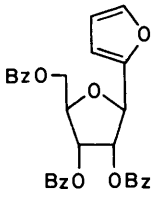
C-Nucleosides. 12.† Synthesis of 2′-Deoxy Pyridazinone *C*-Nucleoside from 2-(2,3,5-Tri-*O*-benzoyl-β-D-ribofuranosyl)furan

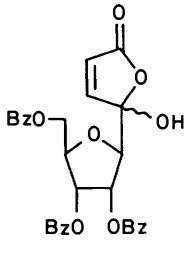
Isamu Maeba,* Takashi Iijima, Yoko Matsuda, and Chihiro Ito
Faculty of Pharmacy, Meijo University, Tempaku, Nagoya 468 Japan

The versatile 2′-deoxy-β-D-ribofuranosyl-*C*-nucleoside precursor 4-(1,4-anhydro-3,5-di-*O*-benzoyl-2-deoxy-D-*erythro*-pentofuranosyl)-4-oxobutyric acid (**4**) can be obtained from the furanone (**2**) which was prepared from the glycosylfuran (**1**). The synthesis of 3-(2-deoxy-β-D-*erythro*-pentofuranosyl)pyridazin-6(1*H*)-one (**12**) from (**4**) is described. Catalytic hydrogenation of the γ-keto butenoic acid (**3**) afforded the γ-keto butyric acid (**4**) and its α isomer (**5**) in a 1:1 ratio. Treatment of ester (**6**) with hydrazine hydrate in methanol afforded the dihydropyridazinone (**8**) in 74% yield. Aromatization of compound (**8**) with 2,3-dichloro-5,6-dicyano-1,4-benzoquinone gave the pyridazinone (**10**) in 87% yield. Deprotection of compound (**10**) with methanolic sodium hydroxide afforded compound (**12**) in 77% yield.

During our efforts to develop a general synthetic method for *C*-nucleosides, we have prepared an extremely useful intermediate 2-(2,3,5-tri-*O*-benzoyl-β-D-ribofuranosyl)furan (**1**), from which some novel *C*-nucleosides have been synthesized.[1] We described earlier the synthesis and ring transformation of the furanone glycoside 5-hydroxy-2-(2,3,5-tri-*O*-benzoyl-β-D-ribofuranosyl)-furan-2(5*H*)-one (**2**), which can be obtained from (**1**) by oxidation of its furan ring.[2] Compound (**2**) was converted by elimination into 4-(1,4-anhydro-3,5-di-*O*-benzoyl-2-deoxy-D-*erythro*-pent-1-enofuranosyl)-4-oxobut-2-enoic acid (**3**), which represents a convenient precursor of 2′-deoxy-β-D-ribofuranosyl-*C*-nucleosides, exemplified here by its conversion into 3-(2′-deoxy-β-D-*erythro*-pentofuranosyl)pyridazin-6(1*H*)-one (**12**).



(**1**)



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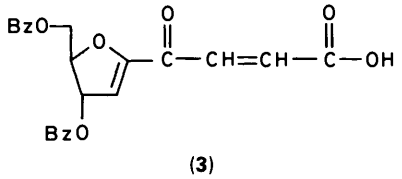
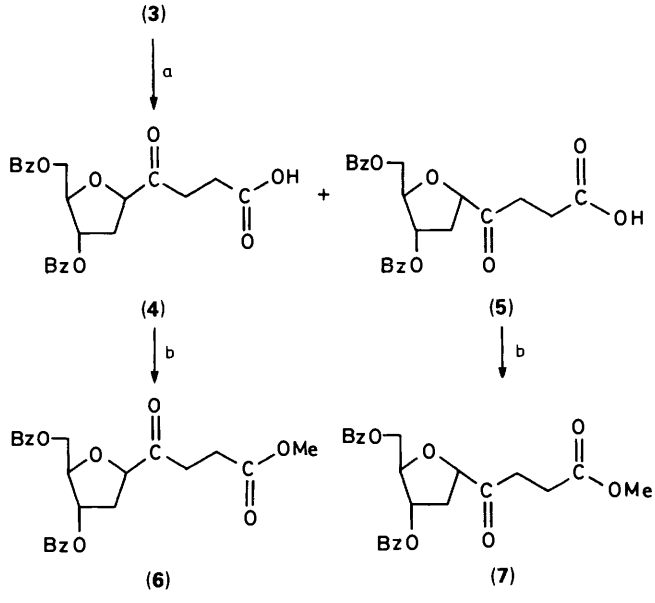


