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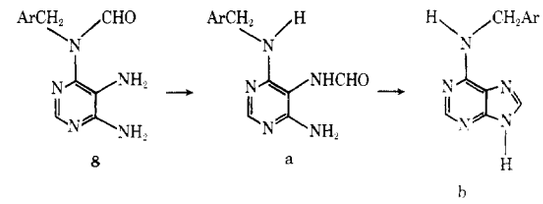

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9-(6-Deoxyhexofuranosyl)adenine Nucleosides. Further Studies on the Acetolysis of Hexofuranosides

Leon M. Lerner

Department of Biochemistry, State University of New York, Downstate Medical Center, Brooklyn, New York 11203

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Methyl 5-*O*-benzoyl-6-deoxy-2,3-*O*-isopropylidene-α-L-talofuranoside was treated with a 10:1 mixture of acetic acid–acetic anhydride containing 5% sulfuric acid. The crude product was coupled with 6-benzamidochloromercuripurine by the titanium tetrachloride method. Removal of blocking groups and chromatography afforded a mixture of nucleosides which were separated by rechromatographing the mixture on an anion-exchange resin. 9-(6-Deoxy-α-L-talofuranosyl)adenine and 9-(6-deoxy-β-L-galactofuranosyl)adenine were obtained in similar amounts. In a like manner, methyl 5-*O*-benzoyl-6-deoxy-2,3-*O*-isopropylidene-β-D-allofuranoside was subjected to the same reaction sequence. In this case too, a mixture of nucleosides was obtained. Separation of the desired 9-(6-deoxy-α-D-altrofuranosyl)adenine was achieved by selective destruction of the allo nucleoside. This was accomplished by short-term oxidation with periodate, reduction of the aldehyde groups with borohydride, and chromatography on an anion-exchange resin. Unlike previous experiments in which only C-2′, C-3′ trans nucleosides were obtained, the sugar derivatives in the present experiments did not undergo complete epimerization at C-2.

In a previous article,[2] reasons for the preparation of nucleosides derived from 6-deoxyhexofuranoses were mentioned and, over the past few years, papers concerned with this subject matter have appeared from this laboratory.[3-5]

A key reaction in some of the synthetic schemes has been acetolysis of appropriately blocked glycosides. During the reaction, acid-labile groups such as anomeric methoxyls and isopropylidene groups are exchanged for acetyl or acetoxyl groups.[6] However, when the acetolysis reaction is performed with a furanose sugar derivative containing three contiguous hydroxyl groups linked to the ring, epimerization at C-2 often occurs if the hydroxyls at C-2 and C-3 are in a cis relationship.[7-9] The best reaction conditions appeared to be a 10:1 acetic acid–acetic anhydride mixture containing 3–5% sulfuric acid.[5,7-9] The reaction has also been scaled up into a useful synthetic tool for the preparation of novel carbohydrates and nucleosides.[2,5,10-12] In the latter case, a number of hexofuranosyl nucleosides with a trans relationship at the C-2′,C-3′ hydroxyl groups have been prepared from hexofuranosides that originally had these hydroxyls in a cis orientation.[5,11,12] In each case, the only major nucleoside product obtained was the one having the C-2′,C-3′ trans arrangement. It was also necessary that C-5 of the sugar be blocked with a benzoyl group rather than an acetate so that acetate exchange and ring rearrangement to the pyranose form did not occur; otherwise, epimerization was incomplete and a substantial amount of the hexopyranosyl nucleoside of the starting sugar was obtained.[5,10-12] The preparation of some new 9-(6-deoxyhexofuranosyl)adenine nucleosides and some interesting developments with the acetolysis reaction are the subject of this article.

