Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3*a*,6*a*-Bis(ethoxycarbonyl)glycoluril (diethyl 2,5-dioxoperhydroimidazo-[4,5-*d*]imidazole-3a,6a-dicarboxylate)

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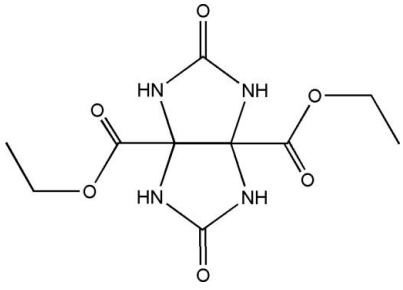
Received 30 October 2007; accepted 6 November 2007

Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.068; wR factor = 0.197; data-to-parameter ratio = 11.7.

The title compound, $C_{10}H_{14}N_4O_6$, crystallizes with two independent molecules in the asymmetric unit. An extensive network of N—H···O and C—H···O intermolecular hydrogen bonds stabilizes the crystal packing. One ethyl group is disordered over two positions; the site occupancy factors are 0.68 and 0.32.

Related literature

For related literature, see: Burnett *et al.* (2003); Chen *et al.* (2007); Himes *et al.* (1978); Hof *et al.* (2002); Isaacs & Witt (2002); Kim *et al.* (2000); Li *et al.* (1994); Moon *et al.* (2003); Rowan *et al.* (1999); Wang *et al.* (2006, 2007); Wu *et al.* (2002).



Experimental

Crystal data

$C_{10}H_{14}N_4O_6$
M_r = 286.25
Orthorhombic, *Pbca*
a = 15.7555 (13) Å
b = 11.2726 (9) Å
c = 28.742 (2) Å
V = 5104.7 (7) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 292 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
Absorption correction: none
29774 measured reflections
4501 independent reflections
2784 reflections with $I > 2\sigma(I)$
R_{int} = 0.097

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.197
S = 1.00
4501 reflections
385 parameters
6 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.86	2.15	2.963 (3)	158
N2—H2···O7^{ii}	0.86	2.10	2.927 (3)	162
N3—H3···O1^{iii}	0.86	2.11	2.937 (3)	161
N4—H4···O8^{iv}	0.86	2.21	2.879 (3)	134
N5—H5···O1^{iv}	0.86	2.22	2.870 (3)	133
N6—H6···O7^{v}	0.86	2.09	2.925 (3)	164
N7—H7···O2^{ii}	0.86	2.16	2.973 (3)	158
N8—H8···O8^{vi}	0.86	2.12	2.963 (3)	165
C16—H16*C*···O3^{vii}	0.96	2.51	3.087 (5)	119
C10—H10*B*···O11^{v}	0.96	2.32	3.162 (5)	146

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Bruker, 1997).

The authors are grateful to Central China Normal University for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2340).

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 doi:10.1107/S1600536807056292

supporting information

Acta Cryst. (2008). E**64**, o125 [https://doi.org/10.1107/S1600536807056292]

3*α*,6*α*-Bis(ethoxycarbonyl)glycoluril (diethyl 2,5-dioxoperhydroimidazo[4,5-*d*]imidazole-3a,6a-dicarboxylate)

Yu-Zhou Wang, Zhi-Guo Wang and Lin Li

S1. Comment

Glycoluril skeleton moiety (Fig. 3) is an important building block for both molecular and supramolecular chemistry. Its derivatives have been used as the basis for molecular capsules (Hof *et al.*, 2002), molecular clips (Rowan *et al.*, 1999), self-complementary facial amphiphiles (Isaacs *et al.*, 2002), and the cucurbit[*n*]uril (CB[*n*]) family (Kim *et al.*, 2000), and its utilization has been explored as a platform for studies of crystal engineering (Wang *et al.*, 2006; Chen *et al.*, 2007). However, relatively few crystal structures are known for glycoluril derivatives without N-substituents. The crystal structures of the reported glycoluril with different substituents exhibit two H-bonded types (Fig. 4). The mode A was found for (*R*=H) (Li *et al.*, 1994), (*R*=CH_3) (Himes *et al.*, 1978), (I, *R*=Ph) (Wu *et al.*, 2002), and so on, and the mode B was observed in the (*R*=Ph) (Moon *et al.*, 2003), (*R*=COO-n-C_3H_7) (Wang *et al.*, 2007). Herein, we report the crystal structure of the title compound (I), which exhibits the mode A of hydrogen bonding (Fig. 4).

The molecular structure of (I) (*R*=$COOC_2H_5$) is shown in Fig. 1. Its crystal structure exhibits the eight-membered rings H-bonding motifs (Fig. 4), which are entirely made up of *N*—*H*···*O*=*C*(imidazolone rings) (Table 1). The two-dimension hydrogen bonding network is shown in Fig. 2, In addition, intermolecular C10—H10B···O11, C16—H16C···O3 interactions (Table 1) contribute to the crystal structure stability.

S2. Experimental

The title compound was synthesized according to literature procedure (Burnett *et al.*, 2003) in 62% isolated yield. Crystals appropriate for data collection were obtained by slow evaporation of CH_3OH solution at room temperature.

