Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

$N^2,N^{2'}$-Bis[2-(ethoxycarbonylmethoxy)-benzylidene]pyridine-2,6-dicarbohydrazide

Feihua Luo, Shemin Lan, Cuixia Cheng and Zongqiu Hu*

Department of Chemistry, Central China Normal University, Wuhan, Hubei 430079, People's Republic of China
Correspondence e-mail: luofh2005@yahoo.com.cn

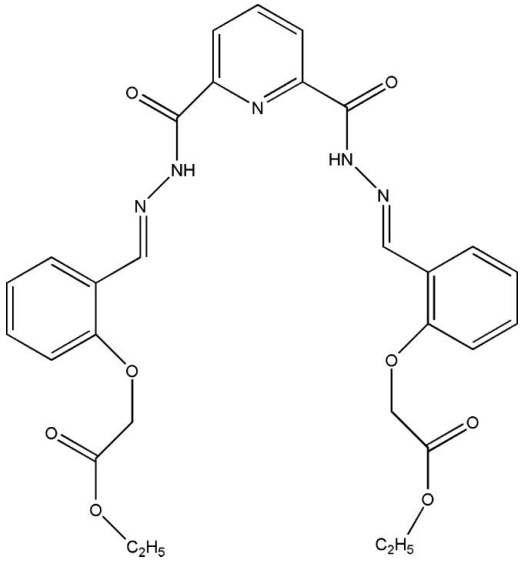
Received 13 November 2007; accepted 30 November 2007

Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.076; wR factor = 0.231; data-to-parameter ratio = 15.4.

In the title compound, $C_{29}H_{29}N_5O_8$, the ester group is disordered over two sites with site-occupancy factors of 0.91/0.09. The crystal structure is stabilized by inter- and intramolecular hydrogen-bond interactions.

Related literature

For related literature, see: Chen *et al.* (1997); Thompson (2002); Zhao *et al.* (2004).



Experimental

Crystal data

$C_{29}H_{29}N_5O_8$
M_r = 575.57
Monoclinic, $P2_1/n$
a = 11.5485 (6) Å
b = 21.6606 (11) Å
c = 12.3596 (9) Å
β = 114.779 (1)°
V = 2807.1 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 299 (2) K
0.32 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
23685 measured reflections
6101 independent reflections
4136 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.076
$wR(F^2)$ = 0.231
S = 1.04
6101 reflections
396 parameters
12 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O5^{i}	0.86	2.53	3.353 (3)	161
C2—H2···O7^{ii}	0.93	2.40	3.304 (4)	165
C17—H17C···O2^{iii}	0.96	2.49	2.896 (8)	105
N2—H2A···N1	0.86	2.34	2.694 (3)	105

Symmetry codes: (i) $-x+2, -y, -z+2$; (ii) $-x+1, -y, -z+2$; (iii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

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

This work was supported by Hubei Education Department, Government of China (grant No. 20040131).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2047).

References

Bruker (2001). *SAINT-Plus* (Version 6.45) and *SMART* (Version 5.628). Bruker AXS Inc., Madison, Wisconsin, USA.
Chen, X., Zhan, S., Hu, C., Meng, Q. & Liu, Y. (1997). *J. Chem. Soc. Dalton Trans.* pp. 245–250.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2001). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Thompson, L. K. (2002). *Coord. Chem. Rev.* **233–234**, 193–206.
Zhao, L., Xu, Z., Grove, H. & Milway, V. A. (2004). *Inorg. Chem.* **43**, 3812–3824.

supporting information

Acta Cryst. (2008). E**64**, o185 [https://doi.org/10.1107/S160053680706477X]

N^2,$N^{2'}$-Bis[2-(ethoxycarbonylmethoxy)benzylidene]pyridine-2,6-dicarbohydrazide

Feihua Luo, Shemin Lan, Cuixia Cheng and Zongqiu Hu

S1. Comment

The tridentate ligands with 2,6-dipicolinoyhydrazone have been intensively studied due to the interesting coordination mode. (Chen *et al.*, 1997; Thompson, 2002; Zhao *et al.*, 2004). We report here the synthesis and crystal structure of a novel tridentate ligand, the title compound (I) (Fig. 1).

The molecular structure contains one pyridine ring and two substitutional benzene rings. The dihedral angles between the pyridine and benzene planes are 6.50 (13)° for C8—C13 and 26.43 (16)° for C20—C25.

The crystal packing is governed by intermolecular hydrogen bonds interactions. Each molecular can serve as donor and acceptor to form the N–H–O hydrogen bonds with two other neighboring molecules, forming chains parallel to the *a* axis (Fig. 2; Table 1).

