

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

N'-(3,4-Dimethoxybenzylidene)-3,5-dihydroxybenzohydrazide methanol monosolvate

 Qi-Hui Zhang,^{a*} Lian-Di Zhou,^b Chuan-Xun Li,^c
 Shan-Shan Huang^c and Bao-Jing Zhang^c

^aCollege of Chemistry and Chemical Engineering, Chongqing University, Chongqing 400030, People's Republic of China, ^bDepartment of Immunology, Basic Medical College, Chongqing Medical University, Chongqing 400016, People's Republic of China, and ^cSchool of Pharmacy, Dalian Medical University, Dalian 116044, People's Republic of China

Correspondence e-mail: zhangqh11@126.com

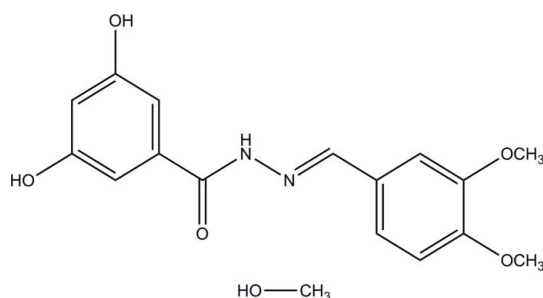
Received 12 July 2011; accepted 23 July 2011

 Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.055; wR factor = 0.162; data-to-parameter ratio = 12.6.

In the title compound, $\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_5 \cdot \text{CH}_4\text{O}$, the two benzene rings in the Schiff base molecule form a dihedral angle of 17.1 (1)°. In the crystal, intermolecular $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds link the components into corrugated sheets parallel to the (101) plane.

Related literature

For the crystal structures of related Schiff base compounds, see: Deng *et al.* (2009); Huang *et al.* (2008). For antibacterial and antitumor activities of Schiff base complexes, see: Brückner *et al.* (2000); Harrop *et al.* (2003); Ren *et al.* (2002).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_5 \cdot \text{CH}_4\text{O}$
 $M_r = 348.35$
 Monoclinic, $P2_1/c$
 $a = 12.467$ (3) Å
 $b = 12.201$ (3) Å
 $c = 11.149$ (3) Å
 $\beta = 91.191$ (3)°

$V = 1695.5$ (7) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 295$ K
 $0.22 \times 0.18 \times 0.15$ mm

Data collection

Bruker APEX2 CCD
 diffractometer
 8351 measured reflections

2984 independent reflections
 1794 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.041$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.055$
 $wR(F^2) = 0.162$
 $S = 1.00$
 2984 reflections
 236 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.68$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.32$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O2}-\text{H2} \cdots \text{O6}$	0.82	1.92	2.728 (3)	168
$\text{O1}-\text{H1} \cdots \text{O5}^{\text{i}}$	0.82	1.93	2.742 (3)	172
$\text{O6}-\text{H6} \cdots \text{O4}^{\text{ii}}$	0.82	2.19	2.973 (4)	161

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *APEX2*; data reduction: *SAINTE-Plus* (Bruker, 2007); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

This project was supported by the Fundamental Research Funds for the Central Universities (project No. CDJRC10220006).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5134).

References

- Brückner, C., Rettig, S. J. & Dolphin, D. (2000). *Inorg. Chem.* **39**, 6100–6106.
 Bruker (2007). *APEX2* and *SAINTE-Plus*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Deng, S., Han, L., Huang, S., Zhang, H., Diao, Y. & Liu, K. (2009). *Acta Cryst.* **E65**, o721.
 Harrop, T. C., Olmstead, M. M. & Mascharak, P. K. (2003). *Chem. Commun.* pp. 410–411.
 Huang, S. S., Diao, Y. P. & Kang, T. G. (2008). *Z. Kristallogr. New Cryst. Struct.* **223**, 167–168.
 Ren, S., Wang, R., Komatsu, K., Bonaz-Krause, P., Zyrianov, Y., McKenna, C. E., Csