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Isopropyl 6-amino-2,5-anhydro-6-*N*-(2,5-anhydro-6-azido-3,6-dideoxy-L-*lyxo*-hexonyl)-3,6-dideoxy-L-*lyxo*-hexonate 2.5-hydrate

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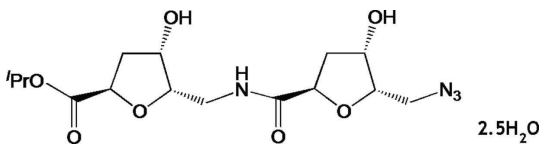
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.004 Å; R factor = 0.041; wR factor = 0.105; data-to-parameter ratio = 8.8.

The title compound, $C_{15}H_{24}N_4O_7\cdot2.5H_2O$, was crystallized with great difficulty from an aqueous solution after several months at 278 K. The crystal structure contains two independent molecules in the asymmetric unit, together with five molecules of water. The independent molecules, which have a very similar geometry, are related by a non-crystallographic pseudo-twofold rotation axis approximately perpendicular to the *bc* plane. Some intramolecular hydrogen bonds are present in both of the independent molecules. Moreover, the water molecules play a key role in the formation of a hydrogen-bond network.

Related literature

The corresponding L-*arabino* dimer, non-hydrate, exhibits one internal hydrogen bond similar to the one observed in the title compound (Cooper *et al.*, 2003). The L-*arabino* dimer adopts an extended conformation in contrast to the substantially folded conformation of the title compound reported here.

For related literature, see: Claridge *et al.* (2005); Gruner *et al.* (2002); Risseeuw *et al.* (2007); Schweizer (2002); Trabocchi *et al.* (2005); Watterson *et al.* (2003).



Experimental

Crystal data

$C_{15}H_{24}N_4O_7\cdot2.5H_2O$
M_r = 417.42
Triclinic, $P1$
a = 5.7521 (1) Å
b = 9.0344 (2) Å
c = 20.7349 (3) Å
α = 95.1380 (11)°
β = 96.3386 (12)°
γ = 108.4637 (6)°
V = 1006.91 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 150 K
0.30 × 0.10 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.99, T_{max} = 1.00
(expected range = 0.988–0.998)
6989 measured reflections
4529 independent reflections
3861 reflections with $I > 2\sigma(I)$
R_{int} = 0.000

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.105
S = 0.98
4509 reflections
515 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C20—H202···N1^{i}	0.96	2.43	3.340 (4)	158
C120—H1202···N101^{ii}	0.96	2.56	3.477 (4)	161
N109—H1091···O54	0.89	2.03	2.880 (4)	159
N9—H91···O53	0.86	2.08	2.905 (4)	160
O22—H221···O56^{iii}	0.82	1.93	2.744 (4)	173
O57—H571···O55^{iv}	0.83	2.06	2.852 (4)	161
O56—H562···O55^{iv}	0.82	2.04	2.847 (4)	167
O53—H532···O19	0.85	2.13	2.969 (4)	175
O55—H552···O23	0.83	1.96	2.786 (4)	170
O54—H542···O119	0.82	2.09	2.912 (4)	178
O122—H1221···O57^{iii}	0.82	1.93	2.748 (4)	176
O57—H572···O126^{v}	0.84	1.99	2.820 (4)	172
O54—H541···N103	0.84	2.18	2.990 (4)	163
O55—H551···O123	0.82	1.99	2.790 (4)	165
O53—H531···N3	0.83	2.27	3.038 (4)	152
O56—H561···O26^{vi}	0.81	2.02	2.822 (4)	170
O26—H261···O123^{vii}	0.84	1.85	2.684 (4)	175
O126—H1261···O23^{viii}	0.83	1.87	2.694 (4)	177

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

Data collection: *COLLECT* (Nonius, 1997); cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2559).

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supplementary materials

Acta Cryst. (2007). E**63**, o4719-o4720 [**doi:10.1107/S1600536807056905**]

Isopropyl 6-amino-2,5-anhydro-6-*N*-(2,5-anhydro-6-azido-3,6-dideoxy-L-*lyxo*-hexonyl)-3,6-dideoxy-L-*lyxo*-hexonate 2.5-hydrate

D. D. Le Pevelen, A. A. Edwards, G. E. Tranter, D. J. Watkin and G. W. J. Fleet

Comment

Sugar amino acids (SAAs) are highly functionalized stereodiverse scaffolds which have commonly been utilized as dipeptide isosteres and molecular scaffolds for compound libraries and foldamers (Schweizer, 2002, Gruner *et al.*, 2002, Trabocchi *et al.*, 2005). A recent review highlights the range of cyclically constrained SAA scaffolds available (Risseeuw *et al.*, 2007). To successfully utilize SAA systems for the preparation of biologically active systems, a comprehensive understanding of their conformational flexibility and preference is necessary. The 2,5-*cis* related furanose-based SAAs have been repeatedly found to adopt β-turn-type conformations whereas only one of the numerous 2,5-*O-trans* related systems (Fig.4) has exhibited a conformational preference for a compact hydrogen bonded conformation - the D-*talo* octamer of SAA 1 adopted a left-handed helical conformation (Claridge *et al.*, 2005). Two analogous systems have now been prepared and are being studied to ascertain their conformational preference. The oligomers of the *L-lyxo* SAA 2 (n = 1, 3, 7) and *L-arabino* SAA 3 (n = 1, 3) are crystalline solids. The *L-arabino* dimer 4 has been previously reported (Cooper *et al.*, 2003) and herein we report the crystal structure of the *L-lyxo* dimer 5.

The crystal structure contains two molecules of the title compound in the asymmetric unit, together with five molecules of water of crystallization (Fig. 1). The independent molecules are related by a non-crystallographic pseudo-2-fold rotation axis approximately perpendicular to the *bc* plane of form (0.08+*X*, 0.84-Y, 0.38-*Z*). The geometry of the two molecules is very similar (Fig.2). After superimposing the two molecules, including the H atoms, the r.m.s. positional deviations were 0.38 Å, the r.m.s. bond deviations were 0.0103 Å, and the r.m.s. torsion angle deviations were 4.16°.

