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Note

Synthesis of 2-(D-*allo*- and D-*altro*-pentitol-1-yl)pyridines and their 2′,5′-anhydro derivatives

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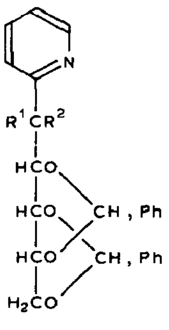
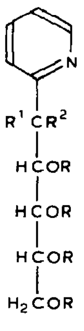
The fusion of 2-trimethylsilylpyridine (**1**) with 2,4:3,5-di-*O*-benzylidene-*aldehydo*-D-ribose (**2**) by Ogawa *et al.*[1,2] gave a mixture of the D-*allo* and D-*altro* isomers (**3** and **4**) of 2-(2,4:3,5-di-*O*-benzylidenepentitol-1-yl)pyridine which was not fractionated. These compounds can be considered as acyclic sugar *C*-nucleosides, and can be converted into cyclic *C*-nucleosides by acid-catalysed intramolecular dehydration of the polyhydroxyalkyl chain[3–10]. The cyclisation of the pentahydroxypentyl chains joined to π-excessive heterocycles produces an anomeric mixture of 1′,4′- and/or 1′,5′-anhydro derivatives, depending on the



3 $R^1 = H, R^2 = OH$
4 $R^1 = OH, R^2 = H$



5 $R = R^1 = H, R^2 = OH$
6 $R = R^2 = H, R^1 = OH$

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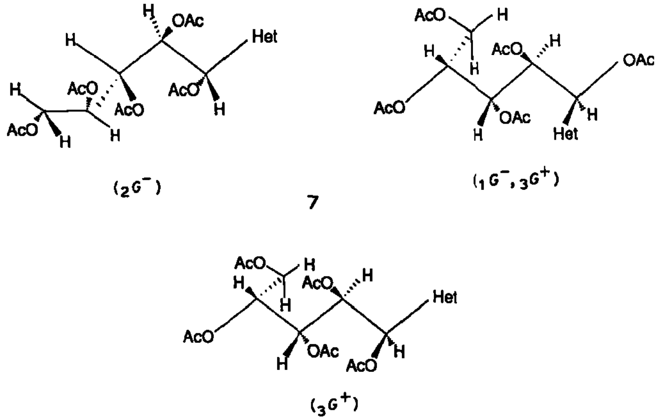
reaction conditions[3–8]. We now describe the preparation of 2′,5′-anhydro derivatives from **3** and **4**. Recently, Belmans *et al.*[11] failed to cyclodehydrate the epimeric mixture of **3** and **4** using HCl under various conditions.

The condensation of **1** and **2** gave two major products **3** and **4** (combined yield, 60%) with similar polarities, which could be separated partially by chromatography on silica gel. The overlapping fractions were resolved by preparative t.l.c. The assignment of configurations to **3** and **4** was based on the configuration of the sugar precursor and is consistent with the Richtmyer–Hudson rules[12] as applied to **5** and **6**. The presence of the pentahydroxypentyl side-chain was proved by the consumption of 4 mol. equiv. of periodate and by the preparation of the penta-acetates **7** and **8**.

The *J* values for the chain protons (Table I) show that the D-*allo* isomer **7** exists as a mixture of the double-sickle ${}_1G^-$, ${}_3G^+$, and ${}_2G^-$ forms[13,14] (Scheme 1). In contrast, the data for the D-*altro* isomer **8** (Table I) indicate conformational homogeneity. The small value (3.3 Hz) of $J_{3',4'}$ shows that H-3′ and H-4′ are not antiperiplanar, and the data are accommodated by the ${}_3G^+$ conformation (Scheme 1) which is free from *syn*-parallel 1,3-interactions.

