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for the Preparation
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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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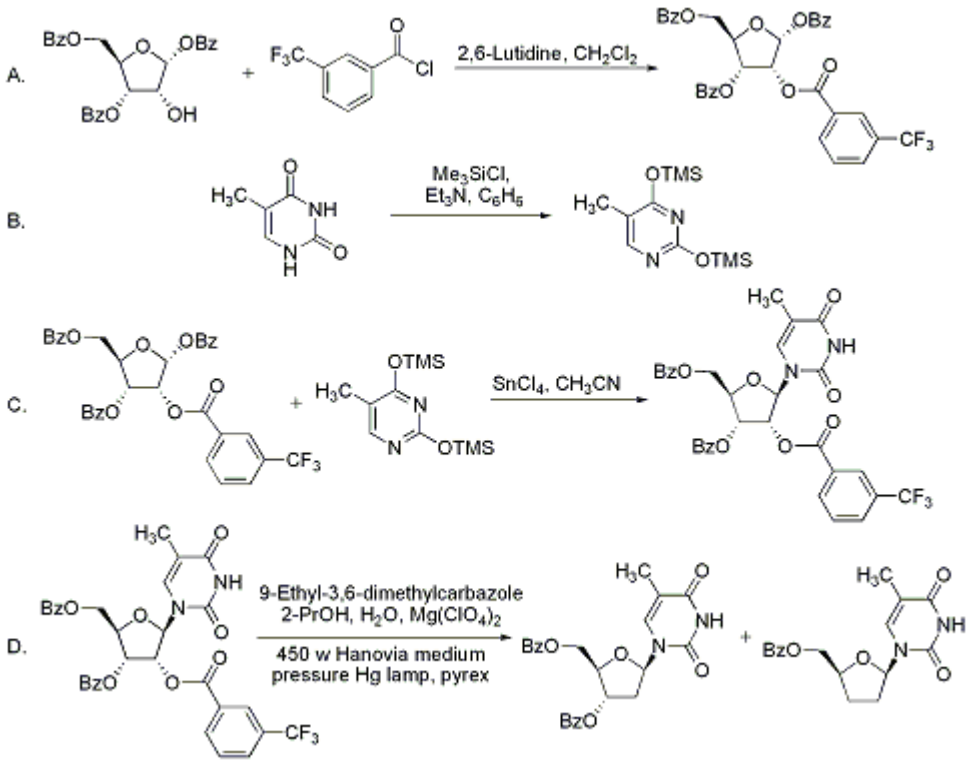
SYNTHESIS OF 2'-DEOXYRIBONUCLEOSIDES: β-3',5'-DI-O-BENZOYLTHYMIDINE

[Thymidine, 3',5'-dibenzoate]



Submitted by Daniel R. Prudhomme, Minnie Park, Zhiwei Wang, Jason R. Buck, and Carmelo J. Rizzo[1] .
Checked by Gilles Chambournier, Jane Djung, and David J. Hart.

1. Procedure

A. 1,3,5-O-Tribenzoyl-2-O-[(3-trifluoromethyl)benzoyl]-α-D-ribofuranose . In an oven-dried, 500-mL, two-necked, round-bottomed flask equipped with a magnetic stir bar and a rubber septum is placed 1,3,5-tri-O-benzoyl-α-D-ribofuranose (5.0 g, 10.8 mmol) (Note 1) and 2,6-lutidine (1.4 g, 13.0 mmol) (Note 2) in 200 mL of dry dichloromethane (Note 3) under an argon atmosphere. The reaction mixture is cooled to 0°C and 3-(trifluoromethyl)benzoyl chloride (3.3 g, 16.2 mmol) (Note 4) is added dropwise to the stirred solution over 30 min. After the addition, the reaction mixture is warmed to room temperature and stirred overnight. The reaction is quenched with 80 mL of aqueous saturated sodium bicarbonate , the phases are separated and the aqueous phase is extracted with dichloromethane (2 × 200 mL). The combined organic extracts are washed with brine (2 × 100 mL) and dried over anhydrous magnesium sulfate . After filtration, the solvent is removed under reduced pressure to give a yellow oil. Purification by flash column chromatography (140 g of silica gel) (Note 5) and eluting with 25% ethyl acetate in hexanes gives a white solid that can be recrystallized from ethyl acetate/hexanes to yield 1,3,5-O-tribenzoyl-2-O-[(3-trifluoromethyl)benzoyl]-α-D-ribofuranose (5.62 g, 82% yield) as white crystals, mp 109-111°C (lit.[2] mp 102°C) (Note 6).