The conformation of both molecules is more compact than in dimer 4 [N1···N9···C16 = 67.74 (5)° and 65.37 (4)° for dimer 5; 176.39 (7)° for dimer 4]. Like dimer 4, dimer 5 exhibits the internal hydrogen bond between the NH of the amine group and the ether oxygen of the ring containing the azide. In addition, a second hydrogen bond (weaker) is observed between the NH of the amine group and the ether oxygen of the ring containing the isopropyl ester. Water molecules are involved in a hydrogen bonding network in the structure (Fig. 3).

The corresponding *L-arabino* dimer, non-hydrate, exhibits one internal hydrogen bond similar to one observed in the title compound (Cooper *et al.*, 2003). The conformation of the *L-arabino* dimer is more extended than the title compound [N1···N9···C16 = 176.39 (7)° for the *L-arabino* whereas it is 67.74 (5)° and 65.37 (4)° for the two crystallographically independent dimers of the *L-lyxo* reported here].

Experimental

The title compound was prepared by selective deprotection of the corresponding azido ester (Watterson *et al.*, 2003) with subsequent coupling using standard peptide coupling reagents. The absolute chemistry has been determined by the use of *L*-gulono-1,4-lactone as the starting material for synthesis.

The title compound was crystallized with great difficulty from a water solution after several months at 278 K.

Refinement

In the absence of significant anomalous scattering, Friedel pairs were merged and the absolute configuration assigned from the starting material.

The H atoms were all located in a difference map, but those attached to carbon atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.93–0.98, N—H to 0.86 O—H = 0.82 Å) and U_{iso}(H) (in the range 1.2–1.5 times U_{eq} of the parent atom), after which the positions were refined with riding constraints.

The crystal was twinned with twin law 1,0,0; -.995,-1,0; -.796,0,-1 and twin fractions 0.936:0.064.

Of the 4529 independent reflections, 20 reflections were eliminated as they were poorly defined in the vicinity of the beam stop, leaving 4509 reflections in the refinement.

Figures

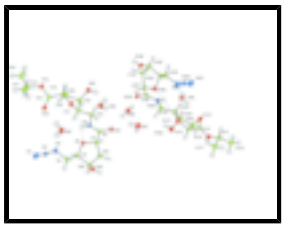

Fig. 1. The asymetric unit with displacement ellipsoids drawn at the 50% probability level. Hydrogen bonds are shown as dotted lines.

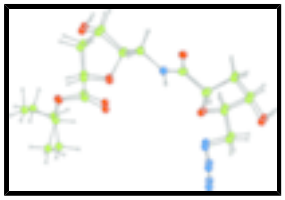

Fig. 2. Superimposition of the two independent molecules of the title compound.

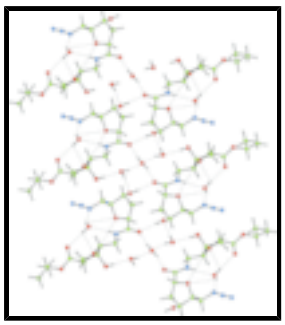

Fig. 3. The hydrogen bond network, viewed along the *a* axis. Hydrogen bonds are shown as dotted lines.

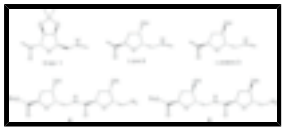

Fig. 4. Family of furanose-based SAA.

Isopropyl 6-amino-2,5-anhydro-6-*N*-(2,5-anhydro-6-azido-3,6-dideoxy-*L*-*lyxo*- hexonyl)-3,6-dideoxy-*L*-*lyxo*-hexonate 2.5-hydrate

Crystal data

$C_{15}H_{24}N_4O_7 \cdot 2.5H_2O$	$Z = 2$
$M_r = 417.42$	$F_{000} = 446$
Triclinic, $P\overline{1}$	$D_x = 1.377$ Mg m^{-3}
Hall symbol: P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 5.7521$ (1) Å	Cell parameters from 3641 reflections
$b = 9.0344$ (2) Å	$\theta = 3$–27º
$c = 20.7349$ (3) Å	$\mu = 0.12$ mm^{-1}
$\alpha = 95.1380$ (11)º	$T = 150$ K
$\beta = 96.3386$ (12)º	Plate, colourless
$\gamma = 108.4637$ (6)º	0.30 × 0.10 × 0.02 mm
$V = 1006.91$ (3) Å^3	

Data collection

Area diffractometer	3861 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.000$
$T = 150$ K	$\theta_{max} = 27.5$º
ω scans	$\theta_{min} = 3.0$º
Absorption correction: multi-scan (DENZO/SCALEPACK; Otwinowski & Minor, 1997)	$h = -7 \rightarrow 7$
$T_{min} = 0.99$, $T_{max} = 1.00$	$k = -11 \rightarrow 10$
6989 measured reflections	$l = -26 \rightarrow 26$
4529 independent reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.06P)^2 + 0.03P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.041$	$(\Delta/\sigma)_{max} = 0.0003$
$wR(F^2) = 0.105$	$\Delta\rho_{max} = 0.29$ e Å^{-3}
$S = 0.98$	$\Delta\rho_{min} = -0.29$ e Å^{-3}
4509 reflections	Extinction correction: None
515 parameters	
3 restraints	
Primary atom site location: structure-invariant direct methods	

