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2,3,4-Triacetoxy-1-[5-(1,2,3,4-tetra-acetoxybutyl)pyrazin-2-yl]butyl acetate

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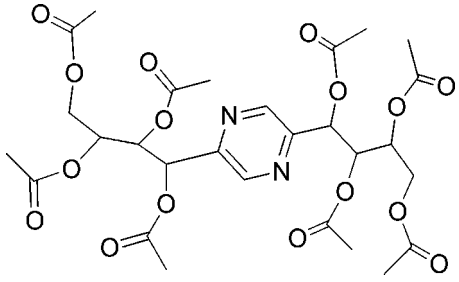
Key indicators: single-crystal X-ray study; T = 159 K; mean σ(C–C) = 0.005 Å; R factor = 0.045; wR factor = 0.103; data-to-parameter ratio = 7.8.

The title compound, $C_{28}H_{36}N_2O_{16}$, was obtained unintentionally in an attempt to synthesize 1,3,4,6-tetra-*O*-acetyl-2-azido-2-deoxy-D-mannopyranose. The crystal packing utilizes methyl–acetoxy C—H···O and methyl–pyrazine C—H···N hydrogen bonding.

Related literature

For general background, see: Vasella *et al.* (1991); Alper *et al.* (1996). For related literature, see: Bovin *et al.* (1981); Paulsen & Stenzel (1978); Paulsen *et al.* (1985); Pavliak & Kovac (1991). For related structures, see: Klein *et al.* (1999); Myers *et al.* (2000), found in a search of the Cambridge Structural Database (Version 5.28; Allen; 2002).



Experimental

Crystal data

$C_{28}H_{36}N_2O_{16}$
M_r = 656.59
Triclinic, $P\overline{1}$
a = 5.6931 (8) Å
b = 9.9132 (15) Å
c = 15.5048 (11) Å
α = 81.344 (2)°
β = 80.635 (2)°
γ = 73.986 (2)°
V = 824.71 (18) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 159 (2) K
0.72 × 0.24 × 0.11 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
7120 measured reflections
3304 independent reflections
2954 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.103
S = 1.11
3304 reflections
424 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···N1^{i}	0.95	2.61	3.338 (5)	134
C6—H6···N4^{ii}	0.95	2.60	3.331 (5)	134
C7—H7B···O2^{ii}	0.98	2.57	3.400 (5)	142
C19—H19A···O12^{iii}	0.98	2.58	3.443 (5)	147
C19—H19B···O10^{ii}	0.98	2.58	3.407 (5)	142
C19—H19C···O2^{iii}	0.98	2.53	3.472 (6)	161
C26—H26B···O14^{i}	0.98	2.60	3.471 (6)	148

Symmetry codes: (i) $x+1, y, z$; (ii) $x-1, y, z$; (iii) $x, y-1, z$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2446).

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supplementary materials

Acta Cryst. (2008). E**64**, o50 [**doi:10.1107/S1600536807062344**]

2,3,4-Triacetoxy-1-[5-(1,2,3,4-tetraacetoxybutyl)pyrazin-2-yl]butyl acetate

G. J. Gainsford and P. A. Benjes

Comment

We were attempting to establish suitable process routes towards 2-azido-2-deoxy-D-*manno*- and -*D*-glucopyranoses utilizing diazo-transfer chemistry as described in the literature (Vasella *et al.*, 1991; Alper *et al.*, 1996). These monosaccharides have been described in the literature as existing in the gum state (Paulsen & Stenzel, 1978; Paulsen *et al.*, 1985). In order to obtain crystalline materials, the acetate derivatives of these sugars were targeted. Both anomers of 1,3,4,6-tetra-*O*-acetyl-2-azido-2-deoxy-*D*-glucopyranose have been reported as crystalline (Paulsen & Stenzel, 1978; Paulsen *et al.*, 1985, Pavliak & Kovac, 1991; Bovin *et al.*, 1981) as has 1,3,4,6-tetra-*O*-acetyl-2-azido-2-deoxy-α-*D*-mannopyranose (Paulsen *et al.*, 1985; Bovin *et al.*, 1981). Although 1,3,4,6-tetra-*O*-acetyl-2-azido-2-deoxy-α- and –β-*D*-mannopyranoses were produced, about one third of the product mixture was the title compound which preferentially crystallized from ethyl acetate/hexanes.

There is only one other pyrazine derivative reported (Allen, 2002) in the CSD (JOQQAH: 2,3-bis(1-Phenyliminoethyl)pyrazine, Klein *et al.*, 1999), and one related piperazine (XERGAC, Myers *et al.*, 2000). Crystal packing is provided through (methyl)C—H···O (acetoxy) and C—H···N (pyrazine) hydrogen bonding (Table 1).