The sugar derivatives needed for the preparation of the nucleosides reported herein were obtained starting from 6-deoxy-L-mannose (L-rhamnose). The synthetic pathway is illustrated in Scheme I for purposes of clarity and was based upon literature methods.[13-15]

Acetolysis of methyl 5-*O*-benzoyl-2,3-*O*-isopropylidene-α-L-talofuranoside (**6**) gave a syrup (**7**) which was condensed with 6-benzamidochloromercuripurine by the titanium tetrachloride method.[16] The blocking groups were removed with sodium methoxide in boiling methanol. Chromatography on an anion-exchange column using Dekker's technique[17] of elution with aqueous methanol gave a product which was shown to be a mixture of at least two nucleosides from the value of the optical rotation and from the rate of consumption of periodate. In the latter case, there was a very rapid initial uptake of periodate corresponding to 50–60% of the total material and then a slow uptake over several days until completion of the oxidation. The mixture was rechromatographed with a more dilute aqueous methanol solution. Two nucleosides separated, both of which were crystallized. The first nucleoside to come off the column was 9-(6-deoxy-α-L-talofuranosyl)adenine (**8**). It had previously been prepared from **6** but had not been obtained in crystalline form.[15] More recently, it was obtained by the reaction of 2′,3′-*O*-isopropylideneadenosine-5′-aldehyde with methylmagnesium iodide and crystallized from ethanol as an hemialcoholate.[18] In the present work, **8** was obtained in an anhydrous, unsolvated form having a melting point considerably higher than that of the hemialcoholate. The optical rotation, rate of periodate consumption, and substrate activity with adenosine deaminase (adenosine aminohydrolase EC 3.5.4.4) verified the identity of **8**.

The second nucleoside eluted from the column was 9-(6-

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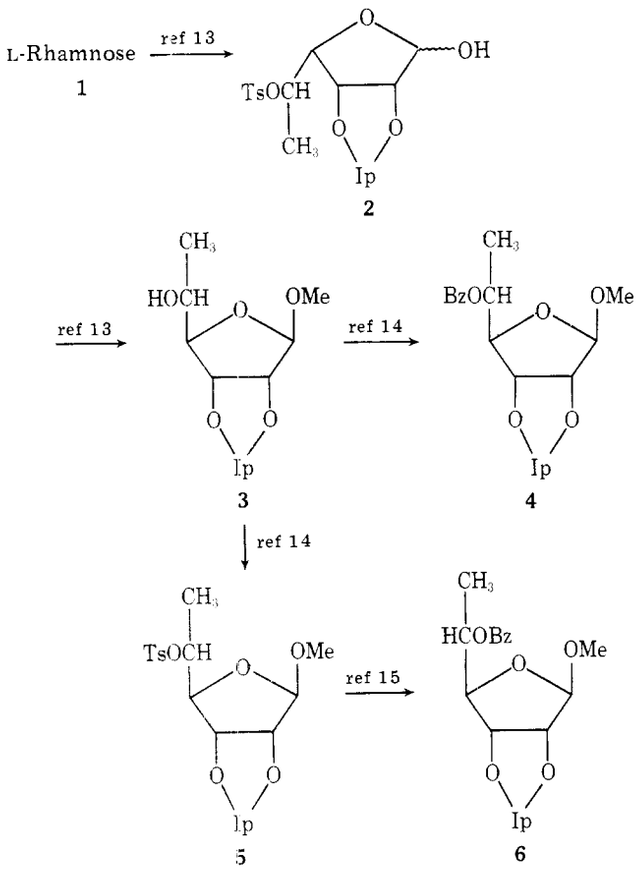
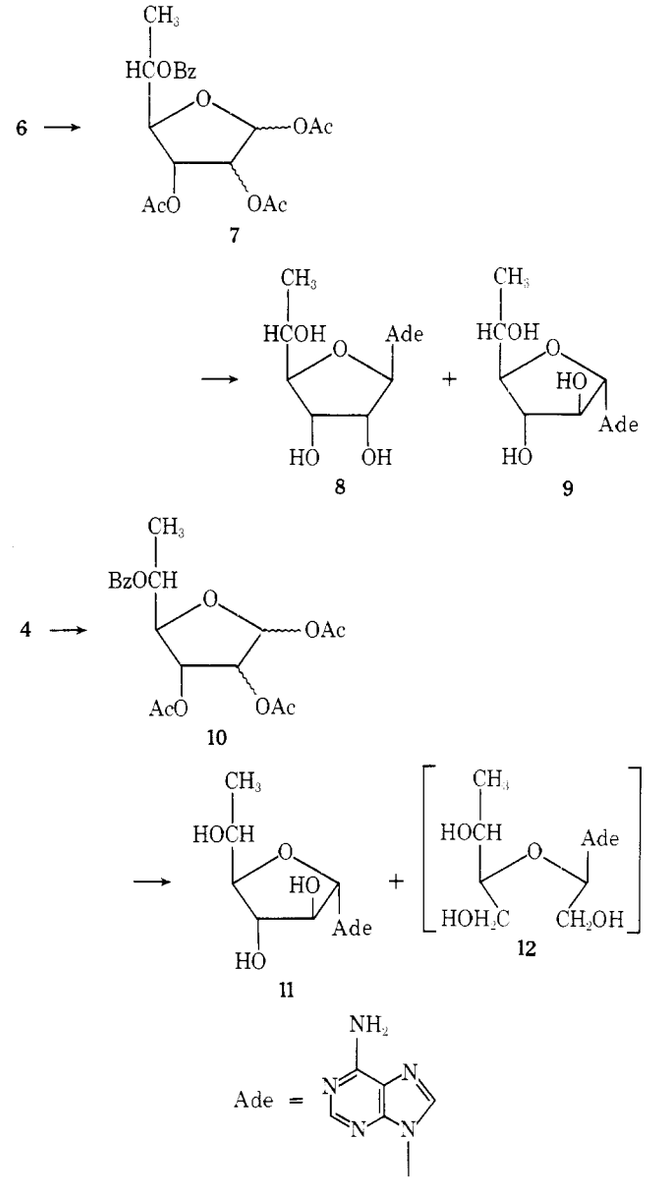
Scheme I