S3. Refinement

One ethyl group (C5—C6) was treted as disordered over two orientations with the occupancies refined to 0.683 (15) / 0.317 (15). All H-atoms were positioned geometrically (C—H 0.96, 0.97 Å; N—H 0.86 Å) and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2–1.5U_{eq} (parent atom).

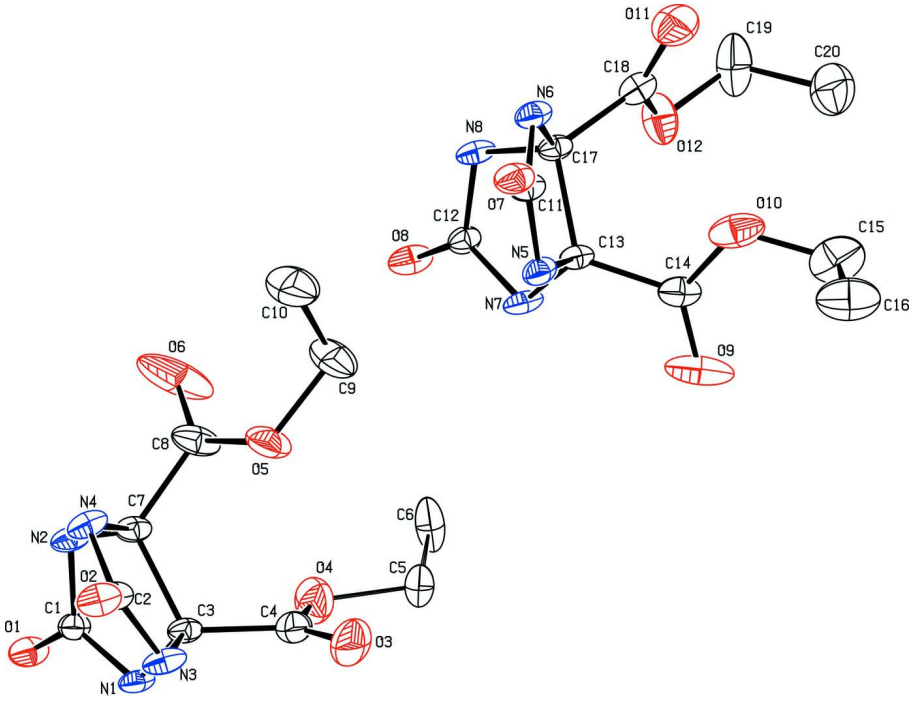

Figure 1

The content of asymmetric unit of (I), showing the atom-labelling scheme and 50% probability displacement ellipsoids. Atoms of the minor disorder components are omitted for clarity.

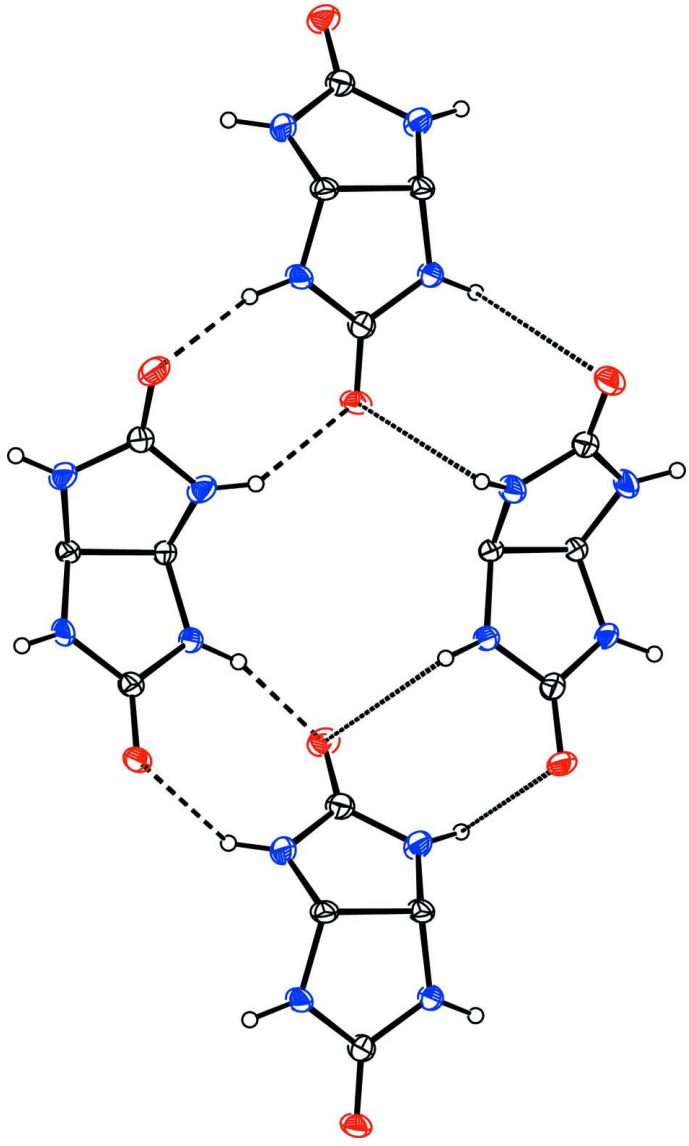

Figure 2

The partial hydrogen bonding network in the crystal structure of the title compound. H-bond drawn as dashed lines. 3*α*,6*α*- Diethoxylcarbonyl are omitted for simplicity.