B. 2,4-Bis(trimethylsilyloxy)-5-methylpyrimidine . In an oven-dried, 1-L, round-bottomed flask is placed thymine (12.6 g, 0.1 mol) (Note 1) and chlorotrimethylsilane (Note 7) in 300 mL of benzene (Note 8) under an argon atmosphere. To the stirred suspension is added triethylamine (20.2 g, 0.2 mol) (Note 9) dropwise via a dropping funnel over 1 hr. After the addition is complete, the reaction is stirred

overnight. The precipitated triethylammonium chloride and unreacted thymine are removed by suction filtration (Note 10) and the solid is washed with dry benzene (2 × 30 mL). The solvent is then removed with a rotary evaporator. The resulting viscous oil is distilled under reduced pressure (bp 94-97°C at 9 mm, lit.[3] bp 124°C at 14 mm) to give 2,4-bis(trimethylsilyloxy)-5-methylpyrimidine (24.0 g, 89% yield). The product solidifies upon standing overnight, mp 73-75°C (lit.[3] mp 63-65°C) (Note 11).

C. 3',5'-Di-O-benzoyl-2'-O-[(3-trifluoromethyl)benzoyl]-5-methyluridine . In an oven-dried, 250-mL, round-bottomed flask equipped with a magnetic stir bar and a rubber septum is placed 1,3,5-tri-O-benzoyl-2-O-[(3-trifluoromethyl)benzoyl]-α-D-ribofuranose (5.6 g, 8.8 mmol) and 2,4-bis (trimethylsilyloxy)-5-methylpyrimidine (2.16 g, 7.1 mmol) in 100 mL of acetonitrile (Note 12). At this stage, the reaction may appear as a white suspension (Note 13). The stirred reaction mixture is cooled to 0°C and tin(IV) chloride (13.75 g, 52.8 mmol) (Note 14) is added dropwise over 15 min with vigorous stirring. Upon addition of tin(IV) chloride, the reaction becomes homogeneous. The reaction mixture is warmed to room temperature and stirred for 6 hr. The reaction is then quenched by pouring the mixture into a 1-L separatory funnel containing 250 mL of aqueous saturated sodium bicarbonate and 200 g of ice. Upon quenching the reaction, voluminous white precipitates appear. Ethyl acetate (200 mL) is added, the separatory funnel is cautiously swirled, and the layers are allowed to separate. The aqueous layer, which now appears milky white, is further extracted with ethyl acetate (2 × 100 mL). The combined extracts are washed with brine (2 × 200 mL) and dried over anhydrous sodium sulfate . After filtration, the solvent is removed under reduced pressure to give a yellow oil. Purification by flash column chromatography (100 g of silica gel) (Note 5) eluting with 25% ethyl acetate in hexanes gives 3',5'-di-O-benzoyl-2'-O-[(3-trifluoromethyl)benzoyl]-5-methyluridine as a white solid (3.52 g, 62% yield), mp 92-95°C (Note 15), (Note 16).