Hydrogen site location: inferred from neighbouring sites

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	1.1480 (8)	−0.1003 (4)	0.54330 (17)	0.0553
N2	1.0155 (6)	−0.0798 (3)	0.50328 (14)	0.0366
N3	0.8662 (6)	−0.0461 (3)	0.46578 (13)	0.0340
C4	0.8007 (6)	−0.1396 (4)	0.39974 (14)	0.0267
C5	0.5885 (6)	−0.1046 (3)	0.36262 (14)	0.0219
O6	0.6720 (4)	0.0549 (2)	0.34711 (10)	0.0231
C7	0.5367 (6)	0.0630 (3)	0.28606 (14)	0.0209
C8	0.3955 (5)	0.1777 (3)	0.29303 (14)	0.0203
N9	0.3844 (5)	0.2368 (3)	0.35259 (12)	0.0219
C10	0.2456 (6)	0.3457 (3)	0.36306 (14)	0.0217
C11	0.2341 (6)	0.3783 (3)	0.43479 (14)	0.0226
O12	0.4819 (4)	0.4688 (3)	0.46789 (10)	0.0254
C13	0.4617 (6)	0.5692 (4)	0.52265 (14)	0.0256
C14	0.5845 (6)	0.5312 (4)	0.58522 (14)	0.0239
O15	0.6542 (4)	0.6540 (3)	0.63143 (10)	0.0285
C16	0.7607 (6)	0.6339 (4)	0.69680 (15)	0.0278
C17	1.0229 (7)	0.6369 (5)	0.69595 (17)	0.0376
C18	0.7380 (7)	0.7645 (5)	0.74312 (16)	0.0398
O19	0.6059 (5)	0.4045 (3)	0.59183 (11)	0.0352
C20	0.1847 (6)	0.5371 (4)	0.52393 (15)	0.0287
C21	0.0758 (5)	0.4798 (3)	0.45240 (14)	0.0224
O22	0.1214 (5)	0.6134 (3)	0.41827 (11)	0.0294
O23	0.2885 (4)	0.2072 (2)	0.24214 (10)	0.0264
C24	0.3585 (6)	−0.1047 (3)	0.26108 (14)	0.0231
C25	0.4800 (6)	−0.2067 (3)	0.29702 (14)	0.0229
O26	0.6771 (4)	−0.2309 (2)	0.26598 (10)	0.0253
N101	1.3609 (6)	0.9360 (4)	−0.16217 (15)	0.0430
N102	1.2486 (5)	0.9162 (3)	−0.12063 (13)	0.0287
N103	1.1022 (5)	0.8837 (3)	−0.08056 (13)	0.0310
C104	1.1920 (6)	0.9798 (4)	−0.01428 (14)	0.0260
C105	0.9768 (5)	0.9462 (3)	0.02350 (13)	0.0201
O106	0.9135 (4)	0.7865 (2)	0.03906 (10)	0.0222
C107	0.8188 (5)	0.7781 (3)	0.10015 (13)	0.0196
C108	0.5566 (6)	0.6648 (3)	0.09328 (14)	0.0207
N109	0.4378 (4)	0.6060 (3)	0.03371 (11)	0.0203
C110	0.1839 (5)	0.4979 (3)	0.02318 (14)	0.0209
C111	0.0841 (5)	0.4630 (3)	−0.04904 (14)	0.0210
O112	0.2180 (4)	0.3763 (3)	−0.08116 (10)	0.0252
C113	0.0622 (6)	0.2792 (3)	−0.13722 (14)	0.0225
C114	0.1831 (6)	0.3203 (3)	−0.19792 (14)	0.0232
O115	0.0628 (4)	0.2117 (3)	−0.24931 (10)	0.0315
C116	0.1684 (6)	0.2270 (4)	−0.31084 (14)	0.0270
C117	0.3500 (7)	0.1386 (4)	−0.31054 (17)	0.0353

C118	−0.0472 (7)	0.1614 (4)	−0.36520 (15)	0.0341
O119	0.3565 (4)	0.4357 (3)	−0.20020 (10)	0.0308
C120	−0.1860 (6)	0.3054 (4)	−0.13939 (15)	0.0255
C121	−0.1882 (5)	0.3623 (4)	−0.06797 (14)	0.0232
O122	−0.2494 (4)	0.2277 (3)	−0.03410 (10)	0.0292
O123	0.4608 (4)	0.6358 (2)	0.14401 (10)	0.0260
C124	0.8307 (5)	0.9474 (3)	0.12536 (14)	0.0217
C125	1.0258 (5)	1.0485 (3)	0.08948 (14)	0.0208
O126	1.2726 (4)	1.0706 (2)	0.12008 (10)	0.0249
O53	0.5427 (6)	0.1600 (3)	0.47955 (12)	0.0464
O54	0.5683 (5)	0.6885 (3)	−0.09160 (11)	0.0364
O55	0.0778 (4)	0.4194 (3)	0.19227 (11)	0.0277
O56	0.8518 (4)	0.5205 (3)	0.29533 (11)	0.0329
O57	0.6683 (4)	0.3181 (3)	0.08906 (11)	0.0334
H41	0.7551	−0.2519	0.4039	0.0338*
H42	0.9401	−0.1129	0.3760	0.0337*
H51	0.4551	−0.1189	0.3905	0.0267*
H71	0.6588	0.1006	0.2563	0.0252*
H102	0.3230	0.4449	0.3463	0.0258*
H101	0.0744	0.2932	0.3406	0.0266*
H111	0.1781	0.2772	0.4522	0.0275*
H131	0.5438	0.6800	0.5186	0.0304*
H161	0.6594	0.5335	0.7080	0.0316*
H171	1.0907	0.6247	0.7382	0.0553*
H172	1.1142	0.7354	0.6830	0.0549*
H173	1.0256	0.5518	0.6646	0.0549*
H181	0.7918	0.7525	0.7877	0.0559*
H182	0.8391	0.8638	0.7319	0.0560*
H183	0.5702	0.7643	0.7391	0.0552*
H202	0.1520	0.6306	0.5396	0.0378*
H201	0.1209	0.4575	0.5521	0.0376*
H211	−0.1038	0.4196	0.4458	0.0290*
H242	0.1963	−0.1175	0.2745	0.0274*
H241	0.3416	−0.1294	0.2131	0.0260*
H251	0.3617	−0.3069	0.3030	0.0266*
H1042	1.2458	1.0917	−0.0194	0.0291*
H1041	1.3265	0.9502	0.0073	0.0287*
H1051	0.8304	0.9573	−0.0026	0.0239*
H1071	0.9237	0.7412	0.1299	0.0225*
H1101	0.0766	0.5433	0.0462	0.0234*
H1102	0.1824	0.4004	0.0408	0.0227*
H1111	0.1136	0.5618	−0.0680	0.0238*
H1131	0.0455	0.1719	−0.1313	0.0255*
H1161	0.2527	0.3387	−0.3139	0.0317*
H1173	0.4150	0.1415	−0.3519	0.0516*
H1171	0.2746	0.0283	−0.3038	0.0516*
H1172	0.4870	0.1902	−0.2752	0.0522*
H1181	0.0107	0.1763	−0.4065	0.0505*
H1182	−0.1693	0.2150	−0.3616	0.0502*

