3$), 12.9 (CH_3), 17.9 (C in Bu^t), 25.5 (CH_3 in Bu^t), 70.4, 71.6 and 71.9 (CH_2), 110.3 (C-5), 127.5 and 128.1 (CH in Ph), 137.4 (C-6), 137.8 (C in Ph), 150.9 (C-2), 163.6 (C-4). MS(C.I.): 391 (100, *M*+1), 374 (15), 358 (14), 345 (5), 333 (88), 302 (79), 273 (19), 259 (15), 91 (36).

3-Benzyloxymethylthymine (**14**). A 0.62 M solution of tetrabutylammonium fluoride in dry THF (2 ml) was added to 3-benzyloxymethyl-1-

(*tert*-butyldimethylsilyloxy)methylthymine (230 mg, 0.6 mmol) and the mixture stirred at ambient temperature under nitrogen for 75 min. Water was added, and the pH was adjusted to 4.5 with acetic acid and the mixture extracted with chloroform (×4). The chloroform solution was washed with saturated aqueous sodium chloride (×3), dried ($MgSO_4$) and evaporated. The crude product was purified by flash chromatography using triethylamine/methanol/chloroform (1:4:100); yield 93 mg (63%). M.p. 145–147°C. Anal. $C_{13}H_{14}N_2O_3$: C,H. ^{1}H NMR (DMSO-d_6): δ 2.09 (d, CH_3, *J* 1 Hz), 4.90 (NCH_2), 5.70 (OCH_2), 7.21 (d of q, H-6, *J* 6 Hz, *J* 1 Hz), 7.4–7.6 (Ph), 10.33 (NH). ^{13}C NMR: (DMSO-d_6) δ 12.9 (CH_3), 70.0 and 72.3 (CH_2), 110.2 (C-5), 127.8 and 128.3 (CH in Ph), 135.4 (C-6), 137.8 (C in Ph), 153.3 (C-2), 161.1 (C-4). MS(C.I.): 247 (8, *M*+1), 229 (4), 217 (18), 167 (5), 149 (13), 144 (5), 140 (22), 127 (11), 102 (100), 91 (48).

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