D. 3',5'-Di-O-benzoylthymidine . In a 500-mL photochemical reaction vessel, (Note 17) equipped with a magnetic stir bar, is placed 3',5'-di-O-benzoyl-2'-O-[(3-trifluoromethyl)benzoyl]-5-methyluridine (3.82 g, 6.0 mmol), magnesium perchlorate hexahydrate (0.46 g, 1.4 mmol) (Note 1) and 9-ethyl-3,6-dimethylcarbazole (200 mg, 0.9 mmol ; for preparation see: Buck, J. P.; Park, M.; Wang, Z.; Prudhomme, D. R.; Rizzo, C. J. *Org. Synth.* **1999**, *77*, 153) in 500 mL of 9:1 2-propanol/water (Note 18). The solution is degassed by bubbling argon through the solution for 1 hr using a syringe needle. The photochemical immersion well, equipped with a Pyrex filter is fitted into the reaction vessel and made air tight by lightly greasing the ground glass joints; an argon atmosphere is maintained. The reaction is cooled with an ice bath (Note 19) and photolyzed with a Hanovia 450W, medium pressure mercury lamp through a Pyrex filter for 1 hr (Note 20). The reaction mixture is transferred to a 1-L flask and the solvent is removed with a rotary evaporator. The residue is dissolved in ethyl acetate (150 mL), washed with aqueous saturated sodium bicarbonate (2 × 30 mL), then brine (30 mL), and the organic phase is dried over magnesium sulfate. After filtration, the solvent is removed under reduced pressure to give a yellow oil. Purification by flash column chromatography (150 g of silica gel) (Note 5) eluting with 50% ethyl acetate in hexanes gives 3',5'-di-O-benzoylthymidine (1.43 g, 53% yield), mp 195-196° C (lit.[4] mp 194-195°C) (Note 21), and 5'-O-benzoyl-3'-deoxythymidine (0.32 g, 16% yield), mp 71-74° C (lit.[5] mp 57-59°C) (Note 22), and recovered starting material (0.41 g, 11% yield) (Note 23).

2. Notes

1. The ribofuranose and magnesium perchlorate hexahydrate were obtained from Aldrich Chemical Company, Inc. , and used as received.
2. 2,6-Lutidine (99%) was obtained from Aldrich Chemical Company, Inc. , in a Sure/Seal bottle and used as received.
3. Dichloromethane was freshly distilled from calcium hydride.
4. 3-(Trifluoromethyl)benzoyl chloride was obtained from Acros Chemicals or Aldrich Chemical Company, Inc. , and used as received.
5. Silica gel (32-63 μm) was obtained from Fisher Scientific Company .
6. The physical properties are as follows: $[\alpha]_D^{21}$ +89.5° ($CHCl_3$, *c* 0.1); IR (KBr) cm^{-1}: 1730 ; ^1H NMR ($CDCl_3$) δ: 4.71 (ABX, 2 H, J_{AB} = 12.1, J_{AX} = 3.5, J_{BX} = 3.1, $\Delta\nu_{AB}$ = 32.6), 4.93 (m, 1 H), 5.79 (dd, 1 H, J = 6.5, 4.4), 5.88 (dd, 1 H, J = 6.5, 2.1), 6.91 (d, 1 H, J = 4.3), 7.31-7.61 (m, 10 H), 7.72 (d, 1 H, J = 4.3), 8.0-8.11 (m, 8 H) ; ^{13}C NMR ($CDCl_3$) δ: 64.0 (t), 70.7 (d), 71.5 (d), 82.8 (d), 94.9 (d), 126.3, 126.4, 128.4, 128.5, 128.6, 128.9, 129.2, 129.3, 129.5, 129.7, 129.8, 130.0, 133.0, 133.4, 133.6, 133.7, 163.5, 165.1, 165.6, 166.0 (s) , several aromatic signals were not resolved; exact mass calcd. for $C_{34}H_{25}F_2O_9$