Treatment of **5** and **6** for several days with boiling 2-propanol–sulphuric acid afforded mainly the 2′,5′-anhydro derivatives **9** and **10**, respectively, each of which consumed 1 mol. equiv. of periodate, indicating the presence of either a 1′,4′- or a 2′,5′-anhydro ring. The ^{1}H- and ^{13}C-n.m.r. data for the triacetates **11** and **12** showed that the dehydration had occurred between C-2′ and C-5′ (see Tables I and II). Thus, the resonance for H-2′ in **11** and **12** appeared at higher field than those for H-1′,3′,4′, and H-1′,3′,4′ but not H-2′,5′,5″ were deshielded by ~1 p.p.m. relative to the resonances in **9** and **10**[9,10]. An acyloxy group causes[15] a down-field (1.5–4 p.p.m.) shift of the resonance of the α-carbon and an up-field shift (1–5 p.p.m.) of the resonance of the β-carbon atom. Thus, the signals of C-2′ in **11** and **12** (two acetoxyl groups in the β-position) were shifted furthest up-field, whereas



7

8

Scheme 1

TABLE I

^{1}H-N.M.R. DATA FOR **3–12**

Compound	H-1	H-2	H-3	H-4	H-5	H-5'	OH	OAc	CHPh	Pyridine				-Ph
										H-6	H-4	H-3	H-5	
3[a]	5.21m	← 4.90–3.70m →							5.76s 5.50s	8.55m	← 7.90–6.95m →			
4[a]	5.11bd $J_{1,2}$ 1 $J_{1,OH}$ 6.0	← 4.50–5.05m →					4.15d		5.68s 5.62s	8.55dq $J_{3,6}$ 1.0 $J_{4,6}$ 2.0 $J_{5,6}$ 4.6	7.66dt $J_{3,4}$ 8.0 $J_{4,5}$ 8.1	← 7.60–6.90m →		
5[b]	4.73t $J_{1,2} = J_{1,OH} = 4.5$	← 4.00–3.30m →					5.44d J 5.5 4.82d J 5.1 4.77d J 5.0 4.58d J 4.7 4.31t $J_{5,OH}$ 5.1 $J_{5',OH}$ 5.1			8.48dq $J_{3,6}$ 1.0 $J_{4,6}$ 2.0 $J_{5,6}$ 4.6	7.67dt $J_{3,4}$ 8.0 $J_{4,5}$ 8.0	7.50bd	7.24dq $J_{3,5}$ 1.7	
6[b]	4.89m $J_{1,2}$ 1	← 5.80–2.90m →								8.47dq $J_{3,6}$ 1.0 $J_{4,6}$ 2.0 $J_{5,6}$ 4.6	7.73dt $J_{3,4}$ 8.0 $J_{4,5}$ 8.0	7.50bd	7.18dq $J_{3,5}$ 1.7	
7[c,d]	6.09d $J_{1,2}$ 5.7	5.79t $J_{2,3}$ 5.7 $J_{2,4}$ −0.5	5.44dd $J_{3,4}$ 4.9 $J_{3,5}$ −0.4 $J_{3,5'}$ −0.4	5.37m $J_{4,5}$ 3.2 $J_{4,5'}$ 7.4	4.38dd $J_{5,5'}$ −12.3	4.12dd		2.17s 2.07s 2.04s 1.99s 1.87s		8.57dq $J_{3,6}$ 1.0 $J_{4,6}$ 1.8 $J_{5,6}$ 4.7	7.67dq $J_{3,4}$ 7.8 $J_{4,5}$ 7.4	7.30bd	7.19dq $J_{3,5}$ 1.3	
7[e]	6.09d $J_{1,2}$ 5.7	5.79t $J_{2,3}$ 5.7	5.45dd $J_{3,4}$ 4.7	5.37m	4.38dd $J_{4,5}$ 3.1 $J_{5,5'}$ −12.1	4.11dd $J_{4,5'}$ 6.9		2.18s 2.08s 2.04s 1.99s 1.87s		8.60dq $J_{3,6}$ 1.0 $J_{4,6}$ 1.8 $J_{5,6}$ 4.7	7.69dq $J_{3,4}$ 7.8 $J_{4,5}$ 7.4	7.34bd	7.22dq $J_{3,5}$ 1.3	