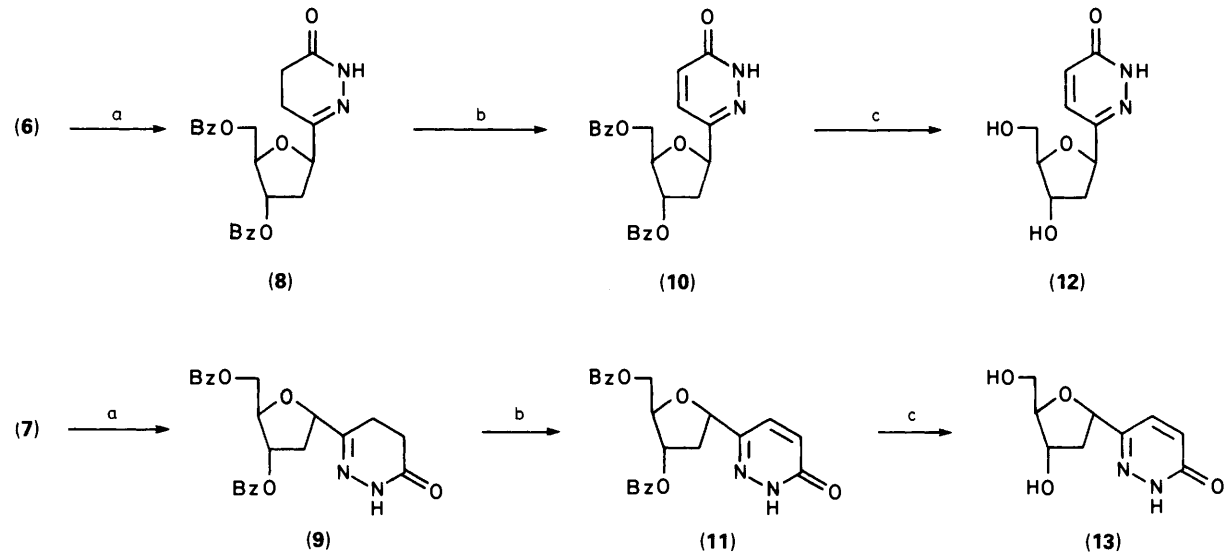
(**3**)

Catalytic hydrogenation of γ-keto butenoic acid (**3**) afforded two relatively unstable major products. These were readily separable by preparative t.l.c. (p.l.c.) and identified as 4-(3,5-di-*O*-benzoyl-2-deoxy-β-D-*erythro*-pentofuranosyl)-4-oxobutyric acid (**4**) and its α isomer (**5**) in a combined yield of 43% and in a 1:1 ratio (Scheme 1). For the assignment of anomeric configuration by ^{1}H n.m.r. spectra, Srivastava and Robins[3] observed that the absorption of 2′- and 2″-H for α anomers extends to both higher and lower fields, providing a large absorption band width as compared with that of the corresponding β anomers (Method A). Another method of assignment of anomeric configuration was proposed by Igolen and co-workers.[4] They observed that the 1′-H signal was broader for the β anomer than for the corresponding α anomer (Method B). Attempts to assign the configuration of compounds (**4**) and (**5**) by the ^{1}H n.m.r. spectra on the basis of the criteria previously described were not practical, since the signal for 1′-, 2′-, and 2″-H in compounds (**4**) and (**5**) appeared as a complex multiplet. Treatment of the butyric acid (**4**) with hydrochloric acid in methanol at room temperature for 12 h afforded the corresponding ester (**6**) in 96% yield. To establish the anomeric configuration of compound (**6**), we prepared the isomer (**7**) starting from (**5**) by the method used for the preparation of



Scheme 1. *Reagents:* a, Pd–C, H_2/THF–EtOH; b, HCl–MeOH

† Part 11, preceding paper.



