Crystal structure of 3,4,5,6-tetra-*O*-acetyl-2-azido-2-deoxy-D-glucose (4-nitrophenyl)-hydrazon, C₂₀H₂₄N₆O₁₀

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The *p*-nitrophenyl hydrazon moiety of per-*O*-acetylated aldoses exhibited similar but weaker activating effect (see ref. 1) for the nucleophilic replacement of AcO-2 than related formazyl group (see refs. 2–4). Treatment of 2,3,4,5,6-penta-*O*-acetyl-D-glucose (4-nitrophenyl)-hydrazon with sodium azide afforded the title compound and the mannose epimer which were separated by column chromatography (see ref. 1). Evidences for the structure of the two products were obtained by ¹H and ¹³C NMR spectroscopy but leaving the assignment of the configuration of C-2 of each acyclic sugar chain uncertain. The gluco configuration of the title compound was proved by X-ray crystallography.

C₂₀H₂₄N₆O₁₀, monoclinic, *P*12₁1 (No. 4), *a* = 10.647(4) Å, *b* = 10.573(3) Å, *c* = 11.121(2) Å, β = 90.80(3)°, *V* = 1251.8 Å³, *Z* = 2, *R*(*F*) = 0.037, *R*_w(*F*²) = 0.097.

Table 1. Parameters used for the X-ray data collection

Crystal:	yellow, block, size 0.20 x 0.40 x 0.80 mm
Wavelength:	Mo K _α radiation (0.71070 Å)
μ:	1.10 cm ⁻¹
Diffractometer:	Enraf-Nonius CAD4
Scan mode:	θ/ω
<i>T</i> _{measurement} :	293 K
2θ _{max} :	50°
<i>N</i> (<i>hkl</i>) _{unique} :	2178
Criterion for <i>I</i> _o :	<i>I</i> _o > 2 σ(<i>I</i> _o)
<i>N</i> (<i>param</i>) _{refined} :	329
Programs:	SHELXS-86, SHELXL-93

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(12)	2a	−0.0683(3)	0.8827(3)	0.6572(3)	0.083
H(1)	2a	0.1138(3)	0.8834(4)	0.7635(3)	0.071
H(2)	2a	0.3152(3)	0.8070(3)	0.7769(3)	0.069
H(3)	2a	0.2010(3)	0.7533(3)	0.9494(3)	0.059
H(4)	2a	0.4133(3)	0.7101(3)	0.9719(3)	0.066
H(5)	2a	0.2702(3)	0.5052(3)	1.0658(3)	0.068
H(6A)	2a	0.4277(4)	0.4830(4)	1.2136(4)	0.085
H(6B)	2a	0.4841(4)	0.4566(4)	1.0861(4)	0.085
H(33A)	2a	−0.019(2)	0.4242(7)	0.937(3)	0.144
H(33B)	2a	−0.067(3)	0.495(2)	0.8210(6)	0.144
H(33C)	2a	−0.134(1)	0.516(2)	0.944(3)	0.144
H(43A)	2a	0.509(1)	0.439(3)	0.711(3)	0.169
H(43B)	2a	0.597(4)	0.400(2)	0.8186(6)	0.169
H(43C)	2a	0.645(3)	0.494(1)	0.721(3)	0.169
H(53A)	2a	0.0627(7)	0.743(3)	1.312(3)	0.143
H(53B)	2a	0.194(3)	0.808(1)	1.296(2)	0.143
H(53C)	2a	0.176(3)	0.707(2)	1.3972(6)	0.143
H(63A)	2a	0.716(3)	0.748(2)	1.231(2)	0.154
H(63B)	2a	0.718(3)	0.775(2)	1.092(2)	0.154
H(63C)	2a	0.8223(4)	0.690(1)	1.151(4)	0.154
H(122)	2a	−0.0481(3)	0.5797(4)	0.5401(3)	0.080
H(123)	2a	−0.2137(3)	0.4700(4)	0.4517(3)	0.082
H(125)	2a	−0.4488(3)	0.7534(4)	0.5260(3)	0.077
H(126)	2a	−0.2857(3)	0.8656(4)	0.6125(3)	0.077

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
O(31)	2a	0.1466(2)	0.5859(2)	0.8917(2)	0.050(1)	0.044(1)	0.056(1)	−0.008(1)	−0.0012(9)	−0.004(1)
O(32)	2a	0.0039(3)	0.7035(3)	0.9839(4)	0.065(2)	0.087(2)	0.136(3)	−0.010(2)	0.032(2)	−0.034(2)
O(41)	2a	0.4144(2)	0.5444(3)	0.8872(2)	0.057(1)	0.067(2)	0.063(1)	0.011(1)	0.008(1)	0.004(1)
O(42)	2a	0.5727(3)	0.6768(5)	0.8488(3)	0.084(2)	0.140(4)	0.106(3)	−0.025(2)	0.029(2)	0.020(2)

Table 3. (Continued)