8[e]	6.09d $J_{1,2}$ 3.3	5.71dd $J_{2,3}$ 8.2	5.41dd $J_{3,4}$ 3.3	5.24m	4.39dd $J_{4,5}$ 3.6 $J_{5,5'}$ −12.0	4.15dd $J_{4,5'}$ 7.2		2.17s 2.09s 2.02s 1.94s	8.59dq $J_{3,6}$ 1.0 $J_{4,6}$ 2.0 $J_{5,6}$ 4.7	7.68dq $J_{3,4}$ 8.1 $J_{4,5}$ 7.4	7.23m	7.22dq $J_{3,5}$ 1.3
9[f]	4.65d $J_{1,2}$ 3.2 $J_{1,OH}$ 1	← 4.15–3.20m →					5.80–3.20m		8.46dq $J_{3,6}$ 0.9 $J_{4,6}$ 1.7 $J_{5,6}$ 4.6	7.76dq $J_{3,4}$ 7.8 $J_{4,5}$ 7.4	7.47m	7.22dq $J_{3,5}$ 1.4
10[f]	4.65m $J_{1,2}$ 2.4	← 4.30–3.70m →			3.86dd $J_{4,5}$ 4.6 $J_{5,5'}$ −8.8	3.43dd $J_{4,5'}$ 2.9	5.70–4.40m		8.46dq $J_{3,6}$ 1.0 $J_{4,6}$ 1.8 $J_{5,6}$ 4.8	7.73dq $J_{3,4}$ 7.8 $J_{4,5}$ 7.2	7.49m	7.20dq $J_{3,5}$ 1.4
11[c]	6.03d $J_{1,2}$ 4.1	4.54dd $J_{2,3}$ 6.4	5.50dd $J_{3,4}$ 5.4	5.30m	4.10dd $J_{4,5}$ 4.7 $J_{5,5'}$ −10.0	3.83dd $J_{4,5'}$ 3.8		2.17s 2.05s 1.87s	8.60dq $J_{3,6}$ 1.0 $J_{4,6}$ 1.8 $J_{5,6}$ 4.8	7.69dq $J_{3,4}$ 7.8 $J_{4,5}$ 7.2	7.33m	7.21dq $J_{3,5}$ 1.4
12[c]	5.90d $J_{1,2}$ 4.2	4.52dd $J_{2,3}$ 6.5	← 5.50–5.15m →		4.22dd $J_{4,5}$ 4.7 $J_{5,5'}$ −10.6	3.84dd $J_{4,5'}$ 3.1		2.21s 2.05s 1.94s	8.59dq $J_{3,6}$ 1.0 $J_{4,6}$ 2.0 $J_{5,6}$ 4.6	7.70dt $J_{3,4}$ 8.0 $J_{4,5}$ 8.0	7.31m	7.21dq $J_{3,5}$ 1.3

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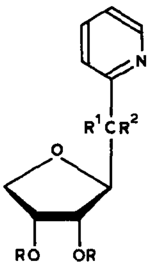
[a] $CDCl_3$ at 90 MHz. [b] $(CD_3)_2SO$ at 90 MHz. [c] $CDCl_3$ at 80.13 MHz. [d] *J* Analysis of the spectrum was carried out by a Panic Program on the Aspect 2000. [e] $CDCl_3$ at 200 MHz. [f] $(CD_3)_2SO$ at 80.13 MHz.

TABLE II
^{13}C-N.M.R. DATA FOR **3–12**

Compound	*C-1*	*C-2*	*C-3*	*C-4*	*C-5*	*COCH$_3$*	*COCH$_3$*	*CHPh*	*Pyridine*					*Benzene*			
									C-2	*C-3*	*C-4*	*C-5*	*C-6*	*C-o*	*C-m*	*C-p*	*C-i*
3[a]	73.7	81.2	72.8*[e]	72.4*	66.6			101.7 101.5	157.7	122.2*	136.3	121.1*	148.0	128.0 127.7	126.1 125.6	129.0 128.6	136.7 136.6
4[a]	73.8	80.6	72.8*	70.4*	66.7			101.7 100.9	159.9	122.3*	136.4	120.7*	148.1	128.1 128.0	126.0 125.8	128.9 128.8	137.1 136.7
5[b]	75.5	73.4*	72.7*	72.0*	62.9				162.1	122.4*	136.3	122.1*	147.6				
6[b]	74.9	73.4*	72.7*	72.0*	62.9				163.5	121.9*	136.3	121.6*	148.2				
7[c]	74.3	72.0*	70.3*	69.7*	62.1	170.4 169.9 169.6 169.1	20.8 20.6		155.9	123.1*	136.4	121.6*	149.4				
8[c]	73.6	71.5*	70.2*	69.2*	61.7	170.3 169.7 169.6 169.3 168.8	20.6 20.5 20.2		155.9	123.1*	136.4	121.0*	149.5				
9[d]	74.0	86.8	70.5*	70.4*	71.5				161.1	121.8*	135.9	121.1*	147.8				
10[d]	72.6*	84.9	71.5*	70.4*	72.3				162.2	121.6*	135.8	120.8*	147.7				
11[c]	75.3	81.3	71.4*	71.3*	70.6	169.6 169.4 169.0	20.7 20.3 20.0		155.8	122.8*	136.3	121.5*	149.2				
12[c]	75.4	81.1	71.8*	71.5*	70.9	169.8 169.3 168.9	20.8 20.5 20.1		156.5	122.9*	136.5	121.3*	149.3				