Scheme 2. *Reagents and conditions:* a, $H_2NNH_2 \cdot H_2O$, MeOH; b, DDQ, benzene, reflux; c, 1M-NaOH–MeOH

Table. Values of $J_{1',2'} + J_{1',2''}$ and $\Delta\delta_{2',2''}$ for anomeric pairs of 2-deoxy-D-ribofuranosyl *C*-nucleosides

Compound	$\Delta\delta_{2',2''}$ (Method A)	$J_{1',2'} + J_{1',2''}$ (Method B)
(8) (β)	*a*	15.8
(9) (α)	*a*	13.5
(10) (β)	0.16	16.1
(11) (α)	0.27	12.8
(12) (β)	0.04[b]	16.2
(13) (α)	0.54	13.4

[a] Unresolved. [b] Clustered multiplets.

compound (**6**). In the ^1H n.m.r. of esters (**6**) and (**7**) the signals for 2′- and 2″-H appeared as a complex multiplet at δ 2.39—2.96 and 2.52—2.64. However, compound (**6**) exhibited a doublet doublet centred at δ 4.71 (1′-H, $J_{1'-2',2''}$ 9.9 Hz and 7.1 Hz, peak width 17.0 Hz). This pattern was very similar to that for compound (**7**), which also exhibited a doublet doublet centred at δ 4.72 (1′-H, $J_{1'-2',2''}$ 9.1 Hz and 3.7 Hz, peak width 12.8 Hz). These results indicate that the configuration of compounds (**6**) and (**7**) are the β and α deoxyribo-*C*-nucleoside, respectively (from Method B).

Reaction of β ester (**6**) with hydrazine hydrate in methanol at room temperature gave 3-(3,5-di-*O*-benzoyl-2-deoxy-β-D-*erythro*-pentofuranosyl)-4,5-dihydropyridazin-6(1*H*)-one (**8**) in 74% yield. The dehydrogenation was carried out by treatment of the dihydropyridazinone (**8**) with 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) and toluene-*p*-sulphonic acid (PTSA) in refluxing benzene to give 3-(3,5-di-*O*-benzoyl-2-deoxy-β-D-*erythro*-pentofuranosyl)pyridazin-6(1*H*)-one (**10**), obtained in 87% yield after p.l.c. purification. The structure of the pyridazinone (**10**) was confirmed by ^1H and ^{13}C n.m.r. and mass spectra. Of particular significance in the ^1H n.m.r. spectrum of compound (**10**) was the presence of doublet appearing at δ 6.91 (*J* 10.1 Hz), which afforded conclusive evidence for the aromatization of the dihydropyridazinone (**8**). Deprotection of compound (**10**) with methanolic sodium hydroxide afforded the pyridazinone (**12**) in 77% yield (Scheme 2). It is reasonable to assume that compounds (**8**), (**10**), and (**12**) have the β configuration, since complete inversion to the other isomer (*i.e.*, from β to α) under the reaction conditions would be highly unlikely. To confirm this, we also attempted to prepare the corresponding α isomers from keto ester (**7**) by procedures analogous to those for the preparations of the β isomers. We have examined the ^1H n.m.r. of several anomeric pairs of 2′-deoxy-*C*-nucleosides (Table) and observed that the absorption of 2′ and 2″-H for α anomers (**11**) and (**13**) extends to both higher and lower fields, giving a larger absorption band width and Δδ as compared with the corresponding β anomers (**10**) and (**12**). The value of $J_{1',2'} + J_{1',2''}$ was broader for the β anomers (**6**), (**8**), (**10**), and (**12**) than for the corresponding α anomers (**7**), (**9**), (**11**), and (**13**). Another technique utilized for assignment of anomeric configuration is an application of the n.O.e. Irradiation of the anomeric proton (δ 5.18, 1′-H) in the pyridazinone (**10**) gave an 8% enhancement of the signal at δ 2.57 assignable to 2′-H and a 5% enhancement of the signal at δ 4.55 assignable to 4′-H (Figure). These data indicate that compound (**10**) is β configuration.