H1183	−0.1213	0.0506	−0.3630	0.0515*
H1201	−0.1914	0.3859	−0.1673	0.0286*
H1202	−0.3197	0.2102	−0.1560	0.0286*
H1211	−0.3012	0.4226	−0.0620	0.0275*
H1242	0.8766	0.9742	0.1731	0.0242*
H1241	0.6743	0.9643	0.1137	0.0250*
H1251	1.0022	1.1497	0.0827	0.0234*
H1091	0.5148	0.6300	−0.0009	0.0231*
H91	0.4608	0.2109	0.3857	0.0261*
H221	0.0327	0.5890	0.3829	0.0461*
H571	0.7637	0.3344	0.1237	0.0469*
H562	0.9155	0.5042	0.2630	0.0526*
H532	0.5703	0.2313	0.5115	0.0748*
H552	0.1297	0.3556	0.2106	0.0412*
H542	0.5083	0.6152	−0.1216	0.0498*
H1221	−0.2784	0.2567	0.0017	0.0410*
H572	0.5423	0.2502	0.0977	0.0473*
H541	0.7156	0.7337	−0.0965	0.0484*
H551	0.1729	0.4774	0.1716	0.0410*
H531	0.6460	0.1158	0.4897	0.0749*
H561	0.7893	0.5840	0.2837	0.0531*
H261	0.6181	−0.2700	0.2273	0.0393*
H1261	1.2778	1.1091	0.1581	0.0364*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.078 (3)	0.0464 (19)	0.0412 (19)	0.0365 (19)	−0.0249 (18)	−0.0114 (15)
N2	0.056 (2)	0.0258 (13)	0.0274 (15)	0.0203 (13)	−0.0092 (14)	−0.0074 (11)
N3	0.0495 (19)	0.0382 (16)	0.0184 (14)	0.0247 (14)	−0.0047 (12)	−0.0018 (11)
C4	0.0340 (18)	0.0325 (16)	0.0174 (14)	0.0191 (14)	−0.0007 (12)	−0.0018 (12)
C5	0.0280 (16)	0.0203 (14)	0.0181 (14)	0.0095 (12)	0.0040 (11)	0.0001 (11)
O6	0.0276 (11)	0.0192 (10)	0.0193 (10)	0.0077 (8)	−0.0068 (8)	−0.0012 (8)
C7	0.0242 (15)	0.0213 (13)	0.0163 (13)	0.0080 (12)	−0.0013 (11)	0.0005 (11)
C8	0.0218 (15)	0.0184 (13)	0.0175 (14)	0.0027 (11)	0.0011 (11)	0.0024 (10)
N9	0.0287 (14)	0.0206 (11)	0.0171 (12)	0.0118 (10)	−0.0027 (10)	0.0001 (9)
C10	0.0248 (15)	0.0222 (14)	0.0183 (14)	0.0101 (12)	−0.0007 (11)	0.0002 (11)
C11	0.0258 (15)	0.0219 (13)	0.0197 (14)	0.0083 (12)	0.0011 (11)	0.0020 (11)
O12	0.0215 (11)	0.0362 (12)	0.0183 (10)	0.0127 (9)	0.0001 (8)	−0.0058 (8)
C13	0.0328 (17)	0.0288 (16)	0.0164 (14)	0.0129 (14)	0.0023 (12)	−0.0002 (11)
C14	0.0250 (16)	0.0279 (15)	0.0174 (14)	0.0077 (12)	0.0016 (12)	0.0010 (11)
O15	0.0355 (13)	0.0285 (11)	0.0188 (10)	0.0103 (9)	−0.0019 (9)	−0.0026 (8)
C16	0.0291 (17)	0.0338 (17)	0.0162 (14)	0.0052 (14)	0.0007 (12)	0.0027 (12)
C17	0.0349 (19)	0.047 (2)	0.0278 (18)	0.0142 (16)	−0.0041 (14)	0.0000 (14)
C18	0.0293 (18)	0.059 (2)	0.0232 (17)	0.0091 (17)	0.0007 (14)	−0.0096 (15)
O19	0.0491 (15)	0.0292 (12)	0.0252 (12)	0.0141 (11)	−0.0040 (10)	0.0005 (9)
C20	0.0354 (18)	0.0356 (17)	0.0232 (16)	0.0220 (15)	0.0078 (13)	0.0037 (13)
C21	0.0167 (14)	0.0295 (15)	0.0253 (15)	0.0132 (12)	0.0045 (11)	0.0031 (12)

