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ISSN 2414-3146

1-(Methyl-α-D-glucopyranosid-6-yl)-3-vinylimidazolium iodide dimethylformamide monosolvate

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Received 28 February 2022
Accepted 8 March 2022

Edited by L. Van Meervelt, Katholieke Universiteit Leuven, Belgium

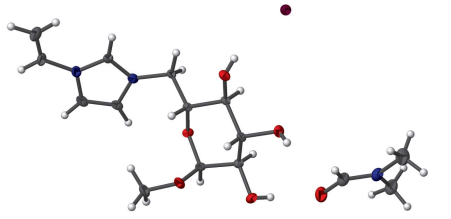
Keywords: crystal structure; carbohydrate; imidazolium.

CCDC reference: 2157239

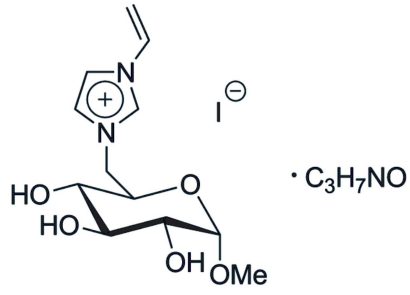
Structural data: full structural data are available from iucrdata.iucr.org

The title solvated molecular salt, [MeGluVIm]I (MeGluVIm = 1-(methyl-α-D-glucopyranosid-6-yl)-3-vinylimidazolium), or $C_{12}H_{19}N_2O_5^+\cdot I^-\cdot C_3H_7NO$, was synthesized from methyl-α-D-6-iodoglucopyranoside and vinylimidazole in DMF. It crystallizes through precipitation from ethyl acetate solution directly after the reaction procedure. The crystal structure consists of an iodide anion and a [MeGluVIm] cation. Furthermore, the crystal structure contains one molecule of DMF, which accepts two O—H···H hydrogen bonds from the OH groups of the glucopyranoside.

3D view



Chemical scheme



Structure description

[MeGluVIm]I is part of a sub-category of ionic liquids, called carbohydrate-based ionic liquids (CHILs; Jopp, 2020). These molecules are defined as ionic organic compounds in which either the cation or the anion consists of an intact carbohydrate moiety. Our group has recently discovered a straightforward synthetic strategy for CHILs, in which methyl-α-D-glucopyranoside is transformed into methyl-α-D-6-iodoglucopyranoside in the first step (Skaanderup *et al.*, 2002) and then in the second step quarternized with an *N*-substituted imidazole of choice to achieve a carbohydrate-based ionic liquid (Schnegas & Jopp, 2021). The title compound [MeGluVIm]I contains a vinylimidazolium ring bound to atom C6 of the glucopyranoside. Fig. 1 shows the asymmetric unit, including one molecule of dimethylformamide, which was used as the reaction solvent. The title compound crystallizes in a monoclinic unit cell. The crystal structure contains three classical hydrogen bonds and additional C—H···O/I interactions (Table 1). One hydrogen bond is formed between O3—H3*A* of the glucopyranoside and O7 of DMF

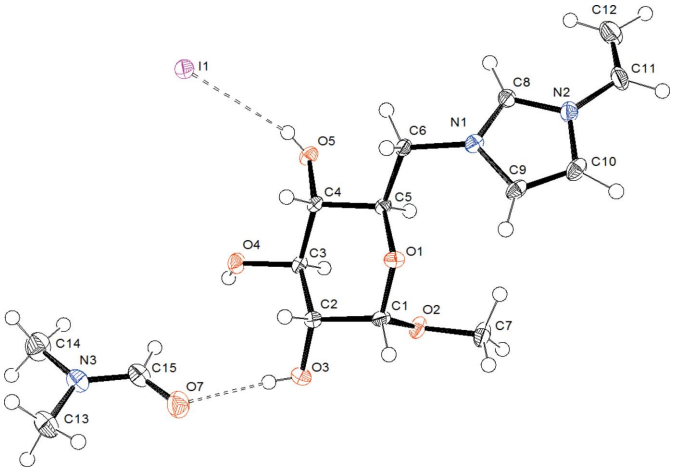


Figure 1
Molecular structure of the title compound. Displacement ellipsoids correspond to 50% probability.