Experimental

Aqueous mannosamine hydrochloride was treated with stoichiometric potassium carbonate and subsequently with a solution of excess triflic azide (trifluromethanesulfonyl azide) in dichloromethane, in the presence of a catalytic amount (1 mol%) of copper(II) sulfate. Methanol was utilized as required to ensure homogeneity of the reaction mixture. Once all the starting material had been consumed, the mixture was concentrated to a syrup and then subjected to per-acetylation by dissolution in pyridine and treatment with excess acetic anhydride in the presence of catalytic 4-(*N*,*N*-dimethylamino)pyridine. The mixture was concentrated to a brown syrup, dissolved in ethyl acetate and passed through a plug of silica gel. Concentration again afforded a brown syrup which appeared to contain a mixture of compounds, two of which were identified (by NMR comparison with authentic samples) as being the desired 1,3,4,6-tetra-*O*-acetyl-2-azido-2-deoxy-α- and –β-*D*-mannopyranoses. Approximately one third of the product mixture was the title compound which preferentially crystallized during an attempted crystallization from ethyl acetate/hexanes. Suitable crystals were grown from ethyl acetate solution by addition of small amounts of hexanes until turbity was observed, heated to a clear solution; crystals obtained on cooling were washed with hexanes.

Refinement

In the absence of significant anomalous sattering, the values of the Flack [(1983). Acta Cryst. A39, 876–881] parameter were indeterminate [Flack & Bernardinelli (2000) J. Appl. Cryst. 33, 1143–1148]. Accordingly, the Friedel-equivalent reflections were merged prior to the final refinements. All H atoms were constrained to their expected geometries (C—H 0.99, 0.98 Å). and refined with U_{iso} 1.2,1.5 times that of the U_{eq} of their carrier atoms.

Figures

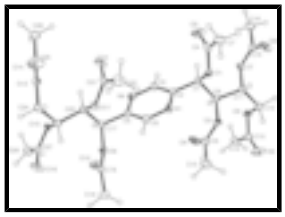


Fig. 1. The structure of (I) with 30% probabilility ellipsoids (*ORTEP-3*; Farrugia, 1997).

2,3,4-Triacetoxy-1-[5-(1,2,3,4-tetraacetoxybutyl)pyrazin-2-yl]butyl acetate

Crystal data

$C_{28}H_{36}N_2O_{16}$	$Z = 1$
$M_r = 656.59$	$F_{000} = 346$
Triclinic, $P\overline{1}$	$D_x = 1.322$ Mg m^{-3}
Hall symbol: P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 5.6931$ (8) Å	Cell parameters from 5104 reflections
$b = 9.9132$ (15) Å	$\theta = 4.4$–26.4°
$c = 15.5048$ (11) Å	$\mu = 0.11$ mm^{-1}
$\alpha = 81.344$ (2)°	$T = 159$ (2) K
$\beta = 80.635$ (2)°	Needle, colorless
$\gamma = 73.986$ (2)°	0.72 × 0.24 × 0.11 mm
$V = 824.71$ (18) Å^3	

Data collection

Siemens SMART CCD area-detector diffractometer	3304 independent reflections
Radiation source: fine-focus sealed tube	2954 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.029$
Detector resolution: 8.192 pixels mm^{-1}	$\theta_{max} = 26.4^\circ$
$T = 159(2)$ K	$\theta_{min} = 2.2^\circ$
φ and ω scans	$h = -3 \rightarrow 7$
Absorption correction: none	$k = -12 \rightarrow 12$
7120 measured reflections	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.045$	$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 0.2195P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.103$	$(\Delta/\sigma)_{max} = 0.009$
$S = 1.11$	$\Delta\rho_{max} = 0.18$ e Å^{-3}