Scheme II



deoxy-β-L-galactofuranosyl)adenine (**9**, 9-β-L-fucofuranosyladenine), which crystallized from water.

Acetolysis of **4** afforded a syrupy product (**10**) which was condensed with 6-benzamidochloromercuripurine in the same manner as in the preparation of nucleosides **8** and **9**. After removal of blocking groups and chromatography on an anion-exchange resin, a crystalline mixture of at least two nucleosides was obtained, as indicated by the wide range of melting and the consumption of periodate. In this case, 35–40% of the total periodate consumed was taken up rapidly, indicating that approximately this much of the mixture was 9-(6-deoxy-β-D-allofuranosyl)adenine. Further attempts to separate these two nucleosides, the second of which was the desired 9-(6-deoxy-α-D-altrofuranosyl)adenine (**11**), failed to resolve them. Since 9-(6-deoxy-β-D-allofuranosyl)adenine can be better prepared from **4** using a route not involving acetolysis,[14] it appeared to be advantageous to use periodate oxidation over a short time to oxidize it selectively, reduce it to the dialcohol, and utilize the anion-exchange column again to isolate the desired nucleoside. It was expected that without the ring hydroxyls the dialcohol would pass through the column well ahead of the nucleoside.[17] In fact, this is what happened. After periodate oxidation, the aldehyde groups were reduced with sodium borohydride, the sodium ions were removed with a cation-exchange resin in the acid form, and the boric acid produced was evaporated as methyl borate. Column chromatography first gave a peak containing the dialcohol **12**, which appeared to be one of a mixture of components, as indicated by the NMR spectrum and the value of the optical rotation. Further purification and characterization of this material was not pursued. The desired product, 9-(6-deoxy-α-D-altrofuranosyl)adenine (**11**), was eluted with 60% aqueous methanol and was obtained in crystalline form from acetone.