S2. Experimental

To a solution of 2-(2-formylphenoxy)acetic acid (1.80 g, 10 mmol) in absolute ethanol (40 ml), a suspension of 2,6-dipicolinoyhydrazine in the same solvent (50 ml) was added at 323 K. The mixture was left to react at reflux for 18 h, then the white needle product was filtered, washed with hot ethanol (20 ml portion) three times and dried in vacuum. Crystals suitable for X-ray diffraction were obtained from acetone-methanol (1:1 *v*/*v*) over a period of about three weeks, and unexpecting the carboxyl from the 2-(2-formylphenoxy)acetic acid was esterified in the ethanol solvent.

S3. Refinement

After their location in the difference map, all H-atoms were fixed geometrically at ideal positions and allowed to ride on the parent C or N atoms with C—H = 0.93Å and N—H = 0.86Å and U_{iso}(H)= 1.2U_{eq}(C and N). The esterified group is disorder over two sites. So, the site-occupancy factors for the two orientations were refined as 0.905 / 0.095. The *SHELX* restrains AFIX, FLAT and ISOR were applied.

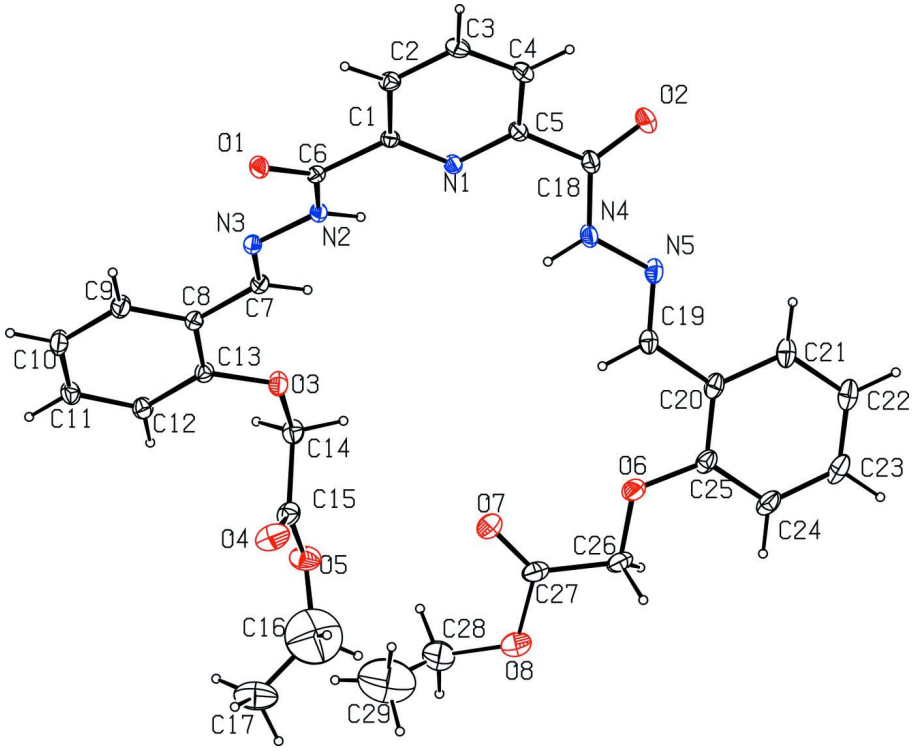


Figure 1

The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 20% probability level.