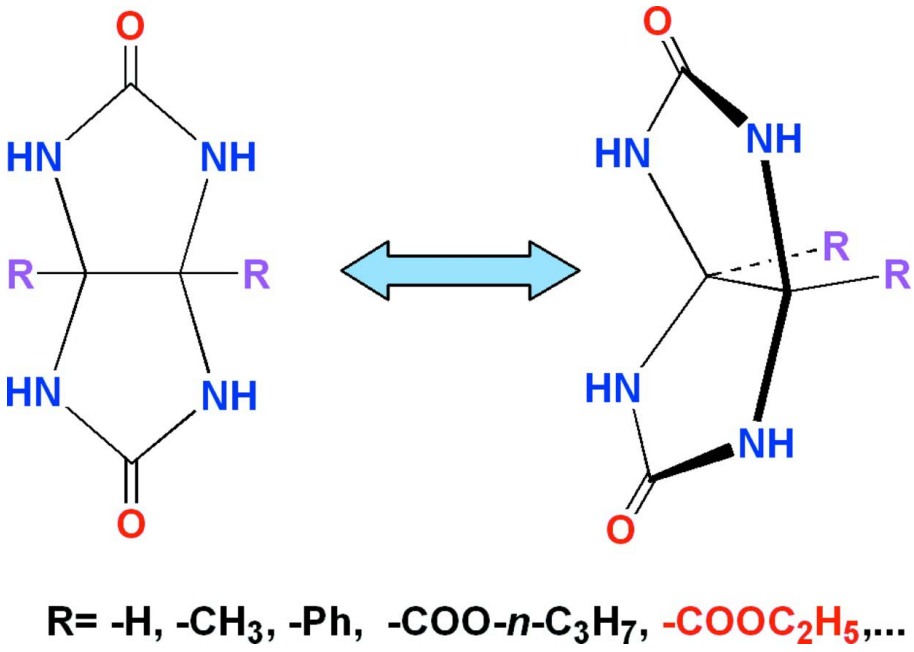

Figure 3

The glycoluril skeleton moiety.

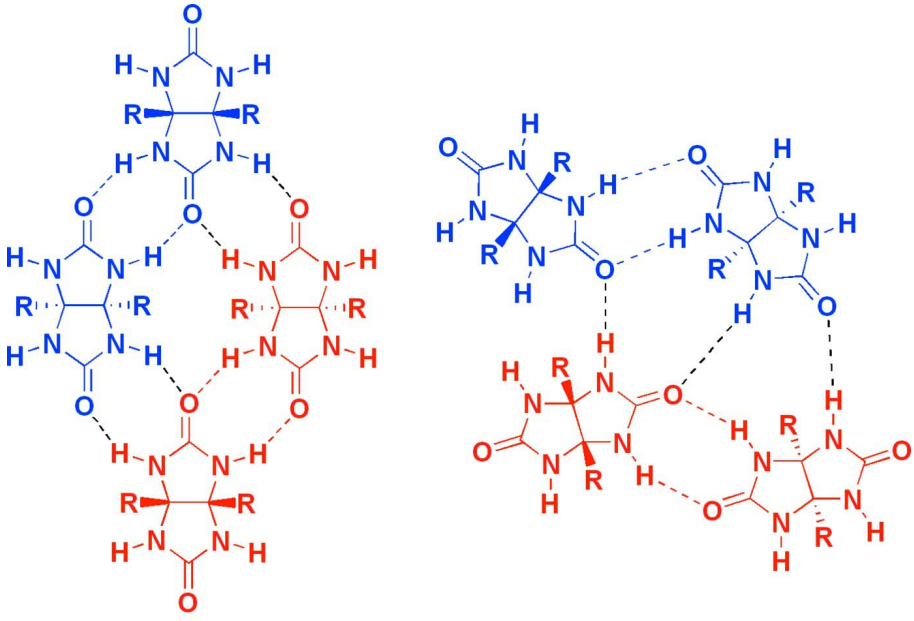

Figure 4

Hydrogen-bonding modes A and B.

diethyl 2,5-dioxoperhydroimidazo[4,5-*d*]imidazole-3a,6a-dicarboxylate

Crystal data

$C_{10}H_{14}N_4O_6$
$M_r = 286.25$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 15.7555\ (13)$ Å
$b = 11.2726\ (9)$ Å
$c = 28.742\ (2)$ Å
$V = 5104.7\ (7)$ Å^3
$Z = 16$
$F(000) = 2400$
$D_x = 1.490$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2687 reflections
$\theta = 2.6–23.5°$
$\mu = 0.12$ mm^{-1}
$T = 292$ K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
29774 measured reflections
4501 independent reflections
2784 reflections with $I > 2\sigma(I)$
$R_{int} = 0.097$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.9°$
$h = -18\rightarrow18$
$k = -11\rightarrow13$
$l = -34\rightarrow29$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.197$
$S = 1.00$
4501 reflections
385 parameters
6 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}