with an H···H length of 2.09 (4) Å. Two additional hydrogen bonds exists between the [MeGluVIm] cation and the iodide anion, which are O4—H4*A*···I1 with 2.71 (5) Å and O5—H5*A*···I1 with 2.75 (5) Å. Fig. 2 gives an alternative view of the cation, indicating the distinctive chair conformation of the glucopyranoside as well as the overall stereochemistry of the compound. The configurations of the stereogenic centres in the chosen cation are *S* (C1), *R* (C2), *S* (C3), *S* (C4) and *R* (C5).

Synthesis and crystallization

Methyl-6-iodo-α-D-glucopyranoside (1.824 g; 6 mmol) and 1-vinylimidazole (0.821 g; 10 mmol) were dissolved in DMF (10 ml) and stirred at 95°C for 24 h. After cooling down, ethyl acetate (80 ml) was added and the flask was stored in a fridge overnight. The solvent was decanted and the precipitated solid was washed with ethyl acetate (3 × 40 ml) and dried under high vacuum to achieve the product as a beige solid (1.752 g; yield 73%). Single crystals of the compound were formed during the precipitation (m.p.: 448–453 K; T_d: 509 K).

^{1}H NMR (300 MHz, D_2O): δ = 3.21–3.30 (*m*, 3H, OCH_3); 3.58 (*dd*, 1H, ^3J = 9.77, ^3J = 3.77, H-2); 3.66–3.75 (*m*, 1H); 3.95 (*dd*, 1H, ^3J = 6.3, ^3J = 3.72); 4.50 (*dd*, 1H, ^3J = 14.55, ^3J = 7.38, H-6a); 4.70 (*dd*, 1H, ^3J = 14.55, ^3J = 2.55, H-6 b); 4.85 (*d*, 1H, ^3J = 3.77, H-1); 5.49 (*dd*, 1H, ^3J = 8.68, ^{3}J = 2.84, vinyl-CH);

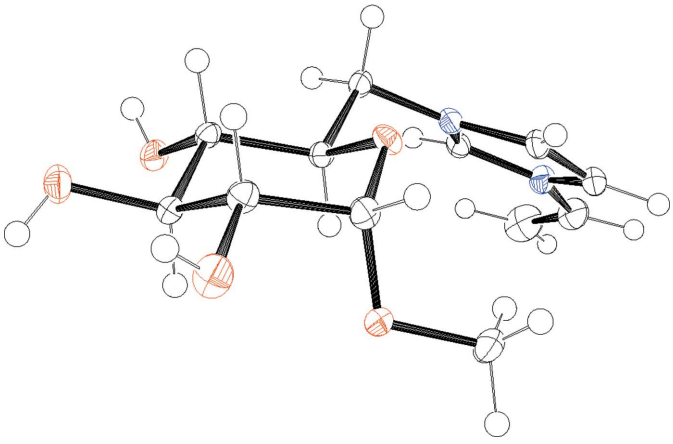


Figure 2
Molecular structure of the title compound. Displacement ellipsoids correspond to 50% probability. The DMF was removed for a clear view of the chair conformation.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O7	0.72 (4)	2.09 (4)	2.797 (4)	167 (5)
O4—H4*A*···I1^{i}	0.78 (5)	2.71 (5)	3.482 (3)	171 (4)
O5—H5*A*···I1	0.74 (5)	2.75 (5)	3.474 (3)	165 (4)
C6—H6*A*···O5^{ii}	0.99	2.46	3.332 (4)	147
C8—H8···O4^{ii}	0.95	2.44	3.252 (4)	143
C8—H8···O5^{ii}	0.95	2.53	3.285 (4)	136
C9—H9···O3^{iii}	0.95	2.51	3.404 (5)	156
C10—H10···O7^{iii}	0.95	2.40	3.159 (5)	137
C11—H11···I1^{iv}	0.95	3.02	3.925 (3)	161
C15—H15···O4	0.95	2.58	3.297 (5)	132