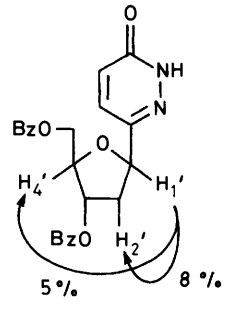


Figure. N.O.e. experiment of compound (**10**)

Experimental

M.p.s were determined on a Yanagimoto apparatus and are uncorrected. Mass spectra were obtained on Hitachi M-52 or M-80 spectrometers. ^1H N.m.r. spectra were measured with JNM-GX-270 and GX-400 (JEOL) spectrometers, with tetramethylsilane as internal standard. ^{13}C N.m.r. spectra were recorded on a JEOL JNM-FX-100 Fourier transform spectrometer operating at 25.00 MHz, with tetramethylsilane as internal standard. Elemental analyses were determined by the analytical centre of this faculty. Analytical t.l.c. was performed on glass plates coated with a 0.5-mm layer of silica gel GF_{254} (Merck). The compounds were detected by u.v. light (254 nm). Column chromatography was performed on silica gel C-200 (74—149 μm, Wakogel).

4-(3,5-*Di*-O-*benzoyl*-2-*deoxy*-β- *and* -α-D-erythro-*pentofuranosyl*)-4-*oxobutyric Acid* (**4**) *and* (**5**).—To a suspension of 10% palladium–carbon (36 mg) in tetrahydrofuran–methanol (1:1; 5 ml) was added a solution of the γ-keto butenoic acid (**3**) (87 mg, 0.2 mmol) in the same solvent (5 ml) and the mixture was stirred under hydrogen at atmospheric pressure for 12 h. After the catalyst was removed by filtration, the solvent was evaporated off under reduced pressure. T.l.c. (chloroform–methanol; 9:1) showed that the syrup contained two components (R_F 0.25 and 0.20). The mixture was separated by p.l.c. with hexane–ethyl acetate (2:1) as developer (× 3). Due to the lability of these compounds good elemental analyses could not be obtained.

Compound (**4**) (18.4 mg, 21%); R_F 0.25; syrup; $\delta_H(CDCl_3)$ 2.38—2.67 (6 H, m, 2- and 3-H_2, and 2'- and 2''-H), 4.56—4.65 (4 H, m, 1'- and 4'-H, and 5'-H_2), 5.55 (1 H, m, 3'-H), and 7.36—8.05 (10 H, m, ArH); $\delta_C(CDCl_3)$ 27.26, 32.47, and 35.22 (C-2, -2', and -3), 64.53 (C-5'), 76.17, 83.31, and 83.78 (C-1', -3', and -4'), 128.53—133.50 (C-Ar), 165.97 and 166.32 (C=O), 177.38 (C-1), and 217.40 (C-4).

Compound (**5**) (19.1 mg, 22%); R_F 0.20; syrup; $\delta_H(CDCl_3)$ 2.59—2.95 (6 H, m, 2- and 3-H_2, and 2'- and 2''-H), 4.45—4.70 (4 H, m, 1'- and 4'-H, and 5'-H_2), 5.52 (1 H, m, 3'-H), and 7.40—8.07 (10 H, m, ArH); $\delta_C(CDCl_3)$ 27.38, 32.88, and 35.10 (C-2, -2', and -3), 64.47 (C-5'), 75.88, 83.25, and 83.54 (C-1', -3', and -4'), 128.53—133.50 (C-Ar), 165.80 and 166.21 (C=O), 177.97 (C-1), and 215.70 (C-4).