Special details

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 $F^2 > \sigma(F^2)$ 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}	Occ. (<1)
C1	0.04208 (19)	0.0660 (3)	0.18565 (10)	0.0315 (8)	
C2	−0.0170 (2)	0.4006 (3)	0.18911 (10)	0.0340 (8)	
C3	0.0813 (2)	0.2558 (2)	0.21072 (10)	0.0304 (7)	
C4	0.1717 (2)	0.2877 (3)	0.22674 (12)	0.0458 (9)	
C5	0.3149 (3)	0.2426 (8)	0.2249 (3)	0.048 (2)	0.683 (15)
H5A	0.3278	0.2316	0.2576	0.058*	0.683 (15)

H5B	0.3233	0.3254	0.2170	0.058*	0.683 (15)
C6	0.3700 (4)	0.1644 (8)	0.1953 (4)	0.063 (3)	0.683 (15)
H6A	0.3601	0.0828	0.2033	0.095*	0.683 (15)
H6B	0.4286	0.1835	0.2007	0.095*	0.683 (15)
H6C	0.3566	0.1767	0.1631	0.095*	0.683 (15)
C5′	0.3127 (6)	0.169 (2)	0.2260 (7)	0.082 (7)	0.317 (15)
H5′1	0.3173	0.0833	0.2257	0.098*	0.317 (15)
H5′2	0.3278	0.1970	0.2569	0.098*	0.317 (15)
C6′	0.3725 (8)	0.223 (2)	0.1899 (9)	0.126 (12)	0.317 (15)
H6′1	0.3641	0.1839	0.1605	0.190*	0.317 (15)
H6′2	0.4302	0.2122	0.1997	0.190*	0.317 (15)
H6′3	0.3607	0.3059	0.1866	0.190*	0.317 (15)
C7	0.0685 (2)	0.2544 (2)	0.15577 (10)	0.0314 (8)	
C8	0.1431 (2)	0.2921 (3)	0.12516 (13)	0.0504 (10)	
C9	0.2477 (3)	0.4440 (4)	0.11294 (16)	0.0836 (15)	
H9A	0.2828	0.4932	0.1329	0.100*	
H9B	0.2830	0.3815	0.1001	0.100*	
C10	0.2119 (3)	0.5147 (4)	0.07577 (17)	0.0965 (17)	
H10A	0.1835	0.4638	0.0540	0.145*	
H10B	0.2565	0.5569	0.0601	0.145*	
H10C	0.1721	0.5704	0.0885	0.145*	
C11	0.4936 (2)	0.4078 (3)	0.06138 (11)	0.0347 (8)	
C12	0.52703 (19)	0.0688 (3)	0.06489 (10)	0.0301 (7)	
C13	0.56575 (19)	0.2511 (2)	0.09612 (10)	0.0296 (7)	
C14	0.6371 (2)	0.2861 (3)	0.12889 (12)	0.0420 (9)	
C15	0.7460 (3)	0.4299 (4)	0.14264 (16)	0.0771 (14)	
H15A	0.7656	0.3726	0.1654	0.093*	
H15B	0.7944	0.4544	0.1241	0.093*	
C16	0.7102 (3)	0.5323 (4)	0.16631 (17)	0.0867 (16)	
H16A	0.6913	0.5892	0.1437	0.130*	
H16B	0.7526	0.5678	0.1858	0.130*	
H16C	0.6629	0.5076	0.1850	0.130*	
C17	0.5822 (2)	0.2525 (2)	0.04197 (10)	0.0296 (7)	
C18	0.6762 (2)	0.2567 (3)	0.02697 (11)	0.0391 (8)	
C19	0.8066 (2)	0.1494 (4)	0.02685 (19)	0.0847 (16)	
H19A	0.8121	0.1722	−0.0056	0.102*	
H19B	0.8237	0.0671	0.0296	0.102*	
C20	0.8641 (3)	0.2246 (4)	0.05570 (17)	0.0871 (16)	
H20A	0.8512	0.3068	0.0505	0.131*	
H20B	0.9220	0.2094	0.0471	0.131*	
H20C	0.8561	0.2060	0.0880	0.131*	
N1	0.05846 (16)	0.1358 (2)	0.22301 (8)	0.0332 (7)	
H1	0.0555	0.1110	0.2513	0.040*	
N2	0.04634 (17)	0.1327 (2)	0.14663 (8)	0.0367 (7)	
H2	0.0368	0.1053	0.1192	0.044*	
N3	0.02328 (17)	0.3466 (2)	0.22512 (9)	0.0389 (7)	
H3	0.0148	0.3650	0.2538	0.047*	
N4	0.00082 (17)	0.3390 (2)	0.14951 (9)	0.0377 (7)	