Symmetry codes: (i) $x, y-1, z$; (ii) $-x, y+\frac{1}{2}, -z+1$; (iii) $-x+1, y+\frac{1}{2}, -z+1$; (iv) $x, y, z-1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_{19}N_2O_5^+\cdot I^-\cdot C_3H_7NO$
M_r	471.29
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	123
a, *b*, *c* (Å)	10.816 (2), 7.0106 (15), 13.169 (3)
β (°)	106.833 (4)
V (Å^3)	955.7 (3)
Z	2
Radiation type	Mo *Kα*
μ (mm^{-1})	1.71
Crystal size (mm)	0.29 × 0.08 × 0.03
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2003)
T_{min}, T_{max}	0.629, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	17430, 6072, 5626
R_{int}	0.038
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.725
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.028, 0.060, 1.03
No. of reflections	6072
No. of parameters	242
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.42, −0.44
Absolute structure	Refined as an inversion twin, 2815 Friedel pairs.
Absolute structure parameter	0.006 (19)

Computer programs: *APEX2* and *SAINT* (Bruker, 2003), *SHELXTL* (Sheldrick, 2008), *SHELXL2014/7* (Sheldrick, 2015) and *ORTEP-3 for Windows* (Farrugia, 2012).

5.86 (*dd*, 1H, ^3J = 15.58, ^3J = 2.85, vinyl-$CH_{2\ -\ a}$); 7.2 (*dd*, 1H, ^3J = 15.58, ^3J = 8.70, vinyl-$CH_{2\ -\ b}$); 7.70 (*d*, 1H, ^{3}J = 2.0, H_{Ar}); 7.86 (*d*, 1H, ^3J = 2.0, H_{Ar}); 9.16 (*s*, 1H).

^{13}C NMR (300 MHz, D_2O): δm= 36.9 (NCH); 50.2 (C-6); 55.1 (OCH_3); 69.2, 40.5, 71.0, 72.8 (C-2, C-3, C-4, C-5); 99.3 (C-1); 109.8 (CH_2); 119.4, 123.8, 128.1 (CH_{Ar}).

HRMS (ESI, *m*/*z*): calculated for $C_{12}H_{19}N_2O_5^+$, 271.1299; measured 271.1306. Calculated for I^-, 126.9040; measured 126.9045.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The crystal studied was refined as a two-component inversion twin.

Funding information

We acknowledge financial support by the Deutsche Forschungsgemeinschaft and the University of Rostock within the funding programme Open Access Publishing.

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full crystallographic data

IUCrData (2022). **7**, x220265 [https://doi.org/10.1107/S2414314622002656]

1-(Methyl-*α*-D-glucopyranosid-6-yl)-3-vinylimidazolium iodide dimethylformamide monosolvate

Sina Lambrecht, Alexander Villinger and Stefan Jopp

3-Ethenyl-1-(methyl-*α*-*D*-glucopyranosid-6-yl)imidazolium iodide dimethylformamide monosolvate

Crystal data

$C_{12}H_{19}N_2O_5^+\cdot I^-\cdot C_3H_7NO$
M_r = 471.29
Monoclinic, $P2_1$
a = 10.816 (2) Å
b = 7.0106 (15) Å
c = 13.169 (3) Å
β = 106.833 (4)°
V = 955.7 (3) Å^3
Z = 2
$F(000)$ = 476
D_x = 1.638 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7185 reflections
θ = 3.2–31.1°
μ = 1.71 mm^{-1}
T = 123 K
Needle, colourless
0.29 × 0.08 × 0.03 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: sealed tube
Detector resolution: 10.4167 pixels mm^{-1}
phi and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2003)
T_{min} = 0.629, T_{max} = 0.746
17430 measured reflections
6072 independent reflections
5626 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 31.0°, θ_{min} = 3.8°
h = −15→15
k = −10→10
l = −19→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.060
S = 1.03
6072 reflections
242 parameters
1 restraint
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0231P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}
Absolute structure: Refined as an inversion twin, 2815 Friedel pairs.
Absolute structure parameter: 0.006 (19)