[a]CDCl$_3$ at 50.3 MHz. [b](CD$_3$)$_2$SO at 50.3 MHz. [c]CDCl$_3$ at 20.15 MHz. [d](CD$_3$)$_2$SO at 20.15 MHz. [e]Assignments marked * may have to be interchanged.



9 $R = R^1 = H, R^2 = OH$
10 $R = R^2 = H, R^1 = OH$
11 $R = Ac, R^1 = H, R^2 = OAc$
12 $R = Ac, R^2 = H, R^1 = OAc$

the signals of C-1′ (one acetoxyl group in the α-position) were shifted furthest down-field. The signal for C-5′ in **9** and **10** was found at 71.5 and 72.3 p.p.m., whereas a signal would be expected at 60–64 p.p.m. for a 1′,4′-anhydro structure since C-5′ would then carry a hydroxyl group[16,17].

The β configuration at C-2′ was assigned on the basis of the known mechanism for the acid-catalysed cyclisation of simple pentitols[18]. The cyclisation reaction of alditols has been suggested to proceed *via* an S_N2 process by displacement of the protonated HO-1′ by HO-4′. In our compounds, protonated HO-5′ would be displaced by HO-2′, thus leading to 2′,5′-anhydro derivatives with the β configuration (retention of configuration). A direct S_N2 displacement reaction has also been postulated for the acid-catalysed cyclisation of 6-azauracils prepared by Bobek *et al.*[10].

The mass spectra of **11** and **12** contained, *inter alia,* weak signals for the molecular ion at *m/z* 337 (0.7, 0.3% of the base peak, *m/z* 43, Ac^+) and ions at *m/z* 187 (60.9, 41.6%) and 151 (48.1, 19.6%) formed by cleavage of the C-1′–C-2′ bond which appears to be characteristic of homonucleosides having this general type of structure[19].

EXPERIMENTAL

General methods. — Solutions were concentrated *in vacuo* at <40°. Melting points were determined with a Gallenkamp apparatus, and are uncorrected. Optical rotations were measured at 22 ±5° with a Perkin–Elmer 141 polarimeter (10-cm cell). T.l.c. was performed on Silica Gel GF_{254} (Merck) with detection by u.v. light or iodine vapour. Column chromatography was performed in the "flash" mode[20], using 10:1 benzene–ethyl acetate. Preparative t.l.c. was performed on Silica Gel PF_{254} (Merck). I.r. spectra (KBr discs or chloroform solutions) were recorded with a Perkin–Elmer 399 spectrometer, and the u.v. spectra with a Pye–Unicam SP8-250 instrument. ^1H-N.m.r. spectra (Table I) were recorded with Bruker WP-80-SY (80.13 MHz), Perkin–Elmer R-32 (90 MHz), and Varian XL-200

(200 MHz) instruments, and ^{13}C-n.m.r. spectra (Table II) with Bruker AC-200 (50.3 MHz) and WP-80-SY (20.15 MHz) instruments. Mass spectra were obtained using an AEI MS-30 spectrometer, operated at a resolution of 1000.

*2-(2,4:3,5-Di-O-benzylidene-*D-allo- (**3**) *and* -D-altro-*pentitol-1-yl)pyridine* (**4**). — A mixture of 2-trimethylsilylpyridine (12.0 g, 79.3 mmol) and 2,4:3,5-di-*O*-benzylidene-*aldehydo*-D-ribose (18.8 g, 57.6 mmol) was heated for 3 h at 110°, then cooled, and stirred with aqueous 50% ethanol (130 mL) and a catalytic amount of pyridinium trifluoroacetate until dissolution occurred. The solution was boiled under reflux for 7 h and then concentrated to dryness, and ethanol was evaporated several times from the residue, which was then crystallised from ethanol. Column chromatography (10:1 benzene–ethyl acetate) of the resulting mixture (14 g, 60%) gave, first, **4** (4.43 g), m.p. 166–168°, $[\alpha]_D$ −58°, $[\alpha]_{578}$ −62°, $[\alpha]_{546}$ −70°, $[\alpha]_{436}$ −120° (*c* 0.5, chloroform), R_F 0.24; λ_{max}^{EtOH} 256, 261, and 267 nm (ε_{mM} 3.7, 4.1, and 2.9); ν_{max} 3280 (OH), 1590, 1570, 1495, and 1470 cm^{-1} (C=C, C=N aromatic).