H4	−0.0254	0.3495	0.1236	0.045*
N5	0.49893 (16)	0.3389 (2)	0.09994 (9)	0.0361 (7)
H5	0.4665	0.3463	0.1238	0.043*
N6	0.53960 (17)	0.3573 (2)	0.02693 (9)	0.0375 (7)
H6	0.5428	0.3853	−0.0009	0.045*
N7	0.54036 (16)	0.1301 (2)	0.10487 (8)	0.0341 (7)
H7	0.5344	0.1003	0.1322	0.041*
N8	0.54353 (16)	0.1409 (2)	0.02829 (8)	0.0334 (7)
H8	0.5323	0.1225	−0.0001	0.040*
O1	0.02606 (15)	−0.04068 (18)	0.18682 (7)	0.0417 (6)
O2	−0.06149 (16)	0.48901 (19)	0.19152 (7)	0.0439 (6)
O3	0.18984 (19)	0.3785 (3)	0.24553 (10)	0.0773 (9)
O4	0.22562 (15)	0.2053 (3)	0.21459 (10)	0.0647 (8)
O5	0.17702 (17)	0.3914 (2)	0.14036 (9)	0.0619 (8)
O6	0.1614 (3)	0.2417 (2)	0.09063 (13)	0.1284 (18)
O7	0.45447 (16)	0.50226 (19)	0.05859 (8)	0.0480 (7)
O8	0.50442 (15)	−0.03524 (18)	0.06237 (7)	0.0413 (6)
O9	0.6426 (2)	0.2439 (2)	0.16701 (10)	0.0796 (10)
O10	0.68161 (17)	0.3742 (2)	0.11215 (9)	0.0650 (8)
O11	0.70598 (17)	0.3366 (2)	0.00593 (10)	0.0706 (9)
O12	0.71686 (14)	0.1612 (2)	0.04116 (10)	0.0616 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0399 (18)	0.0296 (18)	0.0250 (18)	−0.0046 (15)	0.0030 (14)	0.0008 (14)
C2	0.048 (2)	0.0267 (18)	0.0273 (18)	−0.0013 (15)	0.0036 (15)	−0.0002 (14)
C3	0.0477 (19)	0.0223 (16)	0.0212 (17)	−0.0032 (14)	0.0050 (14)	−0.0020 (13)
C4	0.054 (2)	0.049 (2)	0.034 (2)	−0.011 (2)	−0.0007 (17)	0.0040 (18)
C5	0.036 (3)	0.051 (5)	0.056 (4)	−0.007 (3)	−0.010 (3)	−0.007 (4)
C6	0.032 (4)	0.059 (6)	0.099 (6)	−0.007 (3)	−0.006 (4)	−0.025 (5)
C5′	0.098 (15)	0.050 (12)	0.098 (14)	−0.022 (11)	−0.021 (11)	0.007 (11)
C6′	0.16 (2)	0.080 (18)	0.14 (2)	−0.066 (16)	0.005 (16)	−0.046 (18)
C7	0.0505 (19)	0.0201 (16)	0.0237 (18)	−0.0023 (14)	0.0048 (14)	0.0012 (13)
C8	0.080 (3)	0.0243 (18)	0.047 (2)	−0.0075 (18)	0.028 (2)	0.0016 (17)
C9	0.093 (3)	0.084 (3)	0.074 (3)	−0.043 (3)	0.018 (3)	0.020 (3)
C10	0.116 (4)	0.104 (4)	0.070 (3)	−0.037 (3)	0.014 (3)	0.021 (3)
C11	0.051 (2)	0.0272 (18)	0.0262 (18)	0.0004 (16)	−0.0031 (15)	−0.0002 (14)
C12	0.0422 (18)	0.0244 (17)	0.0236 (17)	0.0001 (14)	0.0001 (14)	−0.0021 (13)
C13	0.0467 (19)	0.0227 (16)	0.0193 (16)	0.0016 (14)	−0.0029 (14)	0.0000 (13)
C14	0.068 (2)	0.0253 (17)	0.033 (2)	0.0008 (17)	−0.0117 (18)	−0.0028 (16)
C15	0.075 (3)	0.091 (3)	0.065 (3)	−0.045 (3)	−0.010 (2)	−0.018 (3)
C16	0.126 (5)	0.066 (3)	0.068 (3)	−0.024 (3)	−0.025 (3)	0.002 (3)
C17	0.0462 (18)	0.0217 (16)	0.0209 (17)	−0.0002 (14)	−0.0021 (14)	−0.0014 (12)
C18	0.051 (2)	0.036 (2)	0.030 (2)	−0.0069 (17)	0.0047 (16)	−0.0019 (16)
C19	0.055 (3)	0.075 (3)	0.124 (4)	0.008 (2)	0.019 (3)	−0.028 (3)
C20	0.054 (3)	0.119 (4)	0.088 (4)	−0.010 (3)	0.007 (2)	−0.002 (3)
N1	0.0568 (17)	0.0260 (14)	0.0167 (13)	−0.0072 (12)	0.0020 (12)	0.0029 (11)