O22	0.0383 (13)	0.0297 (11)	0.0242 (11)	0.0189 (10)	−0.0012 (9)	0.0020 (9)
O23	0.0360 (13)	0.0289 (11)	0.0158 (10)	0.0155 (10)	−0.0032 (9)	0.0012 (8)
C24	0.0239 (15)	0.0220 (14)	0.0212 (14)	0.0074 (11)	−0.0007 (11)	−0.0030 (11)
C25	0.0254 (15)	0.0202 (14)	0.0218 (14)	0.0078 (12)	0.0006 (11)	−0.0013 (11)
O26	0.0263 (11)	0.0339 (12)	0.0190 (10)	0.0170 (10)	0.0002 (8)	−0.0027 (9)
N101	0.0458 (19)	0.0470 (18)	0.0308 (16)	0.0052 (15)	0.0179 (15)	−0.0006 (13)
N102	0.0273 (14)	0.0296 (13)	0.0239 (14)	0.0040 (11)	0.0037 (12)	−0.0034 (11)
N103	0.0221 (14)	0.0383 (15)	0.0222 (13)	−0.0035 (11)	0.0061 (11)	−0.0030 (11)
C104	0.0225 (16)	0.0291 (15)	0.0198 (15)	0.0011 (12)	0.0016 (12)	−0.0013 (12)
C105	0.0181 (14)	0.0223 (14)	0.0165 (13)	0.0031 (11)	−0.0004 (11)	0.0010 (11)
O106	0.0235 (11)	0.0208 (10)	0.0203 (10)	0.0038 (8)	0.0074 (8)	−0.0004 (8)
C107	0.0211 (14)	0.0213 (13)	0.0145 (13)	0.0048 (11)	0.0033 (10)	0.0002 (10)
C108	0.0263 (16)	0.0167 (13)	0.0177 (14)	0.0057 (11)	0.0032 (11)	−0.0003 (10)
N109	0.0193 (12)	0.0206 (12)	0.0177 (12)	0.0019 (9)	0.0048 (9)	0.0011 (9)
C110	0.0147 (13)	0.0224 (14)	0.0207 (14)	−0.0002 (11)	0.0037 (11)	0.0001 (11)
C111	0.0216 (15)	0.0206 (13)	0.0200 (14)	0.0054 (11)	0.0042 (11)	0.0023 (11)
O112	0.0177 (10)	0.0380 (12)	0.0178 (10)	0.0095 (9)	−0.0004 (8)	−0.0045 (8)
C113	0.0266 (16)	0.0236 (14)	0.0136 (13)	0.0045 (12)	0.0014 (11)	−0.0008 (10)
C114	0.0254 (15)	0.0254 (15)	0.0172 (14)	0.0092 (12)	−0.0032 (11)	−0.0007 (11)
O115	0.0344 (13)	0.0308 (12)	0.0190 (11)	−0.0021 (10)	0.0062 (9)	−0.0047 (9)
C116	0.0300 (17)	0.0301 (16)	0.0170 (15)	0.0039 (13)	0.0077 (12)	−0.0001 (12)
C117	0.0325 (19)	0.046 (2)	0.0262 (17)	0.0129 (16)	0.0006 (14)	0.0037 (14)
C118	0.039 (2)	0.0441 (19)	0.0194 (16)	0.0157 (16)	0.0030 (13)	−0.0004 (13)
O119	0.0311 (12)	0.0290 (11)	0.0214 (11)	−0.0033 (10)	0.0029 (9)	−0.0020 (9)
C120	0.0192 (14)	0.0306 (16)	0.0208 (15)	0.0017 (12)	−0.0003 (11)	0.0014 (12)
C121	0.0173 (14)	0.0309 (16)	0.0211 (15)	0.0079 (12)	0.0029 (11)	0.0031 (12)
O122	0.0295 (12)	0.0302 (11)	0.0215 (11)	0.0003 (10)	0.0055 (9)	0.0038 (9)
O123	0.0290 (12)	0.0271 (11)	0.0160 (10)	0.0014 (9)	0.0038 (8)	0.0008 (8)
C124	0.0190 (14)	0.0249 (14)	0.0190 (14)	0.0065 (11)	0.0005 (11)	−0.0029 (11)
C125	0.0186 (14)	0.0183 (13)	0.0227 (14)	0.0032 (11)	0.0010 (11)	0.0005 (11)
O126	0.0206 (11)	0.0294 (11)	0.0189 (10)	0.0030 (9)	0.0000 (8)	−0.0035 (8)
O53	0.069 (2)	0.0525 (16)	0.0271 (13)	0.0392 (15)	−0.0041 (12)	−0.0017 (11)
O54	0.0319 (13)	0.0394 (13)	0.0262 (12)	−0.0031 (10)	0.0041 (10)	−0.0010 (9)
O55	0.0279 (11)	0.0278 (10)	0.0268 (10)	0.0073 (9)	0.0035 (9)	0.0076 (8)
O56	0.0373 (13)	0.0404 (13)	0.0287 (12)	0.0227 (11)	0.0047 (10)	0.0077 (10)
O57	0.0291 (12)	0.0381 (13)	0.0264 (12)	0.0009 (10)	0.0048 (9)	0.0060 (10)

Geometric parameters (Å, °)

N1—N2	1.132 (4)	C104—H1041	0.973
N2—N3	1.223 (4)	C105—O106	1.447 (3)
N3—C4	1.486 (4)	C105—C125	1.526 (4)
C4—C5	1.504 (4)	C105—H1051	0.987
C4—H41	0.979	O106—C107	1.432 (3)
C4—H42	0.964	C107—C108	1.514 (4)
C5—O6	1.445 (3)	C107—C124	1.549 (4)
C5—C25	1.521 (4)	C107—H1071	0.968
C5—H51	0.994	C108—N109	1.319 (4)
O6—C7	1.432 (3)	C108—O123	1.251 (3)