Methyl 4-(3,5-*Di*-O-*benzoyl*-2-*deoxy*-β- *and* -α-D-erythro-*pentofuranosyl*)-4-*oxobutyrate* (**6**) *and* (**7**).—Saturated hydrogen chloride–methanol (20 drops) was added to a solution of the acid (**4**) (96 mg, 0.2 mmol) in methanol (10 ml) at 0 °C, and the resulting solution was stirred at room temperature for 12 h. The reaction mixture was neutralized with sodium hydrogen carbonate and then extracted with chloroform (3 × 10 ml). The extracts were combined, washed with water, dried ($MgSO_4$), and evaporated under reduced pressure to afford a syrup, which was purified by p.l.c. with hexane–ethyl acetate (4:1) as developer (× 2).

Compound (**6**) (95 mg, 96%); R_F 0.28; syrup (Found: C, 63.9; H, 5.4. $C_{24}H_{24}O_8 \cdot H_2O$ requires C, 63.87; H, 5.72%); $\delta_H(CDCl_3)$ 2.39—2.96 (6 H, m, 2- and 3-H_2, and 2'- and 2''-H), 3.64 (3 H, s, OMe), 4.53—4.61 (3 H, m, 4'- and 5'-H_2), 4.71 (1 H, dd, $J_{1',2''}$ 7.1 and $J_{1',2'}$ 9.9 Hz, 1'-H), 5.55 (1 H, m, 3'-H), and 7.42—8.06 (10 H, m, ArH); $\delta_C(CDCl_3)$ 27.09, 32.94, and 35.57 (C-2, -2', and -3), 51.72 (OMe), 64.53 (C-5'), 76.17, 83.54, and 83.72 (C-1', -3', and -4'), 128.53—133.44 (C-Ar), 165.91 and 166.15 (C=O), 177.28 (C-1), and 208.50 (C-4).

In the same manner the α isomer (**7**) (82 mg, 91%) was obtained as a syrup from the acid (**5**) (87 mg, 0.02 mmol); R_F 0.22 (Found: C, 64.1; H, 5.6. $C_{24}H_{24}O_8$ requires C, 63.87; H, 5.72%); $\delta_H(CDCl_3)$ 2.52—2.64 (3 H, m, 2-H_2 and 2''-H), 2.71 (1 H, ddd, $J_{2',3'}$ 6.1, $J_{1',2'}$ 9.1, and $J_{2',2''}$ 15.1 Hz, 2'-H), 2.90—3.10 (2 H, m, 3-H_2), 3.61 (3 H, s, OMe), 4.52—4.65 (3 H, m, 4'-H and 5'-H_2), 4.72 (1 H, dd, $J_{1',2''}$ 3.7 and $J_{1',2'}$ 9.1 Hz, 1'-H), 5.52 (1 H, m, 3'-H), and 7.40—8.11 (10 H, m, ArH); $\delta_C(CDCl_3)$ 27.32, 33.17, and 35.16 (C-2, -2', and -3), 51.66 (OMe), 64.41 (C-5'), 75.88, 83.25, and 83.66 (C-1', -3', and -4'), 128.47—133.39 (C-Ar), 165.74 and 166.15 (C=O), 172.93 (C-1), and 210.08 (C-4).

3-(3,5-*Di*-O-*benzoyl*-2-*deoxy*-β- *and* -α-D-erythro-*pentofuranosyl*)-4,5-*dihydropyridazin*-6(1H)-*one* (**8**) *and* (**9**).—To a solution of the ester (**6**) (149 mg, 0.34 mmol) in methanol (5 ml) at 0—5 °C was added a solution of hydrazine hydrate (21 mg, 0.42 mmol) in methanol (1 ml), and the mixture was kept at room temperature for 12 h. The reaction mixture was evaporated to dryness under reduced pressure to afford a syrup, which was purified by p.l.c. with hexane–ethyl acetate (1:1) as developer (× 2).