The UV spectra of **9** and **11** showed that they were *N*-9 substituted nucleosides. The elemental analysis of **11** suggested that it had 0.5 mol of acetone as solvate of crystallization. This was verified by the infrared spectrum which had a carbonyl peak at 1715 cm^{-1} and the NMR spectrum which had the acetone methyl at δ 1.87 and integrated for exactly 0.5 mol of acetone methyl proton per mole of nucleoside. Periodate uptake experiments showed that each nucleoside had the furanose ring form. Rearrangement to a pyranose ring would have resulted in the consumption of 2 mol of periodate and not 1 mol. The rate of consumption was indicative of the relative configuration of the hydroxyl groups at C-2′ and C-3′. Whereas the talo nucleoside **8** completely consumed nearly 1 mol of periodate almost instantly, **9** and **11** each required several days to consume the same amount. It is known from previous experience that it is C-2 of the sugar that epimerizes[2-12] and this is verified again in this work, since the data of the physical properties of the new nucleosides **9** and **11** do not conform to that of 9-(6-deoxy-α-L-idofuranosyl)adenine[2] or 9-(6-deoxy-β-D-glucofuranosyl)adenine,[19] respectively, the nucleosides expected if C-3 would have inverted instead of C-2.

Unfortunately, the NMR spectra of **9** and **11** did not give conclusive information regarding the nature of the anomeric configurations. The anomeric proton of **9** was partially ob-

Table I. Optical Rotations of Nucleosides and Their Alcohols

9-(6′-Deoxyhexofuranosyl)-adenine	$[\alpha]_D$ of nucleoside, deg	Registry no.	$[\alpha]_D$ of nucleoside alcohol deg[a]
α-L-talo (8)	−39.7	35868-16-7	+72
β-L-galacto (9)	+73.2	64811-72-9	−48
α-D-altro (11)	+52.9	64811-73-0	−62

[a] Based upon the calculated dry weight of the alcohol product.

scured by other protons, and the anomeric proton of 11 was a doublet with a coupling constant too large to assign a configuration.[20] It was expected that the nucleosides would have the adenine ring in a configuration trans to the hydroxyl at C-2′.[21] Comparison of the optical rotations of the new nucleosides with the optical rotations of other hexofuranosyl nucleosides[2] appeared to confirm the configurational assignments. The anomeric configuration of **8**, although not demonstrated in the original work,[15] was clearly demonstrated by its preparation from 9-β-D-ribofuranosyladenine (adenosine).[18] Moreover, **8** was a substrate for adenosine deaminase, but **9** and **11** were not. Adenosine deaminase only catalyzes the rapid deamination of adenine nucleosides having a β-D or α-L configuration if other structural requirements[2,22] for substrate activity are fulfilled. Since **9** and **11** have structural features which otherwise would allow them to act as substrates, the fact that they do not can be construed as evidence for the assigned anomeric configurations.

Another argument in support of the assigned anomeric configurations is based upon an observation made in a previous paper.[2] It had been noticed[2] that alcohols derived from nucleosides that have been oxidized with periodate and the aldehyde groups reduced acquire an optical rotation of fairly large value and sign opposite to that of the original nucleoside. The number of asymmetric carbon atoms in the alcohol does not appear to matter. Nucleosides having a β-D or α-L configuration yield alcohols which have a positive optical rotation, and nucleosides having an α-D or β-L configuration yield alcohols which have a negative optical rotation. Table I reports the optical rotations of nucleosides **8**, **9** and **11** and the alcohols derived from them. These data also support the assignments of anomeric configuration of the nucleosides.