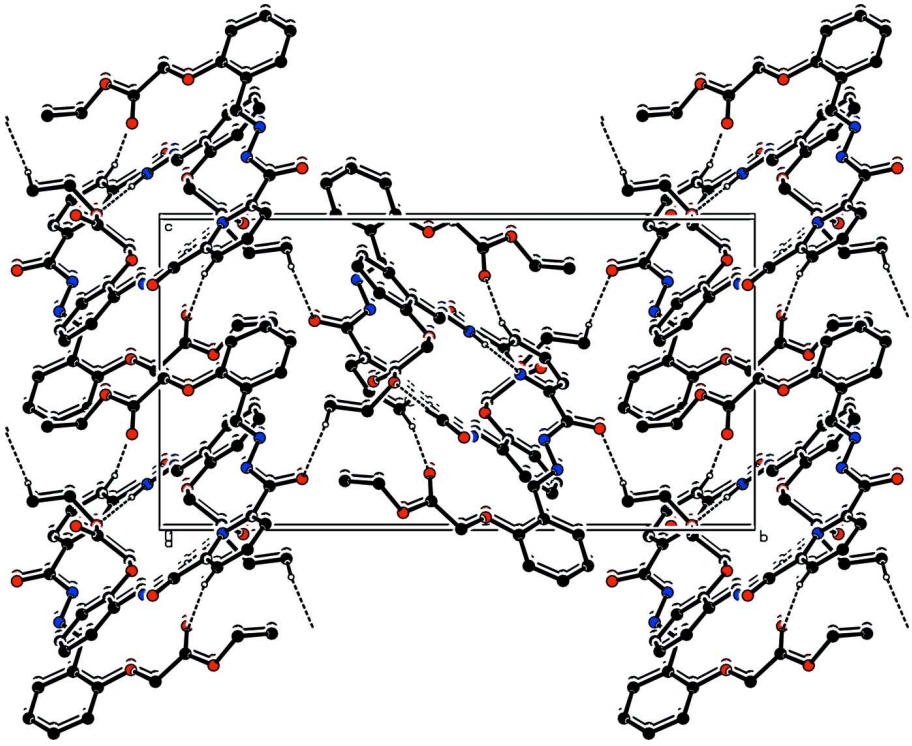


Figure 2

A view of the packing of (I). Hydrogen bonds are shown by dashed lines.

N^2,$N^{2'}$—Bis[2-(ethoxycarbonylmethoxy)benzylidene]pyridine- 2,6-dicarbohydrazide

Crystal data

$C_{29}H_{29}N_5O_8$
M_r = 575.57
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 11.5485 (6) Å
b = 21.6606 (11) Å
c = 12.3596 (9) Å
β = 114.779 (1)°
V = 2807.1 (3) Å^3
Z = 4
F(000) = 1208
D_x = 1.362 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
μ = 0.10 mm^{-1}
T = 299 K
Block, colorless
0.32 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
23685 measured reflections
6101 independent reflections
4136 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 27.0°, θ_{min} = 1.9°
h = −14→14
k = −27→27
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.076$
$wR(F^2) = 0.231$
$S = 1.04$
6101 reflections
396 parameters
12 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
$w = 1/[\sigma^2(F_o^2) + (0.1307P)^2 + 0.5522P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.52$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.4448 (2)	−0.07839 (10)	1.0814 (2)	0.0440 (5)	
C2	0.3444 (3)	−0.09691 (12)	1.1079 (2)	0.0539 (6)	
H2	0.3247	−0.0755	1.1632	0.065*	
C3	0.2749 (3)	−0.14779 (13)	1.0499 (3)	0.0633 (7)	
H3	0.2074	−0.1617	1.0658	0.076*	
C4	0.3065 (3)	−0.17779 (12)	0.9680 (3)	0.0605 (7)	
H4	0.2622	−0.2129	0.9291	0.073*	
C5	0.4054 (3)	−0.15491 (11)	0.9445 (2)	0.0506 (6)	
C6	0.5243 (2)	−0.02429 (11)	1.1467 (2)	0.0453 (5)	
C7	0.8231 (2)	0.02966 (12)	1.1877 (2)	0.0497 (6)	
H7	0.8370	0.0019	1.1370	0.060*	
C8	0.9182 (3)	0.07674 (11)	1.2480 (2)	0.0496 (6)	
C9	0.9095 (3)	0.11453 (14)	1.3352 (3)	0.0642 (7)	
H9	0.8419	0.1094	1.3566	0.077*	
C10	0.9998 (3)	0.15966 (15)	1.3905 (3)	0.0721 (8)	
H10	0.9925	0.1848	1.4484	0.087*	
C11	1.1004 (3)	0.16721 (14)	1.3596 (2)	0.0641 (7)	
H11	1.1616	0.1973	1.3974	0.077*	
C12	1.1111 (3)	0.13062 (13)	1.2734 (2)	0.0577 (7)	
H12	1.1789	0.1363	1.2524	0.069*	
C13	1.0217 (2)	0.08554 (12)	1.2180 (2)	0.0499 (6)	
C14	1.1296 (3)	0.05037 (14)	1.1008 (3)	0.0640 (7)	
H14A	1.1350	0.0124	1.0615	0.077*	
H14B	1.2078	0.0546	1.1727	0.077*	
C15	1.1167 (3)	0.10434 (17)	1.0195 (3)	0.0712 (8)	