3304 reflections
424 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

$\Delta\rho_{min} = -0.19$ e Å^{-3}
Extinction correction: none

Special details

Experimental. Crystal decay was monitored by repeating the initial 10 frames at the end of the data collection and analyzing duplicate reflections. The standard 0.8 mm diameter collimator was used.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.1773 (4)	0.7258 (2)	0.61777 (15)	0.0260 (5)
O2	0.3590 (5)	0.9030 (3)	0.5790 (3)	0.0582 (10)
O3	0.2283 (4)	0.4418 (2)	0.70327 (15)	0.0253 (5)
O4	0.5514 (5)	0.2533 (3)	0.6832 (2)	0.0477 (8)
O5	0.4938 (4)	0.4054 (3)	0.88213 (16)	0.0324 (6)
O6	0.3058 (6)	0.2322 (4)	0.9229 (3)	0.0688 (11)
O7	0.4371 (4)	0.6945 (2)	0.79743 (16)	0.0275 (5)
O8	0.1083 (5)	0.8765 (3)	0.8170 (3)	0.0589 (10)
O9	0.6049 (4)	0.2219 (2)	0.33102 (15)	0.0260 (5)
O10	0.9351 (5)	0.0465 (3)	0.3595 (2)	0.0520 (8)
O11	0.4486 (4)	0.5073 (2)	0.24872 (15)	0.0256 (5)
O12	0.5623 (5)	0.7043 (3)	0.2567 (2)	0.0472 (8)
O13	0.8382 (4)	0.5509 (3)	0.06940 (16)	0.0310 (6)
O14	0.5172 (6)	0.7186 (4)	0.0242 (3)	0.0676 (11)
O15	0.9951 (4)	0.2621 (2)	0.15420 (16)	0.0280 (5)
O16	0.8672 (5)	0.0803 (3)	0.1278 (3)	0.0581 (9)
N1	0.3344 (5)	0.5037 (3)	0.51401 (19)	0.0284 (7)
N4	0.8257 (5)	0.4504 (3)	0.43623 (19)	0.0264 (6)
C1	0.4152 (5)	0.6276 (4)	0.6262 (2)	0.0230 (7)
H1	0.5363	0.6814	0.6313	0.028*
C2	0.5011 (5)	0.5497 (3)	0.5451 (2)	0.0229 (7)
C3	0.7430 (6)	0.5242 (4)	0.5062 (2)	0.0256 (7)
H3	0.8550	0.5599	0.5296	0.031*
C4	0.1747 (7)	0.8635 (4)	0.5934 (2)	0.0313 (8)

C5	0.6607 (5)	0.4030 (3)	0.4065 (2)	0.0221 (7)
C6	0.4154 (6)	0.4294 (4)	0.4455 (2)	0.0292 (8)
H6	0.3034	0.3932	0.4225	0.035*
C7	−0.0781 (7)	0.9536 (4)	0.5866 (3)	0.0410 (9)
H7A	−0.1403	1.0011	0.6399	0.061*
H7B	−0.1858	0.8948	0.5801	0.061*
H7C	−0.0757	1.0244	0.5353	0.061*
C8	0.3913 (6)	0.5271 (4)	0.7099 (2)	0.0241 (7)
H8	0.5576	0.4642	0.7197	0.029*
C9	0.3341 (6)	0.3066 (4)	0.6861 (2)	0.0315 (8)
C10	0.1456 (7)	0.2369 (4)	0.6714 (3)	0.0432 (10)
H10A	0.2276	0.1491	0.6453	0.065*
H10B	0.0356	0.3002	0.6315	0.065*
H10C	0.0494	0.2155	0.7278	0.065*
C11	0.2789 (6)	0.6045 (4)	0.7911 (2)	0.0256 (7)
H11	0.1108	0.6652	0.7816	0.031*
C12	0.2596 (6)	0.5074 (4)	0.8747 (2)	0.0317 (8)
H12A	0.2132	0.5627	0.9258	0.038*
H12B	0.1305	0.4581	0.8743	0.038*
C13	0.4913 (7)	0.2697 (4)	0.9063 (2)	0.0343 (9)
C14	0.7445 (8)	0.1760 (4)	0.9068 (3)	0.0423 (10)
H14A	0.7598	0.1207	0.9645	0.064*
H14B	0.8637	0.2335	0.8948	0.064*
H14C	0.7771	0.1120	0.8613	0.064*
C15	0.3246 (6)	0.8303 (4)	0.8119 (2)	0.0294 (8)
C16	0.5035 (8)	0.9104 (5)	0.8197 (3)	0.0423 (10)
H16A	0.4744	0.9978	0.7794	0.063*
H16B	0.6715	0.8529	0.8048	0.063*
H16C	0.4828	0.9336	0.8802	0.063*
C17	0.7464 (5)	0.3247 (4)	0.3258 (2)	0.0241 (7)
H17	0.9255	0.2753	0.3242	0.029*
C18	0.7229 (6)	0.0834 (4)	0.3507 (2)	0.0280 (8)
C19	0.5532 (7)	−0.0086 (4)	0.3588 (3)	0.0428 (10)
H19A	0.6211	−0.0818	0.3189	0.064*
H19B	0.3923	0.0483	0.3433	0.064*
H19C	0.5341	−0.0531	0.4194	0.064*
C20	0.7033 (5)	0.4248 (3)	0.2418 (2)	0.0226 (7)
H20	0.8146	0.4894	0.2331	0.027*
C21	0.4017 (6)	0.6466 (4)	0.2599 (2)	0.0293 (8)
C22	0.1323 (7)	0.7117 (5)	0.2744 (3)	0.0479 (11)
H22A	0.1015	0.8148	0.2673	0.072*
H22B	0.0661	0.6801	0.3341	0.072*
H22C	0.0516	0.6832	0.2316	0.072*
C23	0.7427 (6)	0.3479 (4)	0.1605 (2)	0.0258 (7)
H23	0.6290	0.2847	0.1694	0.024 (9)*
C24	0.7002 (7)	0.4464 (4)	0.0770 (2)	0.0313 (8)
H24A	0.7533	0.3918	0.0256	0.038*
H24B	0.5225	0.4938	0.0779	0.038*
C25	0.7234 (7)	0.6849 (4)	0.0426 (3)	0.0349 (9)