Special details

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

Refinement. All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.98 (methyl groups), 0.99Å (methylene groups), 1.00Å (methine groups) or 0.95 Å (aryl CH) and with U_{iso}(H) = 1.5 times U_{eq}(C) (methyl groups) or with U_{iso}(H) = 1.2 times U_{eq}(C) (methylene groups, aryl CH, methine groups). Torsion angles of all methyl groups were allowed to refine.
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 $F^2 > 2\sigma(F^2)$ 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.
Refined as a two-component inversion twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.1863 (3)	1.0859 (4)	0.3364 (2)	0.0151 (5)
N2	0.1065 (3)	1.1296 (4)	0.1676 (2)	0.0179 (6)
O1	0.3511 (2)	0.8245 (4)	0.4934 (2)	0.0146 (5)
O2	0.3182 (3)	0.5032 (4)	0.4445 (2)	0.0184 (5)
O3	0.4060 (3)	0.3939 (4)	0.6541 (3)	0.0222 (6)
O4	0.2120 (3)	0.5948 (4)	0.7282 (2)	0.0197 (5)
O5	0.0572 (2)	0.8579 (4)	0.5786 (2)	0.0169 (5)
C1	0.3882 (3)	0.6322 (5)	0.5202 (3)	0.0150 (7)
H1	0.4821	0.6180	0.5263	0.018*
C2	0.3673 (3)	0.5847 (5)	0.6269 (3)	0.0156 (6)
H2	0.4240	0.6703	0.6816	0.019*
C3	0.2266 (3)	0.6217 (5)	0.6246 (3)	0.0137 (6)
H3	0.1679	0.5327	0.5733	0.016*
C4	0.1910 (3)	0.8273 (5)	0.5914 (3)	0.0129 (6)
H4	0.2438	0.9163	0.6464	0.015*
C5	0.2174 (3)	0.8639 (4)	0.4853 (3)	0.0123 (6)
H5	0.1607	0.7793	0.4299	0.015*
C6	0.1951 (3)	1.0688 (4)	0.4498 (3)	0.0139 (6)
H6A	0.1141	1.1156	0.4618	0.017*
H6B	0.2671	1.1487	0.4921	0.017*
C7	0.3439 (4)	0.5229 (5)	0.3446 (3)	0.0238 (8)
H7A	0.3086	0.4128	0.2997	0.036*
H7B	0.4374	0.5295	0.3558	0.036*
H7C	0.3033	0.6400	0.3097	0.036*
C8	0.0831 (3)	1.1454 (4)	0.2617 (3)	0.0153 (7)
H8	0.0055	1.1915	0.2729	0.018*
C9	0.2788 (3)	1.0274 (5)	0.2893 (3)	0.0174 (7)
H9	0.3618	0.9775	0.3244	0.021*
C10	0.2286 (4)	1.0547 (5)	0.1843 (3)	0.0200 (7)
H10	0.2698	1.0273	0.1313	0.024*
C11	0.0197 (3)	1.1684 (9)	0.0656 (3)	0.0238 (7)
H11	0.0524	1.1636	0.0060	0.029*
C12	−0.1014 (4)	1.2099 (7)	0.0494 (3)	0.0322 (11)
H12A	−0.1368	1.2158	0.1075	0.039*
H12B	−0.1547	1.2344	−0.0205	0.039*
H3A	0.426 (4)	0.391 (7)	0.711 (3)	0.014 (12)*