Compound (**8**) (107 mg, 74%); R_F 0.25; syrup (Found: C, 64.0; H, 5.4; N, 6.5. $C_{23}H_{22}N_2O_6 \cdot \frac{1}{2}H_2O$ requires C, 63.65; H, 5.21; N, 6.30%); $\delta_H(CDCl_3)$ 2.36—2.62 (6 H, m, 4- and 5-H_2, and 2'- and 2''-H), 4.47—4.56 (2 H, m, 4'- and 5'-H), 4.85 (1 H, dd, $J_{1',2''}$ 6.2 and $J_{1',2'}$ 9.6 Hz, 1'-H), 5.60 (1 H, m, 3'-H), 7.43—8.07 (10 H, m, ArH), and 8.29 (1 H, s, NH); $\delta_C(CDCl_3)$ 20.59 and 26.03 (C-4 and -5), 35.86 (C-2'), 64.47 (C-5'), 76.64, 80.15, and 83.13 (C-1', -3', and -4'), 128.53—133.34 (C-Ar), 153.22 (C-3), 166.15 and 167.61 (C-6 and C=O).

In the same manner the α isomer (**9**) (132 mg, 87%) was obtained as a syrup from the ester (**7**) (162 mg, 0.032 mmol); R_F 0.20 (Found: C, 62.9; H, 5.1; N, 6.2. $C_{23}H_{22}N_2O_6 \cdot H_2O$ requires C, 62.74; H, 5.49; N, 6.36%); $\delta_H(CDCl_3)$ 2.44—2.53 (3 H, m, 4-H_2 and 2''-H), 2.66—2.82 (3 H, m, 2'-H and 5-H_2), 4.50—4.58 (3 H, m, 4'-H and 5'-H_2), 4.91 (1 H, dd, $J_{1',2''}$ 5.4 and $J_{1',2'}$ 8.1 Hz, 1'-H), 5.56 (1 H, m, 3'-H), 7.36—8.07 (10 H, m, ArH), and 8.38 (1 H, s, NH); $\delta_C(CDCl_3)$ 20.94 and 26.15 (C-4 and -5), 35.39 (C-2'), 64.41 (C-5'), 76.17, 79.74, and 82.31 (C-1', -3', and -4'), 128.53—133.50 (C-Ar), 154.21 (C-3), and 165.97, 166.26, and 167.49 (C-6 and C=O).

3-(3,5-*Di*-O-*benzoyl*-2-*deoxy*-β- *and* -α-D-erythro-*pentofuranosyl*)*pyridazin*-6(1H)-*one* (**10**) *and* (**11**).—A solution of the dihydropyridazinone (**8**) (30 mg, 0.07 mmol), DDQ (32.3 mg, 0.14 mmol) and PTSA (13.5 mg, 0.07 mmol) in benzene (2 ml) was heated under reflux for 2 h. The reaction mixture was evaporated to dryness under reduced pressure to afford a syrup, which was purified by p.l.c. with hexane–ethyl acetate (1:1) as developer (× 2).

Compound (**10**) (26 mg, 87%); R_F 0.24; syrup (Found: M^+, 420.1315. $C_{23}H_{20}N_2O_6$ requires M, 420.1319); $\delta_H(CDCl_3)$ 2.41 (1 H, ddd, $J_{2'',3'}$ 6.1, $J_{1',2''}$ 10.4, and $J_{2',2''}$ 14.0 Hz, 2''-H), 2.57 (1 H, ddd, $J_{2',3'}$ 1.0, $J_{1',2'}$ 5.7, and $J_{2',2''}$ 14.0 Hz, 2'-H), 4.53—4.70 (3 H, m, 4'-H and 5'-H_2), 5.18 (1 H, dd, $J_{1',2'}$ 5.7 and $J_{1',2''}$ 10.4 Hz, 1'-H), 5.63 (1 H, apparent d, 3'-H), 6.91 (1 H, d, J 10.1 Hz, 5-H), 7.42—8.09 (11 H, m, 4-H and ArH), and 12.00 (1 H, br s, NH); $\delta_C(CDCl_3)$ 38.02 (C-2'), 64.59 (C-5'), 76.76, 78.39, and 83.48 (C-1', -3', and -4'), 128.53—133.50 (C-Ar), 130.58 and 131.63 (C-4 and -5), 147.49 (C-3), and 162.29, 166.03, and 166.15 (C-6 and C=O).