N2	0.0675 (19)	0.0236 (14)	0.0190 (14)	−0.0060 (13)	0.0017 (13)	−0.0022 (11)
N3	0.0638 (18)	0.0330 (16)	0.0200 (15)	0.0150 (14)	0.0035 (13)	−0.0003 (12)
N4	0.0609 (18)	0.0311 (15)	0.0210 (14)	0.0071 (13)	−0.0028 (13)	−0.0015 (12)
N5	0.0539 (17)	0.0299 (15)	0.0244 (15)	0.0095 (13)	0.0050 (12)	0.0032 (12)
N6	0.0602 (18)	0.0292 (15)	0.0231 (15)	0.0072 (13)	0.0018 (13)	0.0023 (12)
N7	0.0615 (18)	0.0248 (14)	0.0160 (13)	−0.0035 (13)	−0.0016 (12)	0.0017 (11)
N8	0.0534 (17)	0.0297 (14)	0.0170 (13)	−0.0078 (12)	−0.0035 (12)	−0.0005 (11)
O1	0.0726 (17)	0.0228 (12)	0.0297 (13)	−0.0122 (11)	0.0029 (11)	0.0000 (10)
O2	0.0629 (16)	0.0333 (13)	0.0355 (14)	0.0145 (12)	0.0033 (11)	−0.0008 (10)
O3	0.098 (2)	0.0696 (19)	0.065 (2)	−0.0339 (17)	−0.0148 (17)	−0.0141 (17)
O4	0.0439 (15)	0.079 (2)	0.071 (2)	0.0037 (15)	−0.0027 (14)	−0.0057 (17)
O5	0.0844 (19)	0.0537 (16)	0.0475 (16)	−0.0306 (14)	0.0233 (14)	0.0021 (13)
O6	0.202 (4)	0.067 (2)	0.117 (3)	−0.056 (2)	0.124 (3)	−0.054 (2)
O7	0.0780 (18)	0.0339 (14)	0.0320 (13)	0.0195 (13)	−0.0011 (12)	0.0031 (11)
O8	0.0710 (16)	0.0255 (13)	0.0273 (12)	−0.0093 (11)	−0.0039 (11)	−0.0007 (10)
O9	0.133 (3)	0.0509 (17)	0.0548 (19)	−0.0262 (17)	−0.0532 (19)	0.0164 (14)
O10	0.0821 (19)	0.0751 (18)	0.0378 (15)	−0.0376 (16)	−0.0125 (14)	0.0008 (14)
O11	0.0692 (19)	0.083 (2)	0.0593 (19)	−0.0114 (16)	0.0114 (15)	0.0253 (16)
O12	0.0470 (16)	0.0454 (15)	0.092 (2)	0.0059 (12)	0.0072 (14)	−0.0009 (15)

Geometric parameters (Å, º)

C1—O1	1.229 (4)	C11—N6	1.352 (4)
C1—N2	1.352 (4)	C11—N5	1.357 (4)
C1—N1	1.356 (4)	C12—O8	1.228 (3)
C2—O2	1.220 (4)	C12—N8	1.355 (4)
C2—N3	1.358 (4)	C12—N7	1.357 (4)
C2—N4	1.362 (4)	C13—N7	1.444 (3)
C3—N3	1.433 (4)	C13—N5	1.449 (4)
C3—N1	1.443 (3)	C13—C14	1.519 (4)
C3—C4	1.541 (5)	C13—C17	1.578 (4)
C3—C7	1.592 (4)	C14—O9	1.198 (4)
C4—O3	1.191 (4)	C14—O10	1.308 (4)
C4—O4	1.307 (4)	C15—C16	1.455 (6)
C5—O4	1.497 (5)	C15—O10	1.481 (4)
C5—C6	1.502 (7)	C15—H15A	0.9700
C5—H5A	0.9700	C15—H15B	0.9700
C5—H5B	0.9700	C16—H16A	0.9600
C6—H6A	0.9600	C16—H16B	0.9600
C6—H6B	0.9600	C16—H16C	0.9600
C6—H6C	0.9600	C17—N6	1.426 (4)
C5′—O4	1.469 (9)	C17—N8	1.452 (3)
C5′—C6′	1.527 (10)	C17—C18	1.543 (5)
C5′—H5′1	0.9700	C18—O11	1.182 (4)
C5′—H5′2	0.9700	C18—O12	1.317 (4)
C6′—H6′1	0.9600	C19—O12	1.478 (4)
C6′—H6′2	0.9600	C19—C20	1.493 (6)
C6′—H6′3	0.9600	C19—H19A	0.9700

C7—N2	1.440 (3)	C19—H19B	0.9700
C7—N4	1.442 (4)	C20—H20A	0.9600
C7—C8	1.529 (4)	C20—H20B	0.9600
C8—O6	1.179 (4)	C20—H20C	0.9600
C8—O5	1.314 (4)	N1—H1	0.8600
C9—C10	1.448 (6)	N2—H2	0.8600
C9—O5	1.488 (4)	N3—H3	0.8600
C9—H9A	0.9700	N4—H4	0.8600
C9—H9B	0.9700	N5—H5	0.8600
C10—H10A	0.9600	N6—H6	0.8600
C10—H10B	0.9600	N7—H7	0.8600
C10—H10C	0.9600	N8—H8	0.8600
C11—O7	1.233 (4)		