C16	1.1906 (13)	0.1507 (5)	0.8951 (12)	0.351 (9)	
H16A	1.0988	0.1557	0.8546	0.421*	
H16B	1.2167	0.1310	0.8384	0.421*	
C17	1.2435 (8)	0.2131 (3)	0.9119 (7)	0.170 (3)	
H17A	1.3109	0.2167	0.9905	0.255*	
H17B	1.1777	0.2425	0.9025	0.255*	
H17C	1.2766	0.2211	0.8538	0.255*	
C18	0.4322 (3)	−0.18466 (12)	0.8482 (3)	0.0622 (7)	
C19	0.5350 (3)	−0.12858 (14)	0.6412 (3)	0.0598 (7)	
H19	0.5493	−0.0881	0.6690	0.072*	
C20	0.5563 (3)	−0.14547 (14)	0.5363 (2)	0.0620 (7)	
C21	0.5237 (3)	−0.20391 (16)	0.4848 (3)	0.0759 (9)	
H21	0.4880	−0.2327	0.5177	0.091*	
C22	0.5437 (4)	−0.21969 (19)	0.3858 (3)	0.0829 (10)	
H22	0.5205	−0.2586	0.3517	0.100*	
C23	0.5977 (4)	−0.17794 (19)	0.3382 (3)	0.0875 (12)	
H23	0.6115	−0.1887	0.2718	0.105*	
C24	0.6317 (4)	−0.12035 (17)	0.3872 (3)	0.0819 (10)	
H24	0.6685	−0.0923	0.3540	0.098*	
C25	0.6114 (3)	−0.10395 (14)	0.4863 (2)	0.0665 (8)	
C26	0.7136 (5)	−0.00699 (17)	0.5070 (3)	0.0916 (12)	
H26A	0.6602	0.0095	0.4288	0.110*	
H26B	0.7864	−0.0274	0.5029	0.110*	
C27	0.7567 (4)	0.04350 (17)	0.5975 (3)	0.0922 (12)	
O5	1.2125 (3)	0.10572 (13)	0.9880 (3)	0.0981 (9)	
C28	0.8947 (11)	0.1314 (4)	0.6564 (9)	0.231 (8)	0.905 (13)
H28A	0.9670	0.1425	0.6399	0.278*	0.905 (13)
H28B	0.9264	0.1098	0.7320	0.278*	0.905 (13)
C29	0.8259 (13)	0.1885 (5)	0.6639 (14)	0.306 (9)	0.905 (13)
H29A	0.8077	0.2135	0.5945	0.459*	0.905 (13)
H29B	0.8784	0.2114	0.7339	0.459*	0.905 (13)
H29C	0.7476	0.1774	0.6683	0.459*	0.905 (13)
O8	0.8081 (10)	0.0907 (3)	0.5612 (5)	0.155 (3)	0.905 (13)
O8′	0.8681 (14)	0.0661 (10)	0.589 (2)	0.047 (7)*	0.095 (13)
C28′	0.896 (5)	0.1302 (12)	0.605 (10)	0.23 (7)*	0.095 (13)
H28C	0.8669	0.1504	0.6593	0.277*	0.095 (13)
H28D	0.8721	0.1532	0.5319	0.277*	0.095 (13)
C29′	1.038 (3)	0.111 (6)	0.664 (13)	0.30 (7)*	0.095 (13)
H29D	1.0792	0.1245	0.6148	0.453*	0.095 (13)
H29E	1.0438	0.0668	0.6715	0.453*	0.095 (13)
H29F	1.0786	0.1295	0.7411	0.453*	0.095 (13)
N1	0.47564 (19)	−0.10620 (9)	1.00079 (17)	0.0458 (5)	
N2	0.6383 (2)	−0.02049 (9)	1.14131 (17)	0.0475 (5)	
H2A	0.6588	−0.0466	1.0999	0.057*	
N3	0.7217 (2)	0.02594 (9)	1.20320 (17)	0.0485 (5)	
N4	0.4772 (2)	−0.14655 (10)	0.7900 (2)	0.0606 (6)	
H4A	0.4935	−0.1087	0.8119	0.073*	
N5	0.4975 (2)	−0.16823 (11)	0.6943 (2)	0.0645 (6)	