H4A	0.186 (4)	0.495 (7)	0.740 (4)	0.025 (12)*
H5A	0.051 (4)	0.940 (7)	0.612 (4)	0.018 (12)*
I1	0.06251 (2)	1.18116 (3)	0.77975 (2)	0.02003 (6)
N3	0.4772 (3)	0.4326 (5)	1.0319 (3)	0.0235 (7)
O7	0.5214 (3)	0.4185 (5)	0.8731 (2)	0.0306 (7)
C13	0.6115 (5)	0.4439 (8)	1.0952 (4)	0.0328 (10)
H13A	0.6663	0.4637	1.0485	0.049*
H13B	0.6363	0.3249	1.1349	0.049*
H13C	0.6225	0.5508	1.1450	0.049*
C14	0.3817 (4)	0.4304 (7)	1.0890 (4)	0.0341 (10)
H14A	0.3828	0.5529	1.1250	0.051*
H14B	0.4016	0.3275	1.1416	0.051*
H14C	0.2960	0.4092	1.0391	0.051*
C15	0.4445 (5)	0.4205 (6)	0.9273 (4)	0.0241 (8)
H15	0.3550	0.4126	0.8906	0.029*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0161 (13)	0.0129 (12)	0.0185 (14)	−0.0016 (10)	0.0086 (11)	−0.0002 (11)
N2	0.0226 (14)	0.0170 (14)	0.0156 (13)	−0.0010 (10)	0.0079 (11)	0.0006 (10)
O1	0.0115 (12)	0.0128 (12)	0.0203 (13)	−0.0008 (9)	0.0061 (10)	0.0012 (10)
O2	0.0274 (14)	0.0139 (12)	0.0178 (13)	−0.0039 (10)	0.0125 (11)	−0.0033 (11)
O3	0.0272 (15)	0.0191 (12)	0.0205 (14)	0.0079 (10)	0.0072 (12)	0.0051 (11)
O4	0.0252 (13)	0.0200 (12)	0.0170 (12)	0.0001 (10)	0.0109 (10)	0.0032 (10)
O5	0.0146 (12)	0.0199 (12)	0.0185 (12)	0.0001 (9)	0.0081 (10)	−0.0027 (10)
C1	0.0138 (14)	0.0141 (17)	0.0181 (15)	0.0027 (10)	0.0064 (12)	0.0005 (11)
C2	0.0163 (15)	0.0147 (15)	0.0158 (15)	0.0024 (11)	0.0049 (12)	0.0002 (12)
C3	0.0156 (15)	0.0140 (13)	0.0132 (14)	−0.0010 (11)	0.0065 (12)	−0.0021 (11)
C4	0.0130 (14)	0.0130 (14)	0.0135 (15)	0.0004 (11)	0.0052 (12)	−0.0024 (13)
C5	0.0108 (14)	0.0131 (13)	0.0137 (15)	−0.0002 (10)	0.0047 (12)	−0.0014 (12)
C6	0.0170 (15)	0.0132 (13)	0.0135 (15)	0.0008 (11)	0.0075 (12)	−0.0004 (12)
C7	0.038 (2)	0.0196 (17)	0.0184 (17)	−0.0027 (15)	0.0156 (16)	−0.0031 (14)
C8	0.0205 (14)	0.011 (2)	0.0169 (14)	−0.0008 (10)	0.0085 (11)	0.0021 (11)
C9	0.0175 (16)	0.0153 (15)	0.0237 (18)	−0.0017 (12)	0.0129 (14)	−0.0021 (13)
C10	0.0234 (17)	0.0175 (15)	0.0241 (18)	−0.0043 (13)	0.0149 (15)	−0.0032 (14)
C11	0.0362 (17)	0.0203 (18)	0.0148 (13)	0.001 (2)	0.0072 (12)	0.005 (2)
C12	0.042 (2)	0.030 (3)	0.0215 (16)	0.0067 (18)	0.0031 (15)	0.0061 (18)
I1	0.02599 (10)	0.01685 (9)	0.01752 (9)	0.00241 (13)	0.00673 (7)	0.00025 (13)
N3	0.0222 (16)	0.0241 (16)	0.0226 (17)	−0.0012 (13)	0.0040 (13)	0.0042 (14)
O7	0.0305 (16)	0.0392 (17)	0.0239 (15)	0.0009 (13)	0.0108 (13)	0.0044 (13)
C13	0.028 (2)	0.041 (2)	0.024 (2)	0.003 (2)	−0.0027 (18)	0.0025 (19)
C14	0.033 (2)	0.042 (2)	0.031 (2)	−0.0034 (19)	0.0139 (19)	0.005 (2)
C15	0.022 (2)	0.0260 (18)	0.022 (2)	−0.0009 (17)	0.0021 (18)	0.0034 (17)