Anal. Calc. for $C_{24}H_{23}NO_5$: C, 71.10; H, 5.72; N, 3.46. Found: C, 70.80; H, 5.47; N, 3.45.

Eluted second was **3** (3.68 g), m.p. 151–153°, $[\alpha]_D$ −36°, $[\alpha]_{578}$ −37°, $[\alpha]_{546}$ −43°, $[\alpha]_{365}$ −131° (*c* 0.8, chloroform), R_F 0.15; λ_{max}^{EtOH} 257 nm (ε_{mM} 3.1); ν_{max} 3270 (OH), 1580, 1560, 1490, and 1470 cm^{-1} (C=C, C=N aromatic). The ^{1}H- and ^{13}C-n.m.r. data are given in Tables I and II, respectively.

Anal. Found: C, 70.91; H, 5.74; N, 3.63.

The overlapping fractions were purified by preparative t.l.c.

*2-(*D-allo-*Pentitol-1-yl)pyridine* (**5**). — A solution of **3** (1.7 g, 7.41 mmol) in aqueous 60% acetic acid (116 mL) was heated at 100° for 2.25 h and then concentrated to dryness, and ethanol was evaporated several times from the residue. The resulting oil was crystallised from methanol–acetone to give **5** (0.6 g, 61%), m.p. 93–95°, $[\alpha]_D$ +23°, $[\alpha]_{578}$ +24°, $[\alpha]_{546}$ +27°, $[\alpha]_{436}$ +45°, $[\alpha]_{365}$ +69° (*c* 0.54, water); λ_{max}^{EtOH} 256, 262, and 267 nm (ε_{mM} 3.3, 3.7, and 2.8); ν_{max} 3360 and 3240 (OH), 1590, 1570, and 1475 cm^{-1} (C=C, C=N aromatic). Periodate consumption, 3.99 mol/mol.

Anal. Calc. for $C_{10}H_{15}NO_5 \cdot CH_3OH$: C, 51.42; H, 6.98; N, 5.71. Found: C, 51.67; H, 6.76; N, 5.52.

The penta-acetate **7** of **5** had m.p. 65–68°, $[\alpha]_D$ +31°, $[\alpha]_{578}$ +34°, $[\alpha]_{546}$ +39°, $[\alpha]_{436}$ +76°, $[\alpha]_{365}$ +141° (*c* 0.2, chloroform); λ_{max}^{EtOH} 254, 260, and 265 nm (ε_{mM} 3.1, 3.4, and 2.5); ν_{max} 1745 (C=O), 1590, 1570, and 1470 cm^{-1} (C=C, C=N aromatic).

Anal. Calc. for $C_{20}H_{25}NO_{10}$: C, 54.67; H, 5.73; N, 3.20. Found: C, 54.55; H, 5.80; N, 3.14.

*2-(*D-altro-*Pentitol-1-yl)pyridine* (**6**). — Treatment of **4** (1.25 g), as described for **5**, gave **6** (0.46 g, 66%), m.p. 112–114° (from methanol–acetone), $[\alpha]_D$ −52°, $[\alpha]_{578}$ −55°, $[\alpha]_{546}$ −62°, $[\alpha]_{436}$ −112°, $[\alpha]_{365}$ −199° (*c* 0.5, water); λ_{max}^{EtOH} 257, 262, and 268 nm (ε_{mM} 3.1, 3.5, and 2.5); ν_{max} 3360 (OH), 1595, 1570, and 1475 cm^{-1} (C=C, C=N aromatic). Periodate consumption, 3.98 mol/mol.

Anal. Calc. for $C_{10}H_{15}NO_5$: C, 52.42; H, 6.59; N, 6.11. Found: C, 52.16; H, 6.80; N, 6.14.