C7—C8	1.512 (4)	N109—C110	1.457 (4)
C7—C24	1.541 (4)	N109—H1091	0.892
C7—H71	0.986	C110—C111	1.511 (4)
C8—N9	1.317 (4)	C110—H1101	0.984
C8—O23	1.255 (3)	C110—H1102	0.981
N9—C10	1.466 (4)	C111—O112	1.433 (4)
N9—H91	0.864	C111—C121	1.528 (4)
C10—C11	1.503 (4)	C111—H1111	0.981
C10—H102	0.979	O112—C113	1.419 (3)
C10—H101	0.987	C113—C114	1.523 (4)
C11—O12	1.455 (4)	C113—C120	1.516 (4)
C11—C21	1.531 (4)	C113—H1131	0.964
C11—H111	0.983	C114—O115	1.340 (3)
O12—C13	1.427 (4)	C114—O119	1.202 (4)
C13—C14	1.527 (4)	O115—C116	1.472 (3)
C13—C20	1.531 (5)	C116—C117	1.503 (5)
C13—H131	0.978	C116—C118	1.505 (5)
C14—O15	1.324 (4)	C116—H1161	0.983
C14—O19	1.207 (4)	C117—H1173	0.972
O15—C16	1.475 (4)	C117—H1171	0.982
C16—C17	1.502 (5)	C117—H1172	0.977
C16—C18	1.502 (5)	C118—H1181	0.958
C16—H161	0.974	C118—H1182	0.976
C17—H171	0.950	C118—H1183	0.963
C17—H172	0.958	C120—C121	1.526 (4)
C17—H173	0.966	C120—H1201	0.975
C18—H181	0.968	C120—H1202	0.959
C18—H182	0.966	C121—O122	1.426 (4)
C18—H183	0.959	C121—H1211	0.982
C20—C21	1.521 (4)	O122—H1221	0.817
C20—H202	0.959	C124—C125	1.518 (4)
C20—H201	0.974	C124—H1242	0.984
C21—O22	1.421 (4)	C124—H1241	0.968
C21—H211	0.990	C125—O126	1.434 (4)
O22—H221	0.816	C125—H1251	0.985
C24—C25	1.519 (4)	O126—H1261	0.826
C24—H242	0.977	O53—H532	0.845
C24—H241	0.988	O53—H531	0.833
C25—O26	1.429 (4)	O54—H542	0.825
C25—H251	0.973	O54—H541	0.838
O26—H261	0.836	O55—H552	0.830
N101—N102	1.126 (4)	O55—H551	0.817
N102—N103	1.236 (4)	O56—H562	0.822
N103—C104	1.501 (4)	O56—H561	0.807
C104—C105	1.502 (4)	O57—H571	0.825
C104—H1042	0.979	O57—H572	0.837
N1—N2—N3	172.3 (4)	N103—C104—H1042	109.2
N2—N3—C4	114.5 (3)	C105—C104—H1042	108.3
N3—C4—C5	108.8 (2)	N103—C104—H1041	108.7

N3—C4—H41	109.8	C105—C104—H1041	111.3
C5—C4—H41	110.5	H1042—C104—H1041	111.5
N3—C4—H42	110.7	C104—C105—O106	109.5 (2)
C5—C4—H42	109.3	C104—C105—C125	115.0 (2)
H41—C4—H42	107.8	O106—C105—C125	104.8 (2)
C4—C5—O6	109.6 (2)	C104—C105—H1051	110.7
C4—C5—C25	114.9 (2)	O106—C105—H1051	108.4
O6—C5—C25	105.0 (2)	C125—C105—H1051	108.2
C4—C5—H51	108.1	C105—O106—C107	109.6 (2)
O6—C5—H51	110.7	O106—C107—C108	112.3 (2)
C25—C5—H51	108.6	O106—C107—C124	106.4 (2)
C5—O6—C7	109.4 (2)	C108—C107—C124	110.9 (2)
O6—C7—C8	112.1 (2)	O106—C107—H1071	108.1
O6—C7—C24	106.8 (2)	C108—C107—H1071	108.1
C8—C7—C24	110.8 (2)	C124—C107—H1071	111.1
O6—C7—H71	107.1	C107—C108—N109	118.0 (2)
C8—C7—H71	108.1	C107—C108—O123	118.6 (2)
C24—C7—H71	111.9	N109—C108—O123	123.4 (3)
C7—C8—N9	117.9 (2)	C108—N109—C110	121.2 (2)
C7—C8—O23	118.5 (3)	C108—N109—H1091	119.9
N9—C8—O23	123.6 (3)	C110—N109—H1091	118.9
C8—N9—C10	120.9 (2)	N109—C110—C111	110.1 (2)
C8—N9—H91	119.1	N109—C110—H1101	110.7
C10—N9—H91	120.0	C111—C110—H1101	108.8
N9—C10—C11	110.1 (2)	N109—C110—H1102	108.6
N9—C10—H102	110.9	C111—C110—H1102	110.2
C11—C10—H102	109.5	H1101—C110—H1102	108.3
N9—C10—H101	107.6	C110—C111—O112	108.8 (2)
C11—C10—H101	107.7	C110—C111—C121	116.7 (2)
H102—C10—H101	111.0	O112—C111—C121	104.9 (2)
C10—C11—O12	108.5 (2)	C110—C111—H1111	109.7
C10—C11—C21	115.7 (2)	O112—C111—H1111	107.0
O12—C11—C21	104.2 (2)	C121—C111—H1111	109.2
C10—C11—H111	108.4	C111—O112—C113	109.0 (2)
O12—C11—H111	109.0	O112—C113—C114	109.4 (2)
C21—C11—H111	110.8	O112—C113—C120	107.0 (2)
C11—O12—C13	108.8 (2)	C114—C113—C120	112.9 (2)
O12—C13—C14	109.3 (2)	O112—C113—H1131	107.0
O12—C13—C20	106.7 (2)	C114—C113—H1131	109.6
C14—C13—C20	111.0 (3)	C120—C113—H1131	110.7
O12—C13—H131	111.2	C113—C114—O115	110.2 (2)
C14—C13—H131	108.4	C113—C114—O119	125.2 (3)
C20—C13—H131	110.2	O115—C114—O119	124.6 (3)
C13—C14—O15	110.5 (3)	C114—O115—C116	117.1 (2)
C13—C14—O19	124.2 (3)	O115—C116—C117	107.9 (3)
O15—C14—O19	125.2 (3)	O115—C116—C118	106.2 (3)
C14—O15—C16	117.7 (2)	C117—C116—C118	113.4 (3)
O15—C16—C17	109.8 (3)	O115—C116—H1161	109.7
O15—C16—C18	105.7 (3)	C117—C116—H1161	110.2