O1	0.48826 (19)	0.01162 (8)	1.20160 (18)	0.0624 (5)
O2	0.4086 (3)	−0.23957 (10)	0.8248 (2)	0.0996 (9)
O3	1.02504 (19)	0.04682 (9)	1.13173 (18)	0.0656 (5)
O4	1.0338 (3)	0.14056 (15)	0.9878 (3)	0.1093 (10)
O6	0.6451 (3)	−0.04805 (10)	0.5434 (2)	0.0817 (7)
O7	0.7445 (4)	0.04619 (14)	0.6864 (3)	0.1194 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0488 (14)	0.0403 (11)	0.0461 (12)	0.0015 (10)	0.0229 (11)	0.0034 (9)
C2	0.0552 (16)	0.0564 (14)	0.0615 (15)	0.0004 (12)	0.0357 (13)	0.0032 (12)
C3	0.0571 (17)	0.0625 (16)	0.0783 (18)	−0.0119 (13)	0.0362 (15)	0.0049 (14)
C4	0.0641 (18)	0.0484 (14)	0.0702 (17)	−0.0156 (12)	0.0294 (15)	−0.0037 (13)
C5	0.0544 (15)	0.0410 (12)	0.0551 (14)	−0.0033 (11)	0.0218 (12)	−0.0011 (10)
C6	0.0559 (15)	0.0435 (12)	0.0435 (11)	−0.0011 (11)	0.0276 (11)	0.0004 (10)
C7	0.0554 (16)	0.0532 (13)	0.0437 (12)	−0.0045 (11)	0.0241 (12)	−0.0040 (10)
C8	0.0541 (16)	0.0523 (13)	0.0404 (12)	−0.0062 (11)	0.0179 (11)	−0.0022 (10)
C9	0.0680 (19)	0.0727 (18)	0.0581 (15)	−0.0111 (15)	0.0326 (14)	−0.0141 (14)
C10	0.081 (2)	0.0769 (19)	0.0575 (16)	−0.0147 (16)	0.0282 (16)	−0.0250 (15)
C11	0.0633 (19)	0.0597 (16)	0.0570 (15)	−0.0138 (13)	0.0132 (14)	−0.0102 (13)
C12	0.0525 (16)	0.0636 (16)	0.0526 (14)	−0.0087 (13)	0.0175 (12)	−0.0044 (12)
C13	0.0470 (15)	0.0558 (14)	0.0430 (12)	−0.0035 (11)	0.0150 (11)	−0.0027 (11)
C14	0.0549 (17)	0.0726 (18)	0.0733 (18)	−0.0041 (14)	0.0354 (15)	−0.0145 (15)
C15	0.067 (2)	0.087 (2)	0.0686 (18)	−0.0041 (18)	0.0376 (17)	−0.0082 (17)
C16	0.193 (11)	0.44 (3)	0.43 (3)	−0.015 (16)	0.141 (14)	0.00 (2)
C17	0.174 (7)	0.146 (5)	0.196 (7)	0.031 (5)	0.084 (6)	0.079 (5)
C18	0.074 (2)	0.0473 (14)	0.0674 (17)	−0.0043 (13)	0.0313 (15)	−0.0132 (13)
C19	0.0563 (17)	0.0623 (16)	0.0621 (15)	−0.0010 (13)	0.0260 (14)	−0.0172 (13)
C20	0.0576 (17)	0.0735 (18)	0.0513 (14)	0.0163 (14)	0.0194 (13)	−0.0088 (13)
C21	0.068 (2)	0.086 (2)	0.0734 (19)	0.0007 (16)	0.0289 (16)	−0.0301 (17)
C22	0.079 (2)	0.094 (2)	0.0650 (19)	0.0171 (19)	0.0195 (18)	−0.0254 (18)
C23	0.107 (3)	0.106 (3)	0.0428 (15)	0.044 (2)	0.0248 (17)	−0.0030 (17)
C24	0.112 (3)	0.086 (2)	0.0483 (15)	0.035 (2)	0.0341 (17)	0.0146 (16)
C25	0.083 (2)	0.0669 (17)	0.0476 (14)	0.0270 (16)	0.0255 (14)	0.0058 (13)
C26	0.147 (4)	0.081 (2)	0.0652 (19)	0.010 (2)	0.063 (2)	0.0169 (17)
C27	0.137 (4)	0.083 (2)	0.077 (2)	0.000 (2)	0.065 (2)	0.0156 (18)
O5	0.0941 (19)	0.0998 (18)	0.129 (2)	0.0022 (15)	0.0749 (19)	0.0165 (16)
C28	0.39 (2)	0.184 (9)	0.194 (9)	−0.151 (12)	0.190 (13)	−0.057 (8)
C29	0.231 (14)	0.303 (19)	0.37 (2)	0.035 (14)	0.113 (15)	0.096 (17)
O8	0.246 (7)	0.139 (4)	0.126 (4)	−0.065 (5)	0.123 (5)	−0.012 (3)
N1	0.0486 (12)	0.0415 (10)	0.0518 (11)	−0.0030 (9)	0.0254 (10)	−0.0039 (9)
N2	0.0548 (13)	0.0465 (10)	0.0472 (10)	−0.0079 (9)	0.0272 (10)	−0.0092 (9)
N3	0.0526 (13)	0.0501 (11)	0.0452 (10)	−0.0082 (9)	0.0230 (9)	−0.0063 (9)
N4	0.0723 (16)	0.0505 (12)	0.0698 (14)	−0.0116 (11)	0.0404 (13)	−0.0221 (11)
N5	0.0727 (17)	0.0625 (14)	0.0621 (13)	−0.0029 (12)	0.0319 (13)	−0.0205 (12)
O1	0.0775 (13)	0.0528 (10)	0.0756 (12)	−0.0095 (9)	0.0504 (11)	−0.0167 (9)
O2	0.162 (3)	0.0512 (12)	0.1062 (18)	−0.0229 (14)	0.0769 (19)	−0.0250 (12)

