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Crystal structure of 6-(2-bromoacetamido) tetrahydro-2*H*-pyran-2,3,4,5-Tetraol tetraacetate, $C_{16}H_{22}BrNO_{10}$

Table 1: Data collection and handling.

Crystal:	Block, colorless
Size:	0.180 × 0.160 × 0.140 mm
Wavelength:	Mo K_{α} radiation ($\lambda = 0.71073 \text{ \AA}$)
μ :	1.997 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II CCD, ϕ and ω -scans
$2\theta_{\max}$, completeness:	25.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	16232, 3917, 0.0391
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 3266
$N(\text{param})_{\text{refined}}$:	258
Programs:	Bruker programs [1], ShelX [2]

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Abstract

$[C_{16}H_{22}BrNO_{10}]$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 9.7453(13) \text{ \AA}$, $b = 9.9023(13) \text{ \AA}$, $c = 21.885(3) \text{ \AA}$, $V = 2111.9(5) \text{ \AA}^3$, $Z = 4$, $R_{\text{gt}}(F) = 0.0357$, $wR_{\text{ref}}(F^2) = 0.0981$, $T = 296(2) \text{ K}$.

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Source of materials

The synthesis process of aminosugars derivatives is carried out using 2,3,4,6-tetra-O-acetyl- β -D-glucopyranosyl bromide (5 g, 12.16 mmol) as starting material, and reacting in the first step with NaN_3 (0.95 g, 14.59 mmol) in DMSO (60 mL) stirring at room temperature, the resulting product to react in the second step with 10% Pd/C in H_2 using celite to remove Pd/C, filtered and concentrated under reduced pressure at 30 °C giving a light yellow power. Finally triethylamine (0.8 mL, 5.78 mmol) and the light yellow powder (1 g, 2.88 mmol) in 10 mL CH_2Cl_2 were added to a solution of bromoacetyl bromide (0.3 mL, 3.46 mmol) in CH_2Cl_2 at 0 °C and stirred at room temperature. The products were extracted with ethyl acetate, concentrated under reduced pressure and recrystallize from petroleum ether and ethyl acetate to give 0.82 g of the title compound. **m.p.:** 99.2–103.5 °C. **Elemental analysis** calculated for $C_{16}H_{22}BrNO_{10}$: C, 41.04%; H, 4.74%; N, 2.99%; found: C, 40.91%; H, 4.66%; N, 3.17%.

Experimental details

H atoms were included in calculated positions as riding atoms, with C–H = 0.96–0.97 Å with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2U_{\text{eq}}(\text{C})$ for all other H atoms.

Comment

Aminosugars are components of a variety of biologically active and important compounds, including mono- and

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.4051(5)	0.1516(4)	0.44483(19)	0.0401(10)
H1	0.3756	0.2462	0.4453	0.048
C2	0.2784(4)	0.0627(4)	0.45412(18)	0.0370(9)
H2	0.3049	−0.0327	0.4552	0.044
C3	0.1793(4)	0.0872(4)	0.4034(2)	0.0383(9)
H3	0.1447	0.1799	0.4058	0.046
C4	0.2493(4)	0.0649(4)	0.34152(18)	0.0367(9)
H4	0.2759	−0.0301	0.3376	0.044
C5	0.3758(5)	0.1534(4)	0.33682(19)	0.0384(9)
H5	0.3487	0.2486	0.3383	0.046
C6	0.5154(5)	0.1343(4)	0.4920(2)	0.0482(10)
H6A	0.5955	0.1868	0.4807	0.058
H6B	0.4830	0.1662	0.5313	0.058
C7	0.6291(5)	−0.0396(5)	0.5433(2)	0.0522(12)
C8	0.6426(7)	−0.1898(6)	0.5484(3)	0.0826(19)
H8A	0.7195	−0.2195	0.5245	0.124
H8B	0.5604	−0.2319	0.5334	0.124
H8C	0.6564	−0.2142	0.5904	0.124
C9	0.2330(5)	0.0205(5)	0.5605(2)	0.0460(11)
C10	0.1680(7)	0.0832(5)	0.6150(2)	0.0665(15)
H10A	0.0706	0.0882	0.6089	0.100
H10B	0.2042	0.1724	0.6207	0.100
H10C	0.1872	0.0293	0.6504	0.100
C11	−0.0626(5)	0.0423(6)	0.4009(3)	0.0636(15)
C12	−0.1644(6)	−0.0710(7)	0.3965(4)	0.105(3)
H12A	−0.2558	−0.0348	0.3970	0.157
H12B	−0.1526	−0.1309	0.4306	0.157
H12C	−0.1499	−0.1197	0.3591	0.157
C13	0.1068(6)	−0.0027(7)	0.2590(2)	0.0651(15)
C14	0.0101(8)	0.0509(9)	0.2113(3)	0.110(3)
H14A	0.0366	0.0172	0.1719	0.165
H14B	0.0137	0.1477	0.2111	0.165
H14C	−0.0816	0.0219	0.2204	0.165
C15	0.5053(5)	0.2267(3)	0.24866(18)	0.0385(8)
C16	0.5886(5)	0.1801(4)	0.19465(19)	0.0437(10)
H16A	0.5878	0.0823	0.1925	0.052
H16B	0.6830	0.2096	0.1991	0.052
N1	0.4497(4)	0.1280(3)	0.28217(16)	0.0395(9)
H1A	0.4590	0.0458	0.2701	0.047
O1	0.4654(3)	0.1242(3)	0.38645(12)	0.0394(6)
O2	0.5511(3)	−0.0065(3)	0.49599(13)	0.0420(7)
O3	0.2136(3)	0.1002(3)	0.51061(13)	0.0466(8)
O4	0.0662(3)	−0.0062(3)	0.40703(16)	0.0493(8)
O5	0.1569(3)	0.0993(3)	0.29369(14)	0.0505(8)
O6	0.2943(4)	−0.0830(4)	0.55874(16)	0.0640(10)
O7	−0.0881(4)	0.1582(5)	0.3985(3)	0.109(2)
O8	0.1369(5)	−0.1166(5)	0.2661(2)	0.0998(16)
O9	0.4900(4)	0.3469(3)	0.26018(14)	0.0552(8)
O10	0.6795(5)	0.0407(4)	0.5774(2)	0.0811(13)
Br1	0.51084(5)	0.25512(5)	0.12102(2)	0.0631(2)

polysaccharides, glycolipids, nucleotides, anthracycline anti-tumor agents, and antibiotics [3, 4]. These compounds have been reported to exhibit a variety of pharmacological activities: such as enhanced the immune function of mice [5], antitumor [6], extenuate arthritis symptoms [7], hypoglycemic

effect [8], and antibacterial [9]. The amino groups present in the aminoglycoside antibiotics and polysaccharides play an important role in their biological activities [9–11].

Bond lengths and angles are normal and very similar to those given in the literature for *D*-glucopyranose [12, 13]. The hexopyranosylring adopts a normal ⁴C₁ chair conformation. The four substituents are in equatorial positions to the ring. In the crystal CH···O and CH···Br hydrogen bonding leads to the formation of a three-dimensional network.

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