Geometric parameters (Å, º)

N1—C8	1.324 (4)	C5—H5	1.0000
N1—C9	1.383 (4)	C6—H6A	0.9900
N1—C6	1.472 (4)	C6—H6B	0.9900
N2—C8	1.339 (4)	C7—H7A	0.9800
N2—C10	1.379 (5)	C7—H7B	0.9800
N2—C11	1.424 (4)	C7—H7C	0.9800
O1—C1	1.421 (4)	C8—H8	0.9500
O1—C5	1.446 (4)	C9—C10	1.345 (5)
O2—C1	1.396 (4)	C9—H9	0.9500
O2—C7	1.428 (5)	C10—H10	0.9500
O3—C2	1.416 (4)	C11—C12	1.298 (6)
O3—H3A	0.72 (4)	C11—H11	0.9500
O4—C3	1.430 (4)	C12—H12A	0.9500
O4—H4A	0.78 (5)	C12—H12B	0.9500
O5—C4	1.424 (4)	N3—C15	1.322 (6)
O5—H5A	0.74 (5)	N3—C14	1.442 (6)
C1—C2	1.523 (5)	N3—C13	1.453 (5)
C1—H1	1.0000	O7—C15	1.243 (5)
C2—C3	1.536 (5)	C13—H13A	0.9800
C2—H2	1.0000	C13—H13B	0.9800
C3—C4	1.523 (5)	C13—H13C	0.9800
C3—H3	1.0000	C14—H14A	0.9800
C4—C5	1.527 (5)	C14—H14B	0.9800
C4—H4	1.0000	C14—H14C	0.9800
C5—C6	1.509 (4)	C15—H15	0.9500
C8—N1—C9	108.9 (3)	C5—C6—H6A	109.6
C8—N1—C6	124.9 (3)	N1—C6—H6B	109.6
C9—N1—C6	126.0 (3)	C5—C6—H6B	109.6
C8—N2—C10	108.2 (3)	H6A—C6—H6B	108.1
C8—N2—C11	127.4 (3)	O2—C7—H7A	109.5
C10—N2—C11	124.2 (3)	O2—C7—H7B	109.5
C1—O1—C5	113.8 (3)	H7A—C7—H7B	109.5
C1—O2—C7	112.6 (3)	O2—C7—H7C	109.5
C2—O3—H3A	105 (4)	H7A—C7—H7C	109.5
C3—O4—H4A	117 (4)	H7B—C7—H7C	109.5
C4—O5—H5A	108 (3)	N1—C8—N2	108.5 (3)
O2—C1—O1	112.4 (3)	N1—C8—H8	125.8
O2—C1—C2	108.8 (3)	N2—C8—H8	125.8
O1—C1—C2	109.3 (3)	C10—C9—N1	106.9 (3)
O2—C1—H1	108.8	C10—C9—H9	126.6
O1—C1—H1	108.8	N1—C9—H9	126.6
C2—C1—H1	108.8	C9—C10—N2	107.5 (3)
O3—C2—C1	109.2 (3)	C9—C10—H10	126.2
O3—C2—C3	112.5 (3)	N2—C10—H10	126.2
C1—C2—C3	110.9 (3)	C12—C11—N2	123.8 (3)