O3	0.0621 (12)	0.0755 (12)	0.0701 (12)	−0.0208 (10)	0.0384 (10)	−0.0258 (10)
O4	0.108 (2)	0.129 (2)	0.109 (2)	0.0452 (19)	0.0635 (18)	0.0369 (18)
O6	0.126 (2)	0.0663 (13)	0.0724 (13)	0.0102 (13)	0.0610 (14)	0.0051 (11)
O7	0.193 (3)	0.106 (2)	0.0978 (19)	−0.028 (2)	0.099 (2)	−0.0171 (15)

Geometric parameters (Å, º)

C1—N1	1.334 (3)	C18—N4	1.336 (4)
C1—C2	1.389 (3)	C19—N5	1.263 (4)
C1—C6	1.498 (3)	C19—C20	1.463 (4)
C2—C3	1.375 (4)	C19—H19	0.9300
C2—H2	0.9300	C20—C25	1.388 (4)
C3—C4	1.374 (4)	C20—C21	1.396 (4)
C3—H3	0.9300	C21—C22	1.380 (4)
C4—C5	1.383 (4)	C21—H21	0.9300
C4—H4	0.9300	C22—C23	1.363 (6)
C5—N1	1.335 (3)	C22—H22	0.9300
C5—C18	1.496 (4)	C23—C24	1.370 (5)
C6—O1	1.215 (3)	C23—H23	0.9300
C6—N2	1.348 (3)	C24—C25	1.386 (4)
C7—N3	1.267 (3)	C24—H24	0.9300
C7—C8	1.455 (4)	C25—O6	1.373 (4)
C7—H7	0.9300	C26—O6	1.385 (4)
C8—C9	1.390 (4)	C26—C27	1.493 (5)
C8—C13	1.404 (4)	C26—H26A	0.9700
C9—C10	1.383 (4)	C26—H26B	0.9700
C9—H9	0.9300	C27—O7	1.167 (4)
C10—C11	1.377 (4)	C27—O8	1.349 (5)
C10—H10	0.9300	C27—O8′	1.419 (10)
C11—C12	1.375 (4)	C28—O8	1.475 (7)
C11—H11	0.9300	C28—C29	1.494 (9)
C12—C13	1.377 (4)	C28—H28A	0.9700
C12—H12	0.9300	C28—H28B	0.9700
C13—O3	1.370 (3)	C29—H29A	0.9600
C14—O3	1.413 (3)	C29—H29B	0.9600
C14—C15	1.508 (5)	C29—H29C	0.9600
C14—H14A	0.9700	O8′—C28′	1.420 (10)
C14—H14B	0.9700	C28′—C29′	1.540 (11)
C15—O4	1.171 (4)	C28′—H28C	0.9700
C15—O5	1.318 (4)	C28′—H28D	0.9700
C16—O5	1.447 (9)	C29′—H29D	0.9600
C16—C17	1.460 (9)	C29′—H29E	0.9600
C16—H16A	0.9700	C29′—H29F	0.9600
C16—H16B	0.9700	N2—N3	1.381 (3)
C17—H17A	0.9600	N2—H2A	0.8600
C17—H17B	0.9600	N4—N5	1.380 (3)
C17—H17C	0.9600	N4—H4A	0.8600
C18—O2	1.228 (3)		