C17—C16—C18	113.7 (3)	C118—C116—H1161	109.4
O15—C16—H161	108.2	C116—C117—H1173	109.4
C17—C16—H161	110.4	C116—C117—H1171	112.2
C18—C16—H161	108.9	H1173—C117—H1171	109.1
C16—C17—H171	109.3	C116—C117—H1172	108.5
C16—C17—H172	107.8	H1173—C117—H1172	108.8
H171—C17—H172	111.4	H1171—C117—H1172	108.8
C16—C17—H173	109.7	C116—C118—H1181	109.5
H171—C17—H173	109.3	C116—C118—H1182	111.3
H172—C17—H173	109.3	H1181—C118—H1182	107.8
C16—C18—H181	109.7	C116—C118—H1183	108.2
C16—C18—H182	108.6	H1181—C118—H1183	109.7
H181—C18—H182	110.5	H1182—C118—H1183	110.3
C16—C18—H183	110.9	C113—C120—C121	102.8 (2)
H181—C18—H183	109.3	C113—C120—H1201	109.7
H182—C18—H183	107.7	C121—C120—H1201	111.3
C13—C20—C21	102.3 (2)	C113—C120—H1202	111.2
C13—C20—H202	111.4	C121—C120—H1202	112.7
C21—C20—H202	111.9	H1201—C120—H1202	109.1
C13—C20—H201	110.9	C111—C121—C120	99.5 (2)
C21—C20—H201	112.0	C111—C121—O122	111.5 (2)
H202—C20—H201	108.2	C120—C121—O122	107.6 (2)
C11—C21—C20	99.9 (2)	C111—C121—H1211	112.7
C11—C21—O22	112.1 (2)	C120—C121—H1211	113.9
C20—C21—O22	107.9 (2)	O122—C121—H1211	111.0
C11—C21—H211	113.0	C121—O122—H1221	106.6
C20—C21—H211	113.4	C107—C124—C125	102.8 (2)
O22—C21—H211	110.2	C107—C124—H1242	113.4
C21—O22—H221	109.2	C125—C124—H1242	111.5
C7—C24—C25	102.7 (2)	C107—C124—H1241	111.8
C7—C24—H242	109.9	C125—C124—H1241	109.8
C25—C24—H242	109.6	H1242—C124—H1241	107.5
C7—C24—H241	112.0	C105—C125—C124	101.5 (2)
C25—C24—H241	112.1	C105—C125—O126	108.9 (2)
H242—C24—H241	110.3	C124—C125—O126	112.1 (2)
C5—C25—C24	101.8 (2)	C105—C125—H1251	109.3
C5—C25—O26	108.9 (2)	C124—C125—H1251	112.8
C24—C25—O26	112.0 (2)	O126—C125—H1251	111.6
C5—C25—H251	110.5	C125—O126—H1261	104.4
C24—C25—H251	112.7	H532—O53—H531	103.1
O26—C25—H251	110.6	H542—O54—H541	107.4
C25—O26—H261	107.2	H552—O55—H551	116.3
N101—N102—N103	172.4 (3)	H562—O56—H561	100.7
N102—N103—C104	115.0 (2)	H571—O57—H572	101.3
N103—C104—C105	107.6 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

C20—H202···N1^{i}	0.96	2.43	3.340 (4)	158
C120—H1202···N101^{ii}	0.96	2.56	3.477 (4)	161
N109—H1091···O54	0.89	2.03	2.880 (4)	159
N9—H91···O53	0.86	2.08	2.905 (4)	160
O22—H221···O56^{iii}	0.82	1.93	2.744 (4)	173
O57—H571···O55^{iv}	0.83	2.06	2.852 (4)	161
O56—H562···O55^{iv}	0.82	2.04	2.847 (4)	167
O53—H532···O19	0.85	2.13	2.969 (4)	175
O55—H552···O23	0.83	1.96	2.786 (4)	170
O54—H542···O119	0.82	2.09	2.912 (4)	178
O122—H1221···O57^{iii}	0.82	1.93	2.748 (4)	176
O57—H572···O126^{v}	0.84	1.99	2.820 (4)	172
O54—H541···N103	0.84	2.18	2.990 (4)	163
O55—H551···O123	0.82	1.99	2.790 (4)	165
O53—H531···N3	0.83	2.27	3.038 (4)	152
O56—H561···O26^{vi}	0.81	2.02	2.822 (4)	170
O26—H261···O123^{vii}	0.84	1.85	2.684 (4)	175
O126—H1261···O23^{viii}	0.83	1.87	2.694 (4)	177

Symmetry codes: (i) *x*−1, *y*+1, *z*; (ii) *x*−2, *y*−1, *z*; (iii) *x*−1, *y*, *z*; (iv) *x*+1, *y*, *z*; (v) *x*−1, *y*−1, *z*; (vi) *x*, *y*+1, *z*; (vii) *x*, *y*−1, *z*; (viii) *x*+1, *y*+1, *z*.