O1—C1—N2	125.2 (3)	O9—C14—O10	126.8 (3)
O1—C1—N1	125.8 (3)	O9—C14—C13	121.2 (3)
N2—C1—N1	108.9 (3)	O10—C14—C13	111.5 (3)
O2—C2—N3	126.2 (3)	C16—C15—O10	110.3 (4)
O2—C2—N4	125.6 (3)	C16—C15—H15A	109.6
N3—C2—N4	108.2 (3)	O10—C15—H15A	109.6
N3—C3—N1	116.1 (3)	C16—C15—H15B	109.6
N3—C3—C4	109.7 (3)	O10—C15—H15B	109.6
N1—C3—C4	112.1 (3)	H15A—C15—H15B	108.1
N3—C3—C7	102.3 (2)	C15—C16—H16A	109.5
N1—C3—C7	101.7 (2)	C15—C16—H16B	109.5
C4—C3—C7	114.6 (2)	H16A—C16—H16B	109.5
O3—C4—O4	125.2 (4)	C15—C16—H16C	109.5
O3—C4—C3	123.9 (3)	H16A—C16—H16C	109.5
O4—C4—C3	110.8 (3)	H16B—C16—H16C	109.5
O4—C5—C6	105.5 (5)	N6—C17—N8	116.0 (2)
O4—C5—H5A	110.6	N6—C17—C18	110.0 (3)
C6—C5—H5A	110.6	N8—C17—C18	110.7 (2)
O4—C5—H5B	110.6	N6—C17—C13	103.3 (2)
C6—C5—H5B	110.6	N8—C17—C13	100.9 (2)
H5A—C5—H5B	108.8	C18—C17—C13	115.7 (2)
O4—C5′—C6′	108.3 (9)	O11—C18—O12	126.0 (3)
O4—C5′—H5′1	110.0	O11—C18—C17	123.2 (3)
C6′—C5′—H5′1	110.0	O12—C18—C17	110.9 (3)
O4—C5′—H5′2	110.0	O12—C19—C20	112.0 (4)
C6′—C5′—H5′2	110.0	O12—C19—H19A	109.2
H5′1—C5′—H5′2	108.4	C20—C19—H19A	109.2
C5′—C6′—H6′1	109.5	O12—C19—H19B	109.2
C5′—C6′—H6′2	109.5	C20—C19—H19B	109.2
H6′1—C6′—H6′2	109.5	H19A—C19—H19B	107.9
C5′—C6′—H6′3	109.5	C19—C20—H20A	109.5
H6′1—C6′—H6′3	109.5	C19—C20—H20B	109.5
H6′2—C6′—H6′3	109.5	H20A—C20—H20B	109.5
N2—C7—N4	115.3 (3)	C19—C20—H20C	109.5

N2—C7—C8	110.3 (3)	H20A—C20—H20C	109.5
N4—C7—C8	108.2 (2)	H20B—C20—H20C	109.5
N2—C7—C3	102.8 (2)	C1—N1—C3	113.4 (2)
N4—C7—C3	102.2 (2)	C1—N1—H1	123.3
C8—C7—C3	118.1 (3)	C3—N1—H1	123.3
O6—C8—O5	126.2 (3)	C1—N2—C7	113.0 (2)
O6—C8—C7	122.6 (3)	C1—N2—H2	123.5
O5—C8—C7	111.0 (3)	C7—N2—H2	123.5
C10—C9—O5	108.6 (4)	C2—N3—C3	113.5 (2)
C10—C9—H9A	110.0	C2—N3—H3	123.3
O5—C9—H9A	110.0	C3—N3—H3	123.3
C10—C9—H9B	110.0	C2—N4—C7	112.7 (3)
O5—C9—H9B	110.0	C2—N4—H4	123.7
H9A—C9—H9B	108.4	C7—N4—H4	123.7
C9—C10—H10A	109.5	C11—N5—C13	112.0 (2)
C9—C10—H10B	109.5	C11—N5—H5	124.0
H10A—C10—H10B	109.5	C13—N5—H5	124.0
C9—C10—H10C	109.5	C11—N6—C17	112.3 (2)
H10A—C10—H10C	109.5	C11—N6—H6	123.9
H10B—C10—H10C	109.5	C17—N6—H6	123.9
O7—C11—N6	125.7 (3)	C12—N7—C13	112.1 (2)
O7—C11—N5	125.4 (3)	C12—N7—H7	124.0
N6—C11—N5	108.9 (3)	C13—N7—H7	124.0
O8—C12—N8	125.7 (3)	C12—N8—C17	113.0 (2)
O8—C12—N7	125.5 (3)	C12—N8—H8	123.5
N8—C12—N7	108.8 (2)	C17—N8—H8	123.5
N7—C13—N5	115.5 (3)	C4—O4—C5′	138.1 (10)
N7—C13—C14	110.0 (2)	C4—O4—C5	111.0 (4)
N5—C13—C14	108.2 (2)	C5′—O4—C5	32.5 (7)
N7—C13—C17	103.1 (2)	C8—O5—C9	117.9 (3)
N5—C13—C17	100.8 (2)	C14—O10—C15	118.2 (3)
C14—C13—C17	119.2 (3)	C18—O12—C19	116.9 (3)