C26	0.8816 (7)	0.7823 (4)	0.0403 (3)	0.0421 (10)
H26A	0.8792	0.8427	−0.0159	0.063*
H26B	1.0509	0.7273	0.0466	0.063*
H26C	0.8191	0.8412	0.0887	0.063*
C27	1.0321 (7)	0.1277 (4)	0.1357 (2)	0.0303 (8)
C28	1.2961 (7)	0.0510 (4)	0.1289 (3)	0.0407 (9)
H28A	1.3411	0.0139	0.1879	0.061*
H28B	1.3961	0.1157	0.1010	0.061*
H28C	1.3260	−0.0274	0.0934	0.061*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0195 (10)	0.0230 (13)	0.0343 (13)	−0.0038 (9)	−0.0021 (9)	−0.0040 (10)
O2	0.0329 (15)	0.0335 (18)	0.105 (3)	−0.0122 (13)	−0.0085 (16)	0.0092 (17)
O3	0.0231 (11)	0.0209 (12)	0.0316 (13)	−0.0063 (9)	0.0003 (9)	−0.0051 (10)
O4	0.0324 (15)	0.0315 (16)	0.082 (2)	0.0007 (11)	−0.0141 (14)	−0.0234 (15)
O5	0.0338 (13)	0.0276 (14)	0.0350 (14)	−0.0075 (10)	−0.0045 (10)	−0.0021 (11)
O6	0.052 (2)	0.0374 (19)	0.120 (3)	−0.0234 (16)	−0.018 (2)	0.011 (2)
O7	0.0260 (11)	0.0253 (13)	0.0326 (13)	−0.0041 (9)	−0.0035 (9)	−0.0125 (10)
O8	0.0356 (17)	0.0365 (19)	0.107 (3)	−0.0002 (13)	−0.0094 (16)	−0.0326 (19)
O9	0.0229 (11)	0.0236 (13)	0.0331 (13)	−0.0074 (9)	−0.0047 (9)	−0.0044 (10)
O10	0.0324 (15)	0.0292 (16)	0.092 (3)	−0.0027 (12)	−0.0165 (14)	0.0010 (15)
O11	0.0227 (11)	0.0229 (13)	0.0325 (13)	−0.0041 (9)	−0.0061 (9)	−0.0072 (10)
O12	0.0345 (14)	0.0271 (16)	0.083 (2)	−0.0088 (12)	0.0005 (14)	−0.0237 (15)
O13	0.0344 (13)	0.0266 (14)	0.0307 (14)	−0.0070 (10)	−0.0032 (10)	−0.0022 (11)
O14	0.0464 (19)	0.042 (2)	0.113 (3)	−0.0079 (15)	−0.0280 (19)	0.010 (2)
O15	0.0255 (12)	0.0275 (13)	0.0314 (13)	−0.0073 (10)	0.0021 (9)	−0.0099 (10)
O16	0.0386 (16)	0.0363 (18)	0.107 (3)	−0.0088 (13)	−0.0085 (16)	−0.0329 (18)
N1	0.0184 (12)	0.0431 (19)	0.0266 (16)	−0.0106 (12)	0.0003 (10)	−0.0113 (13)
N4	0.0177 (13)	0.0339 (17)	0.0290 (16)	−0.0072 (11)	−0.0011 (10)	−0.0091 (13)
C1	0.0154 (14)	0.0269 (19)	0.0286 (18)	−0.0065 (13)	−0.0047 (12)	−0.0048 (14)
C2	0.0208 (14)	0.0259 (18)	0.0225 (17)	−0.0064 (13)	−0.0039 (12)	−0.0025 (13)
C3	0.0201 (15)	0.032 (2)	0.0293 (19)	−0.0111 (13)	−0.0030 (12)	−0.0107 (15)
C4	0.0320 (19)	0.029 (2)	0.033 (2)	−0.0072 (16)	−0.0045 (15)	−0.0047 (16)
C5	0.0214 (14)	0.0225 (17)	0.0213 (17)	−0.0043 (12)	−0.0038 (12)	−0.0002 (13)
C6	0.0208 (15)	0.041 (2)	0.0310 (19)	−0.0124 (14)	−0.0043 (13)	−0.0110 (16)
C7	0.038 (2)	0.028 (2)	0.054 (3)	−0.0022 (16)	−0.0120 (18)	−0.0019 (18)
C8	0.0171 (15)	0.0230 (18)	0.0318 (19)	−0.0045 (12)	0.0002 (13)	−0.0069 (14)
C9	0.0294 (19)	0.029 (2)	0.037 (2)	−0.0074 (15)	−0.0025 (14)	−0.0082 (16)
C10	0.039 (2)	0.036 (2)	0.061 (3)	−0.0159 (18)	−0.0094 (19)	−0.009 (2)
C11	0.0259 (17)	0.0274 (19)	0.0243 (18)	−0.0076 (14)	−0.0018 (13)	−0.0058 (15)
C12	0.0341 (18)	0.0256 (19)	0.0307 (19)	−0.0012 (14)	0.0010 (14)	−0.0066 (15)
C13	0.048 (2)	0.030 (2)	0.028 (2)	−0.0131 (18)	−0.0111 (16)	−0.0019 (16)
C14	0.049 (2)	0.028 (2)	0.049 (3)	−0.0037 (18)	−0.0100 (18)	−0.0066 (18)
C15	0.037 (2)	0.0250 (19)	0.0250 (18)	−0.0030 (15)	−0.0057 (14)	−0.0056 (15)
C16	0.053 (3)	0.035 (2)	0.045 (2)	−0.0152 (19)	−0.0136 (19)	−0.0074 (19)
C17	0.0166 (14)	0.0259 (18)	0.0307 (19)	−0.0053 (12)	−0.0017 (12)	−0.0079 (14)