(M^+-F) m/z 615.1480. found m/z 615.1473. Anal. Calcd for $C_{34}H_{25}F_3O_9$: C, 64.34; H, 3.97. Found: C, 63.90; H, 3.97.
7. Chlorotrimethylsilane (99+%) was obtained from Aldrich Chemical Company, Inc. , in a Sure/Seal bottle and used as received or distilled from calcium hydride just prior to use.
8. Benzene was freshly distilled from sodium.
9. Triethylamine (99+%) was obtained from Aldrich Chemical Company, Inc. , and was freshly distilled from calcium hydride.
10. A plastic drying tube filled with Drierite was placed between the filter and the aspirator.
11. The product was stored under argon in a desiccator and appears to be stable for months. This material exhibited the following spectral characteristics: ^{1}H NMR (C_6D_6) δ: 0.3 (s, 9 H), 0.4 (s, 9 H), 1.8 (s, 3 H), 7.8 (s, 1 H) ; exact mass calcd. for $C_{11}H_{19}N_2O_2$ Si_2 (M^+- CH_3) m/z 255.0918. found m/z 255.0963.
12. Anhydrous acetonitrile was obtained from Aldrich Chemical Company, Inc. in a Sure/Seal bottle and used as received.
13. The white insoluble material is partially hydrolyzed bis-TMS-thymine. The degree to which this suspension forms depends on the purity of the bis-TMS-thymine and the water content of the flask and solvents.
14. Neat tin(IV) chloride (99%) was obtained from Aldrich Chemical Company, Inc. in a Sure/Seal bottle and used as received.
15. The physical properties are as follows: $[\alpha]_D^{21}$ −78° ($CHCl_3$, *c* 0.1); ^{1}H NMR ($CDCl_3$) δ: 1.54 (s, 3 H), 4.65 (m, 1 H), 4.70 (ABX, 2 H, J_{AB} = 12.3, J_{AX} = 2.6, J_{BX} = 3.5, $\Delta\nu_{AB}$ = 69.5), 5.75 (t, 1 H, J = 6.1), 5.84 (dd, 1 H, J = 3.8, 6.0), 6.33 (d, 1 H, J = 6.1), 7.33-7.63 (m, 8 H), 7.93 (d, 2 H, J = 7.3), 7.99-8.17 (m, 4 H), 8.78 (br s, 1 H) ; ^{13}C NMR ($CDCl_3$) δ: 12.4, 64.2, 71.6, 74.0, 80.8, 87.3, 112.5, 127.0, 128.7, 128.9, 129.1, 129.4, 129.6, 129.9, 130.0, 130.6, 133.4, 134.0, 135.1, 150.6, 163.7, 164.4, 165.7, 166.2 ; IR (KBr) cm^{-1}: 3373, 3276, 3074, 1725, 1619, 1452, 1267, 1128, 1095, 713 . Anal. Calcd for $C_{32}H_{25}F_3N_2O_9$: C, 60.17; H, 3.95. Found: C, 60.26; H, 4.06.
16. The submitters report an 82% yield for this reaction. The checkers recovered 0.5 g (9%) of starting material (mp 108-110°C) after recrystallization of material from early fractions of the column.
17. The submitters purchased the reaction vessel from Ace Glass, Inc. (Catalog # 7841-15) and used a quartz immersion well purchased from Ace Glass, Inc. (Catalog # 7858-08) with a Pyrex filter and a Hanovia medium pressure, 450 W mercury lamp (Ace Glass, Inc. Catalog #7875). The checkers used the same apparatus except with a jacketed Pyrex immersion well in place of the quartz immersion well with a Pyrex filter.
18. The solvent was previously degassed by bubbling UHP argon through the solvent for 30 min using a gas sparger. HPLC grade 2-propanol was obtained from Aldrich Chemical Company, Inc. , and used as received. The submitters used deionized water and the checkers used HPLC grade water from Aldrich Chemical Company, Inc.
19. The reaction assembly was immersed in a 5-gallon bucket of ice water. The reaction temperature was monitored and never went higher than 20°C. The checkers cooled the immersion well with recirculating water chilled to 10°C and used a 9-L bucket of ice water, and the internal temperature never went higher than 15°C.
20. The rate of reaction is highly variable depending on the UV source. The reaction is easily monitored by TLC on silica gel using ethyl acetate-hexane (1:1) as the eluent and p-anisaldehyde stain. The R_f values and staining colors of starting materials and products follow: 9-ethyl-3,6-dimethylcarbazole: R_f = 0.95 (gray); 3',5'-di-O-benzoyl-2'-O-[(3-trifluoromethyl)benzoyl]-5-methyluridine: R_f = 0.75 (pink-purple); 5'-O-benzoyl-3'-deoxythymidine: R_f = 0.55 (aqua blue); 3',5'-di-O-benzoylthymidine: R_f = 0.30 (brown-black).
21. The submitters indicate that synthetic 3',5'-di-O-benzoylthymidine was identical in all respects to an authentic sample prepared by the benzoylation of thymidine: IR (KBr) cm^{-1}: 1695 ; ^{1}H NMR ($CDCl_3$) δ: 1.62 (s, 3 H), 2.35-2.37 (m, 1 H), 2.67-2.73 (m, 1 H), 4.54 (d, 1 H, J = 2.0), 4.75 (ABX, 2 H, J_{AB} = 12.3, J_{AX} = 3.0, J_{BX} = 2.9, $\Delta\nu_{AB}$ = 36.7), 5.66 (d, 1 H, J = 4.5), 6.47 (d, 1 H, J = 8.7, 5.6), 7.46-7.51 (m, 5 H), 7.60-7.65 (m, 2 H), 8.03-8.09 (m, 4 H), 8.50 (br s, 1 H) ; ^{13}C NMR ($CDCl_3$) δ: 12.7 (q), 38.0 (t), 64.3 (t), 75.0 (d), 82.7 (d), 84.9 (d), 111.7 (s), 128.6 (d), 128.8 (d), 129.0 (s), 129.3 (s), 129.5 (d), 129.8 (d), 133.7 (d, 2C), 134.4 (d), 150.1 (s), 163.2 (s), 166.0 (s), 166.1 (s) . Anal. Calcd for $C_{24}H_{22}N_2O_7$: C, 63.98; H, 4.93. Found: C, 64.07; H, 4.96.
22. Synthetic 5'-O-benzoyl-3'-deoxythymidine was identical in all respects to an authentic sample