Fig. 1

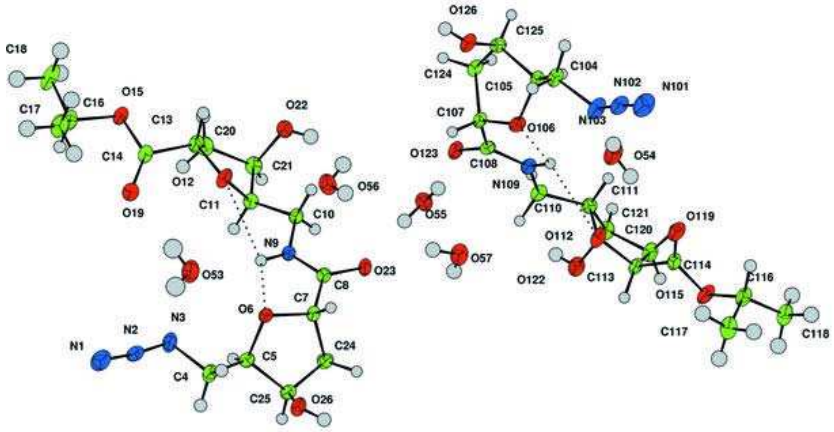

Fig. 2

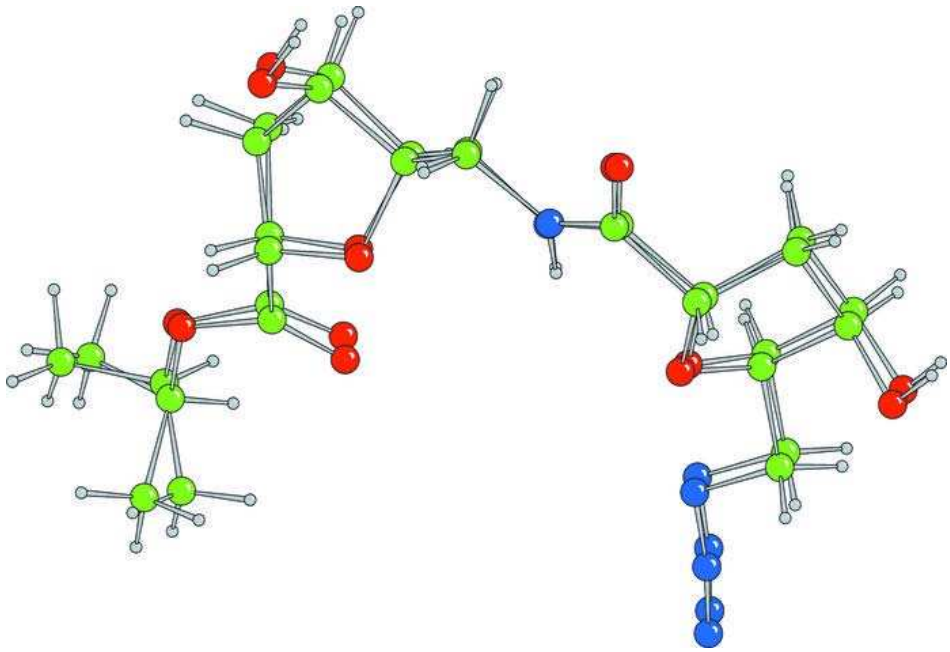

Fig. 3

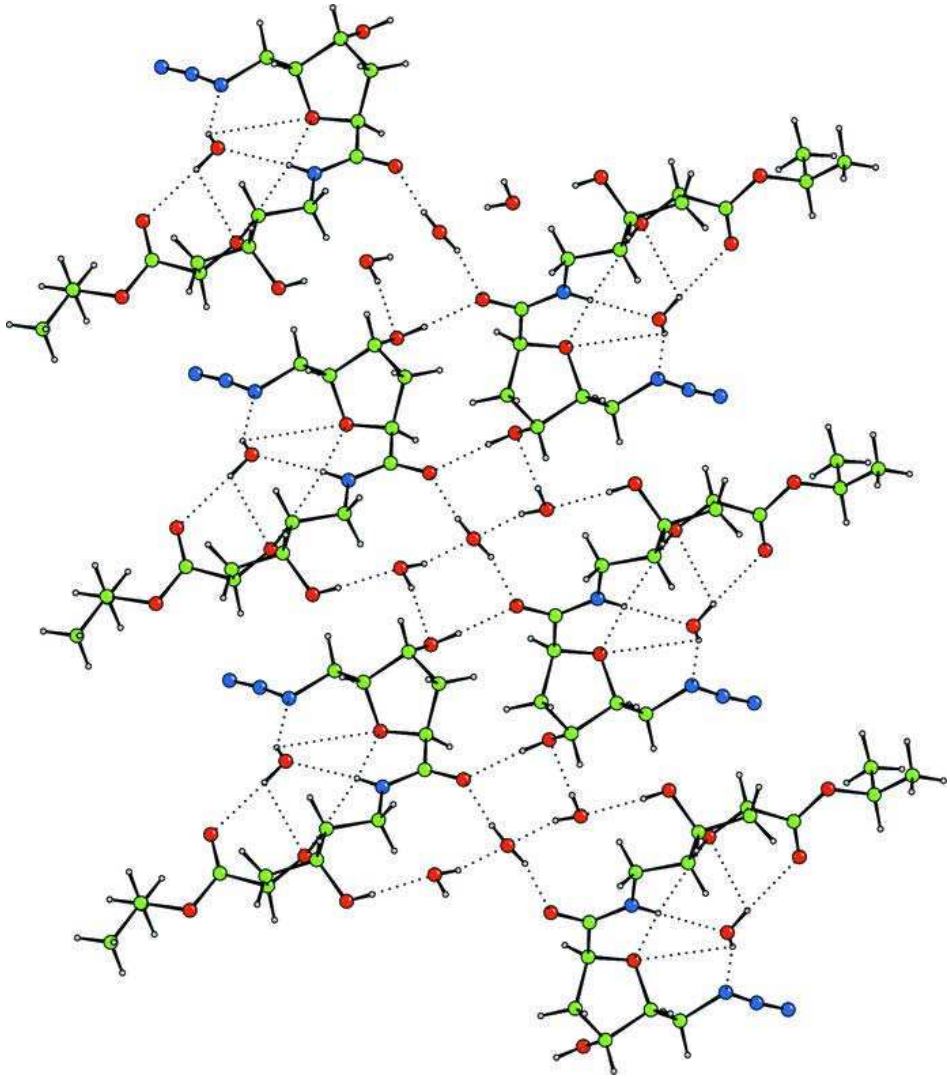

Fig. 4