C18	0.0314 (19)	0.0227 (19)	0.0274 (19)	−0.0044 (15)	0.0017 (14)	−0.0053 (15)
C19	0.045 (2)	0.033 (2)	0.053 (3)	−0.0170 (19)	−0.0065 (19)	−0.0024 (19)
C20	0.0193 (15)	0.0227 (17)	0.0263 (17)	−0.0037 (12)	−0.0025 (12)	−0.0077 (14)
C21	0.0292 (18)	0.024 (2)	0.035 (2)	−0.0031 (15)	−0.0041 (14)	−0.0101 (16)
C22	0.031 (2)	0.039 (3)	0.068 (3)	−0.0009 (17)	−0.0030 (19)	−0.007 (2)
C23	0.0244 (16)	0.0253 (18)	0.0297 (19)	−0.0089 (14)	−0.0020 (13)	−0.0062 (14)
C24	0.0402 (19)	0.031 (2)	0.0278 (19)	−0.0125 (15)	−0.0089 (15)	−0.0066 (15)
C25	0.035 (2)	0.030 (2)	0.034 (2)	−0.0011 (16)	0.0007 (15)	−0.0039 (17)
C26	0.047 (2)	0.029 (2)	0.048 (2)	−0.0081 (18)	−0.0035 (18)	−0.0008 (18)
C27	0.0361 (19)	0.030 (2)	0.0258 (19)	−0.0101 (16)	−0.0002 (14)	−0.0068 (15)
C28	0.036 (2)	0.033 (2)	0.050 (3)	−0.0026 (16)	0.0024 (17)	−0.0120 (19)

Geometric parameters (Å, °)