prepared by the benzoylation of 3'-deoxythymidine: IR (KBr) cm^{-1}: 1694 ; ^1H NMR ($CDCl_3$) δ: 1.70 (d, 3 H, J = 0.9), 1.76-2.21 (m, 3 H), 2.44-2.51 (m, 1 H), 4.40-4.50 (m, 1 H), 4.53 (dd, 1 H, J = 12.2, 4.6), 4.65 (dd, 1 H, J = 12.2, 2.8), 6.1 (dd, 1 H, J = 6.4, 4.2), 7.35 (s, 1 H), 7.45 (t, 2 H, J = 7.5), 7.60 (t, 1 H, J = 7.4), 8.03 (d, 2 H, J = 7.1), 8.98 (br s, 1 H) ; ^{13}C NMR ($CDCl_3$) δ: 12.4, 26.0, 32.3, 65.3, 78.4, 86.1, 110.7, 128.7, 129.6, 133.4, 133.5, 135.1, 150.3, 163.8, 166.0 .

23. In one experiment, the checkers obtained 30%, 53%, and 1% of starting material (SM), monodeoxygenation product (MD) and dideoxygenation product (DD), respectively, after 7 hr of irradiation. In a second experiment, they obtained 3%, 57%, and 5% of SM, MD, and DD, respectively, after 10 hr of irradiation. The checkers also recovered 110 mg (55%) of 9-ethyl-3,6-dimethylcarbazole (mp 56-58°C) from early fractions of the column.

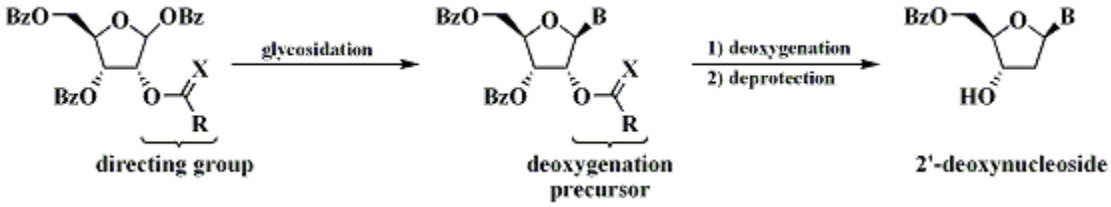
Waste Disposal Information

All toxic materials were disposed of in accordance with "Prudent Practices in the Laboratory"; National Academy Press; Washington, DC, 1995.