In the same manner the α isomer (**11**) (227 mg, 93%) was obtained as a syrup from the dihydropyridazinone (**9**) (245 mg, 0.058 mmol); R_F 0.24 (Found: M^+, 420.1302); $\delta_H(CDCl_3)$ 2.62 (1 H, ddd, $J_{2'',3}$ 3.0, $J_{1',2''}$ 4.7, and $J_{2',2''}$ 14.4 Hz, 2''-H), 2.89 (1 H, ddd, $J_{2',3'}$ 6.7, $J_{1',2'}$ 8.1, and $J_{2',2''}$ 14.4 Hz, 2'-H), 4.54—4.65 (3 H, m, 4'-H and 5'-H_2), 5.26 (1 H, dd, $J_{1',2''}$ 4.7 and $J_{1',2'}$ 8.1 Hz, 1'-H), 5.60 (1 H, m, 3'-H), 7.00 (1 H, d, J 9.8 Hz, 5-H), 7.37—8.12 (11 H, m, 4-H and ArH), and 12.27 (1 H, br s, NH); $\delta_C(CDCl_3)$ 37.15 (C-2'), 64.47 (C-5'), 76.29, 78.57, and 82.78 (C-1', -3', and -4'), 128.53—133.44 (C-Ar), 130.27 and 131.18 (C-4 and -5), 149.01 (C-3), and 162.35, 165.86, and 166.21 (C-6 and C=O).

3-(2-*Deoxy*-β- *and* -α-D-erythro-*pentofuranosyl*)*pyridazin*-6(1H)-*one* (**12**) *and* (**13**).—To a solution of the di-*O*-benzoate (**10**) (124 mg, 0.58 mmol) in methanol (10 ml) at 0 °C was added 1M-NaOH (1.5 ml) during 2 h, and the mixture was then rendered neutral with acetic acid. The reaction mixture was evaporated to dryness under reduced pressure to afford a syrup, which was purified by p.l.c. with chloroform–methanol (6:1) as developer (× 2).

Compound (**12**) (48 mg, 77%); R_F 0.35; syrup (Found: M^+, 212.0818. $C_9H_{12}N_2O_4$ requires M, 212.0796); $\delta_H(CD_3OD)$ 2.11—2.20 (2 H, m, 2'- and 2''-H), 3.66 (2 H, m, 5'-H_2), 3.95 (1 H, m, 4'-H), 4.35 (1 H, m, 3'-H), 5.02 (1 H, dd, $J_{1',2''}$ 7.4 and $J_{1',2'}$ 8.8 Hz, 1'-H), 6.97 (1 H, d, J 9.8 Hz, 5-H), and 7.71 (1 H, d, J 9.8 Hz, 4-H); $\delta_C(CD_3OD)$ 41.95 (C-2'), 63.77 (C-5'), 74.18, 79.80,

and 87.78 (C-1′, -3′, and -4′), 130.93 and 133.85 (C-4 and -5), 150.59 (C-3), and 163.81 (C=O).

In the same manner the α isomer (**13**) (53 mg, 64%) was obtained as a syrup from the di-*O*-benzoate (**11**) (162 mg, 0.39 mmol); R_F 0.35 (Found: M^+, 212.0773); $\delta_H(CD_3OD)$ 2.07 (1 H, ddd, $J_{2'',3'}$ 5.0, $J_{1',2''}$ 6.7, and $J_{2',2''}$ 14.1 Hz, 2″-H), 2.61 (1 H, dt, $J_{2'-1'3'}$ 6.7 and $J_{2',2''}$ 14.1 Hz, 2′-H), 3.56—3.70 (2 H, m, 5′-H_2), 3.99 (1 H, m, 4′-H), 4.35 (1 H, m, 3′-H), 4.99 (1 H, t, $J_{1'-2',2''}$ 6.7 Hz, 1′-H), 6.97 (1 H, d, *J* 9.8 Hz, 5-H), and 7.76 (1 H, d, *J* 9.8 Hz, 4-H); $\delta_C(CD_3OD)$ 41.36 (C-2′), 63.30 (C-5′), 73.42, 79.45, and 88.51 (C-1′, -3′, and -4′), 130.93 and 133.91 (C-4 and -5), 151.81 (C-3), and 163.63 (C=O).

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