O1—C4	1.358 (4)	C8—H8	1.0000
O1—C1	1.446 (3)	C9—C10	1.489 (5)
O2—C4	1.195 (5)	C10—H10A	0.9800
O3—C9	1.357 (4)	C10—H10B	0.9800
O3—C8	1.441 (4)	C10—H10C	0.9800
O4—C9	1.200 (4)	C11—C12	1.503 (5)
O5—C13	1.345 (5)	C11—H11	1.0000
O5—C12	1.443 (4)	C12—H12A	0.9900
O6—C13	1.191 (5)	C12—H12B	0.9900
O7—C15	1.356 (4)	C13—C14	1.484 (6)
O7—C11	1.454 (4)	C14—H14A	0.9800
O8—C15	1.183 (4)	C14—H14B	0.9800
O9—C18	1.364 (4)	C14—H14C	0.9800
O9—C17	1.449 (4)	C15—C16	1.483 (5)
O10—C18	1.186 (4)	C16—H16A	0.9800
O11—C21	1.365 (4)	C16—H16B	0.9800
O11—C20	1.451 (4)	C16—H16C	0.9800
O12—C21	1.196 (4)	C17—C20	1.524 (5)
O13—C25	1.344 (5)	C17—H17	1.0000
O13—C24	1.445 (4)	C18—C19	1.481 (5)
O14—C25	1.197 (5)	C19—H19A	0.9800
O15—C27	1.358 (5)	C19—H19B	0.9800
O15—C23	1.450 (4)	C19—H19C	0.9800
O16—C27	1.189 (4)	C20—C23	1.524 (4)
N1—C6	1.331 (4)	C20—H20	1.0000
N1—C2	1.339 (4)	C21—C22	1.485 (5)
N4—C5	1.329 (4)	C22—H22A	0.9800
N4—C3	1.348 (4)	C22—H22B	0.9800
C1—C2	1.517 (5)	C22—H22C	0.9800
C1—C8	1.524 (5)	C23—C24	1.510 (5)
C1—H1	1.0000	C23—H23	1.0000
C2—C3	1.381 (4)	C24—H24A	0.9900
C3—H3	0.9500	C24—H24B	0.9900
C4—C7	1.481 (5)	C25—C26	1.485 (6)
C5—C6	1.398 (4)	C26—H26A	0.9800

C5—C17	1.514 (5)	C26—H26B	0.9800
C6—H6	0.9500	C26—H26C	0.9800
C7—H7A	0.9800	C27—C28	1.480 (5)
C7—H7B	0.9800	C28—H28A	0.9800
C7—H7C	0.9800	C28—H28B	0.9800
C8—C11	1.529 (4)	C28—H28C	0.9800
C4—O1—C1	116.8 (3)	H14A—C14—H14C	109.5
C9—O3—C8	117.1 (3)	H14B—C14—H14C	109.5
C13—O5—C12	117.4 (3)	O8—C15—O7	122.5 (3)
C15—O7—C11	117.0 (3)	O8—C15—C16	125.3 (4)
C18—O9—C17	117.2 (2)	O7—C15—C16	112.1 (3)
C21—O11—C20	117.7 (2)	C15—C16—H16A	109.5
C25—O13—C24	117.1 (3)	C15—C16—H16B	109.5
C27—O15—C23	116.3 (3)	H16A—C16—H16B	109.5
C6—N1—C2	116.6 (3)	C15—C16—H16C	109.5
C5—N4—C3	116.1 (3)	H16A—C16—H16C	109.5
O1—C1—C2	109.5 (2)	H16B—C16—H16C	109.5
O1—C1—C8	108.3 (2)	O9—C17—C5	108.2 (3)
C2—C1—C8	112.0 (3)	O9—C17—C20	108.2 (3)
O1—C1—H1	109.0	C5—C17—C20	111.3 (3)
C2—C1—H1	109.0	O9—C17—H17	109.7
C8—C1—H1	109.0	C5—C17—H17	109.7
N1—C2—C3	121.4 (3)	C20—C17—H17	109.7
N1—C2—C1	117.3 (3)	O10—C18—O9	122.1 (3)
C3—C2—C1	121.3 (3)	O10—C18—C19	126.5 (4)
N4—C3—C2	122.3 (3)	O9—C18—C19	111.4 (3)
N4—C3—H3	118.9	C18—C19—H19A	109.5
C2—C3—H3	118.9	C18—C19—H19B	109.5
O2—C4—O1	122.2 (3)	H19A—C19—H19B	109.5
O2—C4—C7	125.9 (4)	C18—C19—H19C	109.5
O1—C4—C7	111.9 (3)	H19A—C19—H19C	109.5
N4—C5—C6	121.6 (3)	H19B—C19—H19C	109.5
N4—C5—C17	117.1 (3)	O11—C20—C17	109.4 (2)
C6—C5—C17	121.2 (3)	O11—C20—C23	105.6 (2)
N1—C6—C5	121.9 (3)	C17—C20—C23	113.0 (3)
N1—C6—H6	119.0	O11—C20—H20	109.6
C5—C6—H6	119.0	C17—C20—H20	109.6
C4—C7—H7A	109.5	C23—C20—H20	109.6
C4—C7—H7B	109.5	O12—C21—O11	122.5 (3)
H7A—C7—H7B	109.5	O12—C21—C22	126.8 (4)
C4—C7—H7C	109.5	O11—C21—C22	110.8 (3)
H7A—C7—H7C	109.5	C21—C22—H22A	109.5
H7B—C7—H7C	109.5	C21—C22—H22B	109.5
O3—C8—C1	110.7 (3)	H22A—C22—H22B	109.5
O3—C8—C11	105.3 (2)	C21—C22—H22C	109.5
C1—C8—C11	112.7 (3)	H22A—C22—H22C	109.5
O3—C8—H8	109.3	H22B—C22—H22C	109.5
C1—C8—H8	109.3	O15—C23—C24	110.2 (3)
C11—C8—H8	109.3	O15—C23—C20	106.1 (2)