N3—C3—C4—O3	−4.5 (5)	C8—C7—N2—C1	128.3 (3)
N1—C3—C4—O3	−134.9 (3)	C3—C7—N2—C1	1.5 (3)
C7—C3—C4—O3	109.9 (4)	O2—C2—N3—C3	−170.9 (3)
N3—C3—C4—O4	179.0 (3)	N4—C2—N3—C3	9.1 (4)
N1—C3—C4—O4	48.6 (4)	N1—C3—N3—C2	−112.5 (3)
C7—C3—C4—O4	−66.6 (4)	C4—C3—N3—C2	119.2 (3)
N3—C3—C7—N2	−123.9 (2)	C7—C3—N3—C2	−2.8 (3)
N1—C3—C7—N2	−3.6 (3)	O2—C2—N4—C7	167.8 (3)
C4—C3—C7—N2	117.5 (3)	N3—C2—N4—C7	−12.1 (4)
N3—C3—C7—N4	−4.0 (3)	N2—C7—N4—C2	120.5 (3)
N1—C3—C7—N4	116.3 (2)	C8—C7—N4—C2	−115.5 (3)
C4—C3—C7—N4	−122.6 (3)	C3—C7—N4—C2	9.8 (3)
N3—C3—C7—C8	114.5 (3)	O7—C11—N5—C13	−165.3 (3)
N1—C3—C7—C8	−125.2 (3)	N6—C11—N5—C13	13.4 (4)
C4—C3—C7—C8	−4.1 (4)	N7—C13—N5—C11	−127.2 (3)

N2—C7—C8—O6	20.6 (5)	C14—C13—N5—C11	109.0 (3)
N4—C7—C8—O6	−106.4 (5)	C17—C13—N5—C11	−16.9 (3)
C3—C7—C8—O6	138.3 (4)	O7—C11—N6—C17	175.8 (3)
N2—C7—C8—O5	−165.0 (3)	N5—C11—N6—C17	−2.9 (4)
N4—C7—C8—O5	68.0 (4)	N8—C17—N6—C11	101.9 (3)
C3—C7—C8—O5	−47.4 (4)	C18—C17—N6—C11	−131.5 (3)
N7—C13—C14—O9	−30.6 (4)	C13—C17—N6—C11	−7.4 (3)
N5—C13—C14—O9	96.5 (4)	O8—C12—N7—C13	−179.4 (3)
C17—C13—C14—O9	−149.3 (3)	N8—C12—N7—C13	1.2 (3)
N7—C13—C14—O10	157.0 (3)	N5—C13—N7—C12	99.5 (3)
N5—C13—C14—O10	−75.9 (3)	C14—C13—N7—C12	−137.6 (3)
C17—C13—C14—O10	38.3 (4)	C17—C13—N7—C12	−9.5 (3)
N7—C13—C17—N6	133.5 (2)	O8—C12—N8—C17	−170.5 (3)
N5—C13—C17—N6	13.9 (3)	N7—C12—N8—C17	8.9 (4)
C14—C13—C17—N6	−104.2 (3)	N6—C17—N8—C12	−124.7 (3)
N7—C13—C17—N8	13.3 (3)	C18—C17—N8—C12	109.1 (3)
N5—C13—C17—N8	−106.4 (2)	C13—C17—N8—C12	−13.9 (3)
C14—C13—C17—N8	135.5 (3)	O3—C4—O4—C5′	18.8 (11)
N7—C13—C17—C18	−106.2 (3)	C3—C4—O4—C5′	−164.8 (9)
N5—C13—C17—C18	134.2 (3)	O3—C4—O4—C5	−3.3 (6)
C14—C13—C17—C18	16.0 (4)	C3—C4—O4—C5	173.1 (4)
N6—C17—C18—O11	−0.3 (4)	C6′—C5′—O4—C4	−96 (2)
N8—C17—C18—O11	129.2 (3)	C6′—C5′—O4—C5	−55.2 (16)
C13—C17—C18—O11	−116.8 (4)	C6—C5—O4—C4	−160.8 (7)
N6—C17—C18—O12	179.1 (3)	C6—C5—O4—C5′	47.1 (14)
N8—C17—C18—O12	−51.4 (3)	O6—C8—O5—C9	−2.7 (7)
C13—C17—C18—O12	62.6 (3)	C7—C8—O5—C9	−176.8 (3)
O1—C1—N1—C3	175.7 (3)	C10—C9—O5—C8	84.3 (5)
N2—C1—N1—C3	−4.2 (4)	O9—C14—O10—C15	−0.1 (6)
N3—C3—N1—C1	114.9 (3)	C13—C14—O10—C15	171.7 (3)
C4—C3—N1—C1	−118.0 (3)	C16—C15—O10—C14	−94.1 (4)
C7—C3—N1—C1	4.8 (3)	O11—C18—O12—C19	−4.2 (5)
O1—C1—N2—C7	−178.5 (3)	C17—C18—O12—C19	176.4 (3)
N1—C1—N2—C7	1.4 (4)	C20—C19—O12—C18	78.4 (5)
N4—C7—N2—C1	−108.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.86	2.15	2.963 (3)	158
N2—H2···O7^{ii}	0.86	2.10	2.927 (3)	162
N3—H3···O1^{iii}	0.86	2.11	2.937 (3)	161
N4—H4···O8^{iv}	0.86	2.21	2.879 (3)	134
N5—H5···O1^{iv}	0.86	2.22	2.870 (3)	133
N6—H6···O7^{v}	0.86	2.09	2.925 (3)	164
N7—H7···O2^{ii}	0.86	2.16	2.973 (3)	158
N8—H8···O8^{vi}	0.86	2.12	2.963 (3)	165

C16—H16*C*···O3^{vii}	0.96	2.51	3.087 (5)	119
C10—H10*B*···O11^{v}	0.96	2.32	3.162 (5)	146

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