The results obtained from the acetolysis reaction were somewhat disappointing for preparative purposes. Only a minor portion of the hexofuranosides appear to have epimerized at C-2 and as a result the main products still possessed the cis configuration. Previously, I had reported epimerizations of hexofuranosides which were virtually complete. This was particularly the case when the hydroxyl at C-5 was blocked with a benzoyl group, a group that is not displaced and does not migrate under acetolysis conditions. In the earliest experiments from this laboratory,[4,5] the acetyl group at C-5 appeared to allow rearrangement of the furanose ring to a pyranose ring which prevented a significant portion of the sugar from undergoing epimerization. Proof of this was obtained in the form of the pyranosyl nucleoside of the original sugar. Since pyranosyl nucleosides were not obtained in the present case, there is no reason to believe that this is happening here in spite of the benzoyl group. However, comparison of the structures of the hexofuranosides used in the present work to the ones used previously do reveal a fundamental difference. The cis hydroxyl groups at C-2 and C-3 in the previous work were located on the same side of the furanose ring as the C-4 tail containing C-5 and C-6. In the present work, these two hydroxyl groups were on the opposite side of the ring from the C-4 tail. Therefore, one could easily suspect that the benzoyl group is simply sterically hindering the formation of the proposed[7,8] orthoester ion intermediate. The recent transformation of methyl 5-deoxy-2,3-*O*-isopropylidene-β-D-ribofuranoside into 9-(5-deoxy-α-D-arabinofuranosyl)adenine[23] by the same route would tend to support this view; no D-ribonucleoside was obtained. The problem is that recent unpublished experiments of this laboratory reveal that 5,6-unsaturated hexofuranosides, such as methyl 5,6-dideoxy-2,3-*O*-isopropylidene-β-D-*ribo*-hex-5-enofuranoside, also do not afford good yields of C-2 epimerized products after acetolysis and formation of the nucleosides. Apparently, the success or failure of this reaction with hexofuranosides is greatly determined by thermodynamic factors.

Experimental Section[24]

9-(6-Deoxy-α-L-talofuranosyl)adenine (8) and 9-(6-Deoxy-β-L-galactofuranosyl)adenine (9). Methyl 5-*O*-benzoyl-6-deoxy-2,3-*O*-isopropylidene-α-L-talofuranoside[15] (2.64 g) was acetolyzed for 5 days at room temperature in a mixture containing 5 mL of acetic anhydride, 50 mL of acetic acid, and 2.6 mL of concentrated sulfuric acid. The mixture was poured into 200 mL of ice, stirred until the ice melted, and extracted with chloroform (3 × 40 mL). The extracts were combined, washed with water (2 × 125 mL), saturated sodium bicarbonate (2 × 125 mL), and water (150 mL), and dried. Evaporation of the chloroform and three coevaporations with benzene gave a colorless syrup weighing 1.89 g.

The syrup was dissolved in 160 mL of 1,2-dichloroethane and placed in a three-neck flask fitted with a take-off adapter, a condenser, and a drying tube. 6-Benzamidochloromercuripurine (2.70 g) and Celite-545 (2.70 g) were added, and 25 mL of solvent was distilled. A solution containing 0.75 mL of titanium tetrachloride in 25 mL of fresh 1,2-dichloroethane was then added, and the stirred mixture was heated under reflux for 21 h. The mixture was cooled to room temperature, and 100 mL of saturated sodium bicarbonate was added, stirred for 1.5 h, and filtered by suction. The filter cake was washed with 200 mL of hot 1,2-dichloroethane, the organic layer was separated, and the solvent was evaporated. The residue was dissolved in 100 mL of chloroform, washed with 30% potassium iodide solution (2 × 100 mL) and water (150 mL), and dried. Evaporation of the chloroform gave a yellow foam weighing 2.57 g. This was dissolved in 70 mL of methanol and treated with 7 mL of 1 N methanolic sodium methoxide solution. The solution was refluxed for 1.5 h, cooled to room temperature, and neutralized with Amberlite CG-120 (H^+) ion-exchange resin. The resin was filtered off, the methanol was evaporated, and the residue was coevaporated several times with water to get rid of methyl benzoate. The dark product was dissolved in water and added to the top of a column (15 × 1.8 cm) of Bio-Rad AG 1-X2 (OH^-, 200–400 mesh) ion-exchange resin that had been packed in water. The column was eluted with 30% aqueous methanol and the major UV-absorbing peak was evaporated to a foam and then rechromatographed on a larger column (33 × 2 cm). The column was eluted with water and 14-mL fractions were collected. The solvent was changed to 10% methanol at tube 71 and 12-mL fractions were collected. Fractions 121–228 were pooled and evaporated to a white foam which was dried by coevaporation with ethanol. Crystallization was effected from ethanol upon standing in an open flask. This product was identified as 9-(6-deoxy-α-L-talofuranosyl)adenine (8) and weighed 213 mg. A second crop of crystals weighing 35 mg was also obtained. The crystals were dried over phosphorus pentoxide for 24 h under high vacuum at 100 °C, mp 206–209 °C, softening at 201 °C with the formation of tiny droplets: $[\alpha]^{25}_D$ −39.7° (*c* 0.897, water) (lit.[18] $[\alpha]_D$ −39 ± 2°).