O3—C2—H2	108.0	C12—C11—H11	118.1
C1—C2—H2	108.0	N2—C11—H11	118.1
C3—C2—H2	108.0	C11—C12—H12A	120.0
O4—C3—C4	108.1 (3)	C11—C12—H12B	120.0
O4—C3—C2	110.0 (3)	H12A—C12—H12B	120.0
C4—C3—C2	109.4 (3)	C15—N3—C14	121.8 (4)
O4—C3—H3	109.8	C15—N3—C13	121.5 (4)
C4—C3—H3	109.8	C14—N3—C13	116.7 (4)
C2—C3—H3	109.8	N3—C13—H13A	109.5
O5—C4—C3	109.9 (3)	N3—C13—H13B	109.5
O5—C4—C5	108.6 (3)	H13A—C13—H13B	109.5
C3—C4—C5	108.9 (3)	N3—C13—H13C	109.5
O5—C4—H4	109.8	H13A—C13—H13C	109.5
C3—C4—H4	109.8	H13B—C13—H13C	109.5
C5—C4—H4	109.8	N3—C14—H14A	109.5
O1—C5—C6	105.7 (3)	N3—C14—H14B	109.5
O1—C5—C4	110.4 (3)	H14A—C14—H14B	109.5
C6—C5—C4	112.8 (3)	N3—C14—H14C	109.5
O1—C5—H5	109.3	H14A—C14—H14C	109.5
C6—C5—H5	109.3	H14B—C14—H14C	109.5
C4—C5—H5	109.3	O7—C15—N3	125.3 (5)
N1—C6—C5	110.4 (3)	O7—C15—H15	117.4
N1—C6—H6A	109.6	N3—C15—H15	117.4

C7—O2—C1—O1	65.7 (4)	O5—C4—C5—C6	64.9 (3)
C7—O2—C1—C2	−173.1 (3)	C3—C4—C5—C6	−175.4 (3)
C5—O1—C1—O2	61.2 (4)	C8—N1—C6—C5	117.5 (3)
C5—O1—C1—C2	−59.7 (3)	C9—N1—C6—C5	−56.3 (4)
O2—C1—C2—O3	57.7 (3)	O1—C5—C6—N1	74.9 (3)
O1—C1—C2—O3	−179.2 (3)	C4—C5—C6—N1	−164.3 (3)
O2—C1—C2—C3	−66.8 (3)	C9—N1—C8—N2	−0.8 (4)
O1—C1—C2—C3	56.3 (3)	C6—N1—C8—N2	−175.6 (3)
O3—C2—C3—O4	63.2 (4)	C10—N2—C8—N1	0.9 (4)
C1—C2—C3—O4	−174.2 (3)	C11—N2—C8—N1	176.6 (4)
O3—C2—C3—C4	−178.2 (3)	C8—N1—C9—C10	0.5 (4)
C1—C2—C3—C4	−55.6 (3)	C6—N1—C9—C10	175.1 (3)
O4—C3—C4—O5	−66.2 (3)	N1—C9—C10—N2	0.1 (4)
C2—C3—C4—O5	174.1 (3)	C8—N2—C10—C9	−0.6 (4)
O4—C3—C4—C5	175.0 (3)	C11—N2—C10—C9	−176.5 (4)
C2—C3—C4—C5	55.3 (3)	C8—N2—C11—C12	−6.4 (8)
C1—O1—C5—C6	−176.5 (3)	C10—N2—C11—C12	168.6 (5)
C1—O1—C5—C4	61.2 (3)	C14—N3—C15—O7	−178.8 (4)
O5—C4—C5—O1	−177.0 (3)	C13—N3—C15—O7	−0.4 (7)
C3—C4—C5—O1	−57.3 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O7	0.72 (4)	2.09 (4)	2.797 (4)	167 (5)
O4—H4*A*···I1^{i}	0.78 (5)	2.71 (5)	3.482 (3)	171 (4)
O5—H5*A*···I1	0.74 (5)	2.75 (5)	3.474 (3)	165 (4)
C6—H6*A*···O5^{ii}	0.99	2.46	3.332 (4)	147
C8—H8···O4^{ii}	0.95	2.44	3.252 (4)	143
C8—H8···O5^{ii}	0.95	2.53	3.285 (4)	136
C9—H9···O3^{iii}	0.95	2.51	3.404 (5)	156
C10—H10···O7^{iii}	0.95	2.40	3.159 (5)	137
C11—H11···I1^{iv}	0.95	3.02	3.925 (3)	161
C15—H15···O4	0.95	2.58	3.297 (5)	132

Symmetry codes: (i) $x, y-1, z$; (ii) $-x, y+1/2, -z+1$; (iii) $-x+1, y+1/2, -z+1$; (iv) $x, y, z-1$.