O4—C9—O3	123.2 (3)	C24—C23—C20	113.3 (3)
O4—C9—C10	125.9 (4)	O15—C23—H23	109.0
O3—C9—C10	110.9 (3)	C24—C23—H23	109.0
C9—C10—H10A	109.5	C20—C23—H23	109.0
C9—C10—H10B	109.5	O13—C24—C23	109.7 (3)
H10A—C10—H10B	109.5	O13—C24—H24A	109.7
C9—C10—H10C	109.5	C23—C24—H24A	109.7
H10A—C10—H10C	109.5	O13—C24—H24B	109.7
H10B—C10—H10C	109.5	C23—C24—H24B	109.7
O7—C11—C12	110.0 (3)	H24A—C24—H24B	108.2
O7—C11—C8	106.2 (2)	O14—C25—O13	122.8 (4)
C12—C11—C8	113.6 (3)	O14—C25—C26	125.5 (4)
O7—C11—H11	109.0	O13—C25—C26	111.7 (3)
C12—C11—H11	109.0	C25—C26—H26A	109.5
C8—C11—H11	109.0	C25—C26—H26B	109.5
O5—C12—C11	109.3 (3)	H26A—C26—H26B	109.5
O5—C12—H12A	109.8	C25—C26—H26C	109.5
C11—C12—H12A	109.8	H26A—C26—H26C	109.5
O5—C12—H12B	109.8	H26B—C26—H26C	109.5
C11—C12—H12B	109.8	O16—C27—O15	122.4 (3)
H12A—C12—H12B	108.3	O16—C27—C28	125.9 (4)
O6—C13—O5	122.8 (4)	O15—C27—C28	111.7 (3)
O6—C13—C14	125.6 (4)	C27—C28—H28A	109.5
O5—C13—C14	111.6 (3)	C27—C28—H28B	109.5
C13—C14—H14A	109.5	H28A—C28—H28B	109.5
C13—C14—H14B	109.5	C27—C28—H28C	109.5
H14A—C14—H14B	109.5	H28A—C28—H28C	109.5
C13—C14—H14C	109.5	H28B—C28—H28C	109.5
C4—O1—C1—C2	−101.0 (3)	C8—C11—C12—O5	49.8 (4)
C4—O1—C1—C8	136.6 (3)	C12—O5—C13—O6	−1.4 (6)
C6—N1—C2—C3	1.7 (5)	C12—O5—C13—C14	177.0 (3)
C6—N1—C2—C1	−176.7 (3)	C11—O7—C15—O8	−1.9 (5)
O1—C1—C2—N1	−43.2 (4)	C11—O7—C15—C16	178.3 (3)
C8—C1—C2—N1	77.0 (4)	C18—O9—C17—C5	−106.6 (3)
O1—C1—C2—C3	138.3 (3)	C18—O9—C17—C20	132.6 (3)
C8—C1—C2—C3	−101.5 (3)	N4—C5—C17—O9	149.7 (3)
C5—N4—C3—C2	−0.1 (5)	C6—C5—C17—O9	−33.0 (4)
N1—C2—C3—N4	−1.0 (5)	N4—C5—C17—C20	−91.5 (3)
C1—C2—C3—N4	177.3 (3)	C6—C5—C17—C20	85.8 (4)
C1—O1—C4—O2	0.4 (5)	C17—O9—C18—O10	−3.2 (5)
C1—O1—C4—C7	179.7 (3)	C17—O9—C18—C19	176.8 (3)
C3—N4—C5—C6	0.4 (5)	C21—O11—C20—C17	107.7 (3)
C3—N4—C5—C17	177.7 (3)	C21—O11—C20—C23	−130.4 (3)
C2—N1—C6—C5	−1.4 (5)	O9—C17—C20—O11	66.6 (3)
N4—C5—C6—N1	0.3 (5)	C5—C17—C20—O11	−52.2 (3)
C17—C5—C6—N1	−176.8 (3)	O9—C17—C20—C23	−50.7 (3)
C9—O3—C8—C1	102.4 (3)	C5—C17—C20—C23	−169.5 (2)
C9—O3—C8—C11	−135.4 (3)	C20—O11—C21—O12	6.4 (5)
O1—C1—C8—O3	64.7 (3)	C20—O11—C21—C22	−174.5 (3)