Anal. Calcd for $C_{11}H_{15}N_5O_4$: C, 46.96; H, 5.41 N, 24.80. Found: C, 46.76; H, 5.46; N, 24.98.

Fractions 238–430, the other major peak, were combined and evaporated. 9-(6-Deoxy-β-L-galactofuranosyl)adenine (9) was crystallized from water in two crops to give 223 mg, mp 240–242 °C, with tiny droplets forming shortly before melting: $[\alpha]^{25}_D$ +73.2° (*c* 0.877, water); UV λ_{max} (H_2O) 259 nm (ε 14 980); NMR (Me_2SO-d_6) δ 8.17, 8.03 (both s, 1 proton each, H-8, H-2), ~5.80 (H-1′ overlapping with 2′,3′-OH), 1.06 (d, 3, CH_3).

Anal. Calcd for $C_{11}H_{15}N_5O_4$: C, 46.96; H, 5.41; N, 24.90. Found: C, 46.96; H, 5.30; N, 24.89.

9-(6-Deoxy-α-D-altrofuranosyl)adenine (11). Methyl 2,3-*O*-isopropylidene-5-*O*-benzoyl-6-deoxy-β-D-allofuranoside[14] (4.44 g) was acetolyzed for 7 days in a mixture containing 80 mL of acetic acid,

8 mL of acetic anhydride, and 4.4 mL of concentrated sulfuric acid. The contents were poured into 300 mL of ice and stirred until the ice melted. The mixture was extracted with chloroform (3 × 50 mL), and the extracts were combined, washed with water (2 × 150 mL), saturated sodium bicarbonate (2 × 150 mL), and water (150 mL) and dried. Evaporation of the chloroform and several coevaporations with benzene gave a clear, colorless syrup weighing 2.8 g.