3. Discussion

Nucleosides and nucleoside analogs are a clinically proven class of medicinal agents possessing antiviral and anticancer activity.[6] [7] [8] [9] The currently used sequence for the de novo synthesis of nucleosides involves the reaction of ribose tetraester with the appropriate silylated base under Lewis acid conditions (Vorbrüggen glycosylation).[6,7,8,9,10] [11] Presumably, reaction of ribose tetraester with Lewis acids give an oxacarbenium ion intermediate. Neighboring group participation of the α-C_2-ester group directs glycosylation exclusively to the desired β-face. In the absence of a directing C_2-group, mixtures of anomers are usually obtained. Robins developed an efficient procedure for the conversion of ribonucleosides to 2'-deoxyribonucleosides that relies on the simultaneous protection of the 3'- and 5'-hydroxyl groups with a bifunctional silylating reagent. The 2'-position is then deoxygenated via tin hydride reduction of the corresponding 2'-phenoxythionocarbonate.[12] [13]

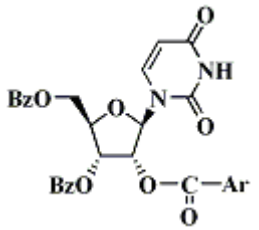
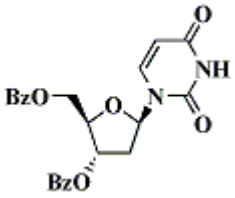
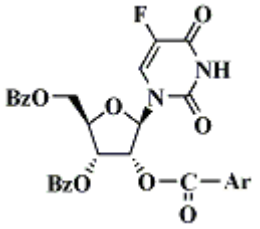
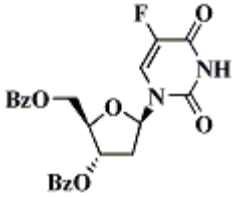
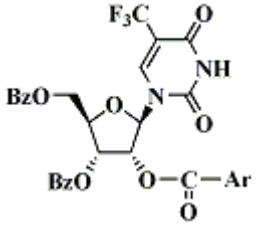
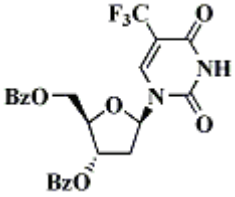
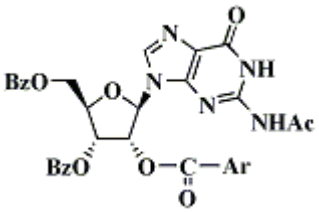
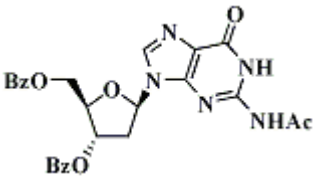
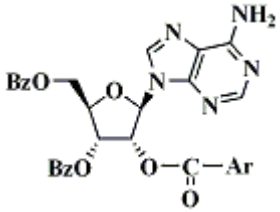
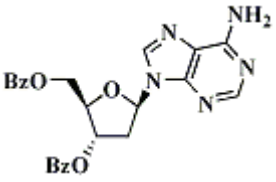
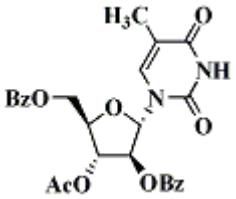
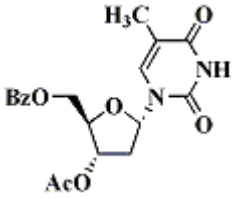
The strategy here was to develop a ribose glycosylation precursor in which the α-C_2-ester could serve as both a directing group for Vorbrüggen glycosylation and a deoxygenation precursor. This strategy was previously employed by Benner for the synthesis of potential antisense nucleosides with modified backbones.[14] In this work, a m-(trifluoromethyl)benzoyl group was used as the directing group/deoxygenation precursor. The submitters subsequently developed this strategy into a general method for the synthesis of β-2'-deoxyribonucleosides.[15] The selective deoxygenation of the 2'-m-(trifluoromethyl)benzoyl group is achieved via a photoinduced electron-transfer (PET) mechanism using stoichiometric 9-methylcarbazole (MCZ) as the electron donor.[16]