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(51)	2a	0.2770(2)	0.6680(2)	1.1558(2)	0.063(1)	0.052(1)	0.053(1)	-0.009(1)	0.002(1)	0.002(1)
O(52)	2a	0.1470(3)	0.5248(3)	1.2312(3)	0.102(2)	0.070(2)	0.104(2)	-0.021(2)	0.025(2)	0.023(2)
O(61)	2a	0.5361(2)	0.6228(3)	1.1547(2)	0.050(1)	0.074(2)	0.077(2)	0.004(1)	-0.010(1)	-0.008(1)
O(62)	2a	0.6925(3)	0.4992(4)	1.0972(4)	0.089(2)	0.088(2)	0.147(3)	0.022(2)	0.032(2)	0.030(2)
O(128)	2a	-0.4253(3)	0.4249(3)	0.3868(3)	0.104(2)	0.076(2)	0.085(2)	-0.017(2)	-0.010(2)	-0.020(2)
O(129)	2a	-0.5533(3)	0.5752(4)	0.4297(3)	0.067(2)	0.142(3)	0.094(2)	-0.008(2)	0.000(2)	-0.044(2)
N(11)	2a	0.0563(2)	0.7475(3)	0.6634(3)	0.060(2)	0.053(2)	0.056(2)	0.009(1)	-0.007(1)	-0.001(1)
N(12)	2a	-0.0567(3)	0.8046(3)	0.6390(3)	0.068(2)	0.047(2)	0.075(2)	0.013(2)	-0.019(2)	-0.012(2)
N(21)	2a	0.2961(3)	0.6506(4)	0.6823(3)	0.071(2)	0.099(3)	0.051(2)	0.016(2)	0.005(1)	-0.012(2)
N(22)	2a	0.2286(4)	0.5590(4)	0.6592(3)	0.098(3)	0.090(3)	0.057(2)	0.037(3)	-0.003(2)	-0.022(2)
N(23)	2a	0.1769(5)	0.4685(5)	0.6297(5)	0.153(4)	0.097(4)	0.119(4)	0.037(3)	-0.023(3)	-0.055(3)
N(127)	2a	-0.4486(3)	0.5299(4)	0.4294(3)	0.078(2)	0.088(3)	0.047(2)	-0.015(2)	0.002(2)	-0.010(2)
C(1)	2a	0.1325(3)	0.8032(4)	0.7344(3)	0.061(2)	0.052(2)	0.050(2)	0.002(2)	-0.005(2)	0.001(2)
C(2)	2a	0.2515(3)	0.7403(3)	0.7708(3)	0.054(2)	0.058(2)	0.048(2)	-0.004(2)	0.000(1)	0.001(2)
C(3)	2a	0.2361(3)	0.6865(3)	0.8989(3)	0.044(2)	0.045(2)	0.048(2)	-0.004(1)	-0.001(1)	-0.002(1)
C(4)	2a	0.3553(3)	0.6392(3)	0.9590(3)	0.048(2)	0.051(2)	0.054(2)	-0.001(2)	-0.001(1)	0.001(2)
C(5)	2a	0.3310(3)	0.5737(3)	1.0785(3)	0.057(2)	0.051(2)	0.050(2)	0.000(2)	-0.004(1)	0.003(2)
C(6)	2a	0.4479(4)	0.5212(4)	1.1369(4)	0.069(2)	0.061(2)	0.067(2)	0.000(2)	-0.014(2)	0.005(2)
C(32)	2a	0.0312(3)	0.6080(4)	0.9338(4)	0.053(2)	0.064(2)	0.069(2)	-0.008(2)	0.004(2)	-0.003(2)
C(33)	2a	-0.0547(4)	0.5014(5)	0.9065(5)	0.073(3)	0.081(3)	0.134(4)	-0.029(3)	0.004(3)	-0.008(3)
C(42)	2a	0.5258(4)	0.5751(6)	0.8363(4)	0.065(2)	0.103(4)	0.066(2)	0.017(3)	0.010(2)	0.031(3)
C(43)	2a	0.5731(5)	0.4678(6)	0.7654(5)	0.111(4)	0.136(5)	0.092(3)	0.056(4)	0.043(3)	0.028(3)
C(52)	2a	0.1880(4)	0.6295(4)	1.2330(4)	0.065(2)	0.067(3)	0.064(2)	-0.003(2)	0.005(2)	0.019(2)
C(53)	2a	0.1519(5)	0.7305(5)	1.3167(4)	0.113(3)	0.091(3)	0.084(3)	0.007(3)	0.038(3)	0.010(3)
C(62)	2a	0.6570(4)	0.6008(5)	1.1314(4)	0.067(2)	0.081(3)	0.069(2)	0.018(2)	0.008(2)	0.016(2)
C(63)	2a	0.7352(4)	0.7132(6)	1.1533(5)	0.064(3)	0.128(5)	0.116(4)	-0.017(3)	0.004(3)	0.011(4)
C(121)	2a	-0.1509(3)	0.7358(3)	0.5851(3)	0.062(2)	0.049(2)	0.050(2)	0.003(2)	-0.009(2)	-0.001(2)
C(122)	2a	-0.1282(3)	0.6149(4)	0.5368(3)	0.068(2)	0.057(2)	0.059(2)	0.007(2)	-0.003(2)	-0.010(2)
C(123)	2a	-0.2271(3)	0.5496(4)	0.4846(3)	0.086(2)	0.055(2)	0.049(2)	0.002(2)	-0.001(2)	-0.010(2)
C(124)	2a	-0.3448(3)	0.6015(4)	0.4811(3)	0.064(2)	0.064(2)	0.042(2)	-0.007(2)	0.001(1)	-0.003(2)
C(125)	2a	-0.3682(3)	0.7195(4)	0.5288(3)	0.061(2)	0.064(2)	0.053(2)	0.008(2)	-0.006(2)	-0.001(2)
C(126)	2a	-0.2709(3)	0.7859(4)	0.5803(3)	0.065(2)	0.051(2)	0.060(2)	0.013(2)	-0.007(2)	-0.008(2)

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