The syrup was reacted with 4.0 g of 6-benzamidochloromercuripurine in 230 mL of 1,2-dichloroethane containing 4 g of Celite-545 and 1.1 mL of titanium tetrachloride as described above for the preparation of **8** and **9**. A yellow foam weighing 3.86 g was obtained which was dissolved in 75 mL of methanol and treated under reflux with 8 mL of 1 N methanolic sodium methoxide. The reaction proceeded for 1.5 h and was neutralized and methyl benzoate removed as described above. The dark residue was dissolved in water and chromatographed on a column (31 × 2 cm) of Bio-Rad AG1-X2 (OH^-, 200–400 mesh) resin. The column was eluted with water (1 L), 10% aqueous methanol (2 L), and 20% aqueous methanol (4 L). The major UV-absorbing peak came off in the latter solvent system. The solvents were evaporated and a little color was removed with Darco G-60 charcoal. After evaporation, a solid residue remained which was crystallized from acetone in several crops to afford 1.243 g. The wide melting range above 108 °C and the $[\alpha]_D$ −13° indicated that this was a mixture. A periodate uptake experiment revealed that at least 35–40% of this material had a cis relationship between C-2′ and C-3′. The solid (1.187 g) was dissolved in 50 mL of water, chilled to 15 °C, and treated with 1.0 g of sodium periodate. The reaction was kept at room temperature for 45 min, 0.3 g of ethylene glycol was added in 3 mL of water, and after another 15 min the mixture was poured into 350 mL of vigorously stirred ethanol. Fifteen minutes later the salt was removed by filtration and washed with two 20-mL portions of ethanol. The solvents were evaporated (30 °C), and the remaining syrup was dissolved in 65 mL of water and treated with a solution containing 0.9 g of sodium borohydride in 10 mL of water. The reaction proceeded for 2 h, the excess borohydride was decomposed and the pH adjusted to neutrality with Bio-Rad AG50W-X8 (H^+) ion-exchange resin. The mixture was filtered through a pad of Celite, the water was evaporated, and the residue was coevaporated three times with methanol to remove boric acid as methyl borate. The residue was dissolved in water and chromatographed on the anion-exchange resin used previously (32 × 2 cm column) and 15-mL fractions were collected using the following solvent systems: water (1L), 10% aqueous methanol (1L), 20% aqueous methanol (2L), and finally 60% aqueous methanol. The major UV-absorbing peaks were in tubes 23–77 and 279–316, the latter being eluted with the last solvent used. The material in tubes 23–77 precipitated from ethanol to afford 511 mg containing the alcohol **12**, but judged to be a mixture of components based upon the optical rotation, $[\alpha]^{25}_D$ −7° (*c* 1.12, water), and NMR spectrum. No further work was done on this sample.

Fractions 279–316 were combined, the solvents were evaporated, and the residue was crystallized from acetone (scratching). Two more recrystallizations from acetone gave 141 mg of pure 9-(6-deoxy-α-D-altrofuranosyl)adenine (**11**) containing 0.5 mol of acetone of crystallization, mp 112–115 °C, to a very, viscous syrup: $[\alpha]^{25}_D$ +52.9° (*c* 0.77, water); UV λ_{max} (H_2O) 260 nm (ε 13 480); IR 1715 cm^{-1} (C=O, acetone); NMR (Me_2SO-d_6) δ 8.17, 8.03 (both s, 1 proton each, H-8, H-2), 5.82 (d, 1, J' = 4 Hz, H-1′), 1.87 (s, 3, acetone CH_3), 1.10 (d, 3, C-6′ CH_3).

Anal. Calcd for $C_{11}H_{15}N_5O_4 \cdot 0.5CH_3C(=O)CH_3$: C, 48.38; H, 5.84; N, 22.57. Found: C, 48.41; H, 5.95; N, 21.89.

Periodate Uptake. The procedure of Rammler and Rabinowitz[25] in which the disappearance of periodate is measured at 300 nm was used to determine periodate consumption. Nucleoside **8** consumed 0.85 molar equiv in less than 5 min, whereas nucleoside **9** required 65.5 h to consume 0.90 molar equiv. Nucleoside **11** consumed 0.91 molar equiv of periodate in 72 h.

Polarimetric Studies. Details of the procedure to oxidize and reduce small samples of the nucleosides appear in a previous article.[2] The results are shown in Table I.

Deamination with Adenosine Deaminase. Deamination was followed at 265 nm at 25 °C in 0.05 M phosphate buffer (pH 7.6).[26] The concentration of nucleosides was approximately 5×10^{-5} M, and 3 mL of this solution was placed in a cuvette and 0.1 mL of enzyme (Sigma Chemical Co.) containing 2.1 units was added. Nucleoside **8** underwent complete deamination in about 3 min; nucleosides **9** and **11** were not substrates even with enzyme levels as high as 53 units per cuvette.

Registry No.—**4**, 29325-26-6; **6**, 28538-27-4; **7** isomer I, 64761-44-0; **7** isomer II, 64761-45-1; **10** isomer I, 64761-46-2; **10** isomer II, 64761-47-3; 6-benzamidochloromercuripurine, 17187-65-4.

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