N1—C1—C2	123.8 (2)	N5—C19—H19	119.6
N1—C1—C6	117.4 (2)	C20—C19—H19	119.6
C2—C1—C6	118.9 (2)	C25—C20—C21	118.1 (3)
C3—C2—C1	118.1 (2)	C25—C20—C19	120.7 (3)
C3—C2—H2	120.9	C21—C20—C19	121.2 (3)
C1—C2—H2	120.9	C22—C21—C20	121.1 (4)
C4—C3—C2	119.0 (2)	C22—C21—H21	119.5
C4—C3—H3	120.5	C20—C21—H21	119.5
C2—C3—H3	120.5	C23—C22—C21	119.6 (3)
C3—C4—C5	118.9 (2)	C23—C22—H22	120.2
C3—C4—H4	120.6	C21—C22—H22	120.2
C5—C4—H4	120.6	C22—C23—C24	120.8 (3)
N1—C5—C4	123.3 (2)	C22—C23—H23	119.6
N1—C5—C18	118.1 (2)	C24—C23—H23	119.6
C4—C5—C18	118.5 (2)	C23—C24—C25	120.0 (4)
O1—C6—N2	123.9 (2)	C23—C24—H24	120.0
O1—C6—C1	121.7 (2)	C25—C24—H24	120.0
N2—C6—C1	114.32 (19)	O6—C25—C24	124.3 (3)
N3—C7—C8	121.0 (2)	O6—C25—C20	115.2 (2)
N3—C7—H7	119.5	C24—C25—C20	120.5 (3)
C8—C7—H7	119.5	O6—C26—C27	106.5 (3)
C9—C8—C13	118.1 (2)	O6—C26—H26A	110.4
C9—C8—C7	121.7 (2)	C27—C26—H26A	110.4
C13—C8—C7	120.2 (2)	O6—C26—H26B	110.4
C10—C9—C8	121.0 (3)	C27—C26—H26B	110.4
C10—C9—H9	119.5	H26A—C26—H26B	108.6
C8—C9—H9	119.5	O7—C27—O8	121.3 (4)
C11—C10—C9	119.7 (3)	O7—C27—O8′	121.4 (10)
C11—C10—H10	120.2	O8—C27—O8′	34.6 (7)
C9—C10—H10	120.2	O7—C27—C26	127.5 (3)
C12—C11—C10	120.5 (3)	O8—C27—C26	111.2 (3)
C12—C11—H11	119.7	O8′—C27—C26	103.2 (10)
C10—C11—H11	119.7	C15—O5—C16	111.2 (6)
C11—C12—C13	120.1 (3)	O8—C28—C29	110.2 (9)
C11—C12—H12	120.0	O8—C28—H28A	109.6
C13—C12—H12	120.0	C29—C28—H28A	109.6
O3—C13—C12	124.1 (2)	O8—C28—H28B	109.6
O3—C13—C8	115.3 (2)	C29—C28—H28B	109.6
C12—C13—C8	120.6 (2)	H28A—C28—H28B	108.1
O3—C14—C15	111.4 (3)	C27—O8—C28	115.7 (5)
O3—C14—H14A	109.3	C27—O8′—C28′	119.3 (13)
C15—C14—H14A	109.3	O8′—C28′—C29′	86 (2)
O3—C14—H14B	109.3	O8′—C28′—H28C	114.2
C15—C14—H14B	109.3	C29′—C28′—H28C	114.2
H14A—C14—H14B	108.0	O8′—C28′—H28D	114.2
O4—C15—O5	123.8 (3)	C29′—C28′—H28D	114.2
O4—C15—C14	125.6 (3)	H28C—C28′—H28D	111.4

O5—C15—C14	110.7 (3)	C28′—C29′—H29D	109.5
O5—C16—C17	125.9 (10)	C28′—C29′—H29E	109.5
O5—C16—H16A	105.8	H29D—C29′—H29E	109.5
C17—C16—H16A	105.8	C28′—C29′—H29F	109.5
O5—C16—H16B	105.8	H29D—C29′—H29F	109.5
C17—C16—H16B	105.8	H29E—C29′—H29F	109.5
H16A—C16—H16B	106.2	C1—N1—C5	116.9 (2)
C16—C17—H17A	109.5	C6—N2—N3	119.08 (19)
C16—C17—H17B	109.5	C6—N2—H2A	120.5
H17A—C17—H17B	109.5	N3—N2—H2A	120.5
C16—C17—H17C	109.5	C7—N3—N2	115.9 (2)
H17A—C17—H17C	109.5	C18—N4—N5	119.7 (2)
H17B—C17—H17C	109.5	C18—N4—H4A	120.1
O2—C18—N4	124.7 (3)	N5—N4—H4A	120.1
O2—C18—C5	120.4 (3)	C19—N5—N4	115.6 (2)
N4—C18—C5	114.8 (2)	C13—O3—C14	118.9 (2)
N5—C19—C20	120.8 (3)	C25—O6—C26	118.5 (2)