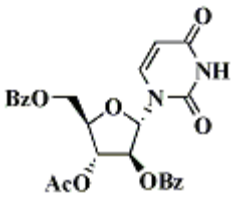
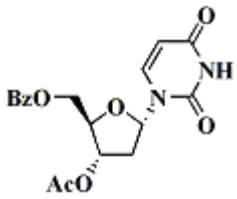
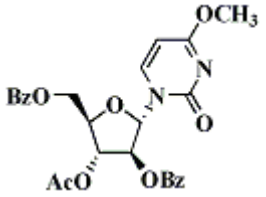
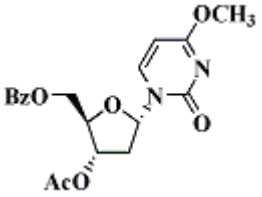
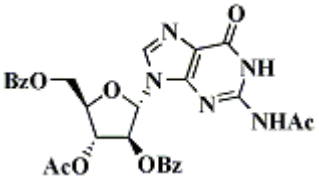
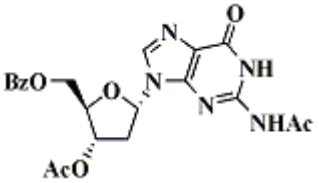
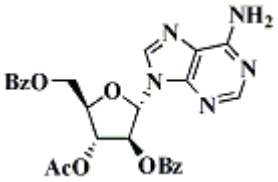
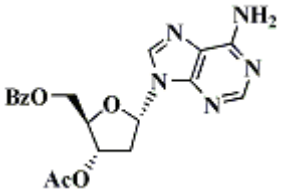
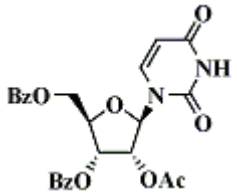
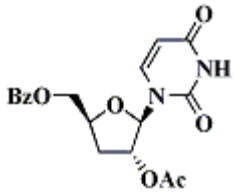
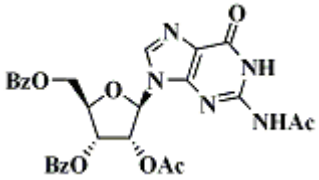
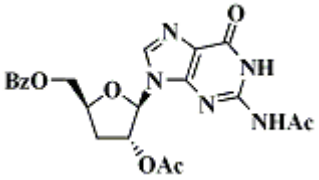
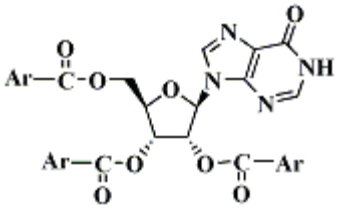
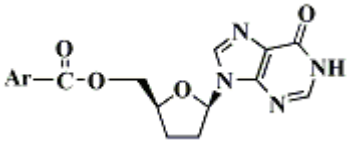
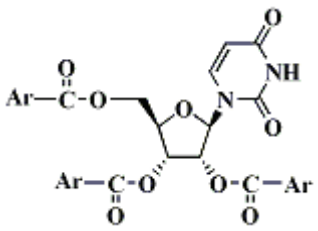
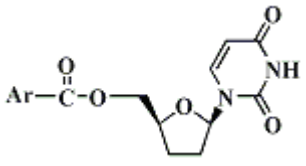