The penta-acetate **8** of **6** had m.p. 68–70°, $[\alpha]_D$ +6°, $[\alpha]_{578}$ +5°, $[\alpha]_{546}$ +5°, $[\alpha]_{436}$ +2°, $[\alpha]_{365}$ −10° (*c* 0.5, chloroform); λ_{max}^{EtOH} 254, 260, and 266 nm (ε_{mM} 3.1, 3.6, and 2.7); ν_{max} 1735 (C=O), 1585, and 1470 cm^{-1} (C=C, C=N aromatic).

Anal. Found: C, 54.52; H, 5.79; N, 3.40.

*2-(2,5-Anhydro-*D-allo*-pentitol-1-yl)pyridine* (**9**). — A solution of **5** (0.67 g, 2.92 mmol) in 5% 2-propanol–sulphuric acid (136 mL) was boiled under reflux for 5 days, then neutralised ($NaHCO_3$), and concentrated under diminished pressure, and the residue was extracted with ethanol. The extract was filtered and concentrated to dryness, and the process was repeated until a homogeneous syrup was obtained. Preparative t.l.c. (5:1:1 chloroform–methanol–acetone) then gave **9** (0.21 g, 35%), m.p. 90–92° (from ethanol), $[\alpha]_D$ +1°, $[\alpha]_{578}$ +2°, $[\alpha]_{546}$ +3°, $[\alpha]_{436}$ +12°, $[\alpha]_{365}$ +38° (*c* 0.6, water); λ_{max}^{EtOH} 255, 260, and 265 nm (ε_{mM} 2.7, 3.1, and 2.3); ν_{max} 3320 (OH), 1595, 1570, and 1475 cm^{-1} (C=C, C=N aromatic) (Nujol). Periodate consumption, 0.98 mol/mol.

Anal. Calc. for $C_{10}H_{13}NO_{10}$: C, 56.86; H, 6.20; N, 6.63. Found: C, 56.62; H, 6.37; N, 6.45.

The triacetate **11** of **9** was isolated as a syrup, $[\alpha]_D$ −11°, $[\alpha]_{578}$ −11°, $[\alpha]_{546}$ −10°, $[\alpha]_{436}$ +4°, $[\alpha]_{365}$ +57° (*c* 0.3, chloroform); λ_{max}^{EtOH} 254, 259, and 264 nm (ε_{mM} 2.6, 2.8, and 2.0); ν_{max} 1740 (C=O), 1590, 1570, and 1475 cm^{-1} (C=C, C=N aromatic).

Anal. Calc. for $C_{16}H_{19}NO_7$: C, 56.97; H, 5.68; N, 4.15. Found: C, 56.78; H, 5.74; N, 4.19.

*2-(2,5-Anhydro-*D-altro*-pentitol-1-yl)pyridine* (**10**). — Treatment of **6** (0.46 g, 2.0 mmol) as described for **9** gave **10** (0.14 g, 33%), m.p. 102–104° (from ethanol), $[\alpha]_D$ −95°, $[\alpha]_{578}$ −98°, $[\alpha]_{546}$ −112°, $[\alpha]_{436}$ −194°, $[\alpha]_{365}$ −314° (*c* 0.5, water); λ_{max}^{EtOH} 254, 259, and 265 nm (ε_{mM} 3.5, 4.1, and 3.0); ν_{max} 3400 and 3180 (OH), 1590, 1565, and 1480 cm^{-1} (C=C, C=N aromatic). Periodate consumption, 0.99 mol/mol.

Anal. Calc. for $C_{10}H_{13}NO_{10}$: C, 56.86; H, 6.20; N, 6.63. Found: C, 56.62; H, 6.28; N, 6.42.

The triacetate **12** of **10** had m.p. 88–90°, $[\alpha]_D$ −85°, $[\alpha]_{578}$ −91°, $[\alpha]_{546}$ −104°, $[\alpha]_{436}$ −179°, $[\alpha]_{365}$ −288° (*c* 0.5, chloroform); λ_{max}^{EtOH} 254, 259, and 265 nm (ε_{mM} 2.2, 2.4, and 1.7); ν_{max} 1730 (C=O), 1585, 1570, and 1460 cm^{-1} (C=C, C=N aromatic).

Anal. Calc. for $C_{16}H_{19}NO_7$: C, 56.97; H, 5.68; N, 4.15. Found: C, 56.63; H, 5.83; N, 3.91.

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