N1—C1—C2—C3	1.8 (4)	C23—C24—C25—C20	−0.2 (5)
C6—C1—C2—C3	−178.0 (2)	C21—C20—C25—O6	−177.9 (3)
C1—C2—C3—C4	−0.5 (4)	C19—C20—C25—O6	1.1 (4)
C2—C3—C4—C5	−1.6 (4)	C21—C20—C25—C24	0.7 (4)
C3—C4—C5—N1	2.7 (4)	C19—C20—C25—C24	179.7 (3)
C3—C4—C5—C18	−175.0 (3)	O6—C26—C27—O7	5.4 (7)
N1—C1—C6—O1	163.9 (2)	O6—C26—C27—O8	−170.8 (6)
C2—C1—C6—O1	−16.3 (4)	O6—C26—C27—O8′	153.9 (10)
N1—C1—C6—N2	−17.8 (3)	O4—C15—O5—C16	−9.6 (8)
C2—C1—C6—N2	162.0 (2)	C14—C15—O5—C16	170.5 (6)
N3—C7—C8—C9	7.8 (4)	C17—C16—O5—C15	99.1 (12)
N3—C7—C8—C13	−171.5 (2)	O7—C27—O8—C28	25.2 (11)
C13—C8—C9—C10	0.2 (4)	O8′—C27—O8—C28	−75.9 (19)
C7—C8—C9—C10	−179.1 (3)	C26—C27—O8—C28	−158.4 (7)
C8—C9—C10—C11	−0.4 (5)	C29—C28—O8—C27	−99.3 (11)
C9—C10—C11—C12	0.6 (5)	O7—C27—O8′—C28′	−64 (6)
C10—C11—C12—C13	−0.7 (4)	O8—C27—O8′—C28′	37 (5)
C11—C12—C13—O3	−179.7 (3)	C26—C27—O8′—C28′	145 (5)
C11—C12—C13—C8	0.5 (4)	C27—O8′—C28′—C29′	146 (7)
C9—C8—C13—O3	179.9 (2)	C2—C1—N1—C5	−0.8 (4)
C7—C8—C13—O3	−0.8 (4)	C6—C1—N1—C5	179.0 (2)
C9—C8—C13—C12	−0.3 (4)	C4—C5—N1—C1	−1.5 (4)
C7—C8—C13—C12	179.0 (2)	C18—C5—N1—C1	176.2 (2)
O3—C14—C15—O4	2.4 (5)	O1—C6—N2—N3	1.3 (4)
O3—C14—C15—O5	−177.7 (3)	C1—C6—N2—N3	−176.90 (19)
N1—C5—C18—O2	152.8 (3)	C8—C7—N3—N2	179.9 (2)
C4—C5—C18—O2	−29.3 (4)	C6—N2—N3—C7	−175.1 (2)
N1—C5—C18—N4	−29.8 (4)	O2—C18—N4—N5	1.4 (5)
C4—C5—C18—N4	148.0 (3)	C5—C18—N4—N5	−175.8 (2)
N5—C19—C20—C25	−172.5 (3)	C20—C19—N5—N4	−178.2 (2)

N5—C19—C20—C21	6.6 (5)	C18—N4—N5—C19	177.1 (3)
C25—C20—C21—C22	−1.0 (5)	C12—C13—O3—C14	3.5 (4)
C19—C20—C21—C22	179.9 (3)	C8—C13—O3—C14	−176.6 (2)
C20—C21—C22—C23	0.9 (5)	C15—C14—O3—C13	−78.7 (3)
C21—C22—C23—C24	−0.3 (5)	C24—C25—O6—C26	−5.8 (5)
C22—C23—C24—C25	0.0 (5)	C20—C25—O6—C26	172.7 (3)
C23—C24—C25—O6	178.3 (3)	C27—C26—O6—C25	−169.8 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O5^{i}	0.86	2.53	3.353 (3)	161
C2—H2···O7^{ii}	0.93	2.40	3.304 (4)	165
C17—H17*C*···O2^{iii}	0.96	2.49	2.896 (8)	105
N2—H2*A*···N1	0.86	2.34	2.694 (3)	105

Symmetry codes: (i) −*x*+2, −*y*, −*z*+2; (ii) −*x*+1, −*y*, −*z*+2; (iii) −*x*+3/2, *y*+1/2, −*z*+3/2.