The submitters recently reported the development of 9-ethyl-3,6-dimethylcarbazole (DMECZ) (see also accompanying procedure) as a donor that can be used in 10-20 mol %.[17] In their original report,[15] PET deoxygenation required 6-8 hr with stoichiometric MCZ at a substrate concentration of 1.4 mM. At higher substrate concentrations, the efficiency of PET deoxygenation dramatically decreased with MCZ. With 10-20 mol % of DMECZ as the donor, the deoxygenation of 3',5'-di-O-benzoyl-2'-O-[(3-trifluoromethyl)benzoyl]-5-methyluridine required 1-10 hr at a much higher substrate concentration, making the current procedure efficient and practical. DMECZ also shows improved reactivity and is able to deoxygenate benzoyl groups as well as m-(trifluoromethyl)benzoyl groups. This improved reactivity allowed the submitters to apply this strategy to the synthesis of α-2'-deoxyribonucleosides,[18] β-3'-deoxyribonucleosides and β-2',3'-dideoxyribonucleosides (Table).

TABLE

SYNTHESIS OF DEOXYRIBONUCLEOSIDES VIA PET DEOXYGENATION

Substrate (Ar= m-$CF_3C_6H_4$–	Donor	Product	Yield
	MCZ		70%
	MCZ		53%
	MCZ		44%
	DMECZ		70%
	DMECZ		58%
	DMCZ		73%
	DMECZ		69%

			
	DMECZ		80%
	DMECZ		81%
	DMECZ		76%
	DMECZ		60%
	DMECZ		71%
	DMECZ		65%
	DMECZ		68%

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References and Notes

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Appendix
Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

3',5'-Di-O-benzoylthymidine:
Thymidine, 3',5'-dibenzoate (9): (35898-30-7)

1,3,5-O-Tribenzoyl-2-O-[(3-trifluoromethyl)benzoyl]-α-D-ribofuranose:
α-D-Ribofuranose, 1,3,5-tribenzoate 2-[3-(trifluoromethyl)benzoate (13); (145828-13-3)

1,3,5-Tri-O-benzoyl-α-D-ribofuranose: Aldrich:
α-D-Ribofuranose 1,3,5-tribenzoate:
Ribofuranose, 1,3,5-tribenzoate, α-D- (8);
α-D-Ribofuranose, 1,3,5-tribenzoate (9); (22224-41-5)

2,6-Lutidine (8);
Pyridine, 2,6-dimethyl- (9); (108-48-5)

3-(Trifluoromethyl)benzoyl chloride:
m-Toluoyl chloride, α,α,α-trifluoro- (8);
Benzoyl chloride, 3-(trifluoromethyl)- (9); (2251-65-2)

2,4-Bis(trimethylsilyloxy)-5-methylpyrimidine:
Pyrimidine, 5-methyl-2,4-bis(trimethylsiloxy)- (8);
Pyrimidine, 5-methyl-2,4-bis[(trimethylsilyl)oxy]- (9); (7288-28-0)

Thymine (8);
2,4(1H,3H)-Pyrimidinedione, 5-methyl- (9); (65-71-4)

Chlorotrimethylsilane:
Silane, chlorotrimethyl- (8,9); (75-77-4)

Benzene: CANCER SUSPECT AGENT (8,9); (71-43-2)

Trimethylamine (8);
Ethanamine, N,N-diethyl- (9); (121-44-8)

3',5'-Di-O-benzoyl-2'-O[(3-trifluoromethyl)benzoyl]-5-methyluridine:
Uridine, 5-methyl-, 3',5'-dibenzoate 2'-[3-(trifluoromethyl)benzoate] (13); (182004-59-7)

Acetonitrile: TOXIC (8,9); (75-05-8)

Tin(IV) chloride:
Tin chloride (8);
Stannane, tetrachloro- (9); (7646-78-8)

Magnesium perchlorate hexahydrate:
Perchloric acid, magnesium salt, hexahydrate (8,9); (13446-19-0)

9-Ethyl-3,6-dimethylcarbazole:
9H-Carbazole, 9-ethyl-3,6-dimethyl- (9); (51545-42-7)

5'-O-Benzoyl-3'-deoxythymidine:
Thymidine, 3'-deoxy-, 5'-benzoate (12); (122621-07-2)