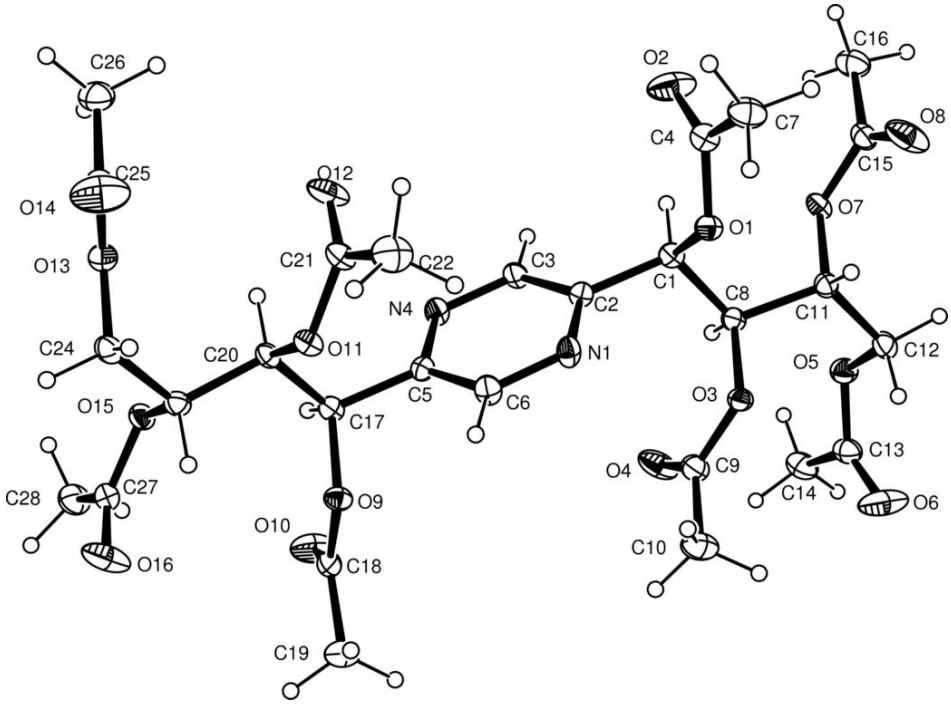
C2—C1—C8—O3	−56.2 (3)	C27—O15—C23—C24	−101.2 (3)
O1—C1—C8—C11	−53.0 (3)	C27—O15—C23—C20	135.7 (3)
C2—C1—C8—C11	−173.9 (2)	O11—C20—C23—O15	−178.5 (3)
C8—O3—C9—O4	6.0 (5)	C17—C20—C23—O15	−59.0 (3)
C8—O3—C9—C10	−173.8 (3)	O11—C20—C23—C24	60.5 (3)
C15—O7—C11—C12	−103.5 (3)	C17—C20—C23—C24	180.0 (3)
C15—O7—C11—C8	133.2 (3)	C25—O13—C24—C23	−137.3 (3)
O3—C8—C11—O7	−178.4 (3)	O15—C23—C24—O13	−68.2 (3)
C1—C8—C11—O7	−57.5 (3)	C20—C23—C24—O13	50.5 (4)
O3—C8—C11—C12	60.6 (3)	C24—O13—C25—O14	−1.0 (6)
C1—C8—C11—C12	−178.5 (3)	C24—O13—C25—C26	178.2 (3)
C13—O5—C12—C11	−134.8 (3)	C23—O15—C27—O16	−2.4 (5)
O7—C11—C12—O5	−69.1 (3)	C23—O15—C27—C28	178.8 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···N1^{i}	0.95	2.61	3.338 (5)	134
C6—H6···N4^{ii}	0.95	2.60	3.331 (5)	134
C7—H7B···O2^{ii}	0.98	2.57	3.400 (5)	142
C19—H19A···O12^{iii}	0.98	2.58	3.443 (5)	147
C19—H19B···O10^{ii}	0.98	2.58	3.407 (5)	142
C19—H19C···O2^{iii}	0.98	2.53	3.472 (6)	161
C26—H26B···O14^{i}	0.98	2.60	3.471 (6)	148

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) *x*−1, *y*, *z*; (iii) *x*, *y*−1, *z*.

Fig. 1


C26
C25
O14
O13
C24
O15
C27
C28
O16
C20
O11
C21
O12
C22
N4
C3
C2
N1
C5
C6
C17
O9
O10
C18
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C1
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C7
C16
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C11
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O5
O3
C13
C14
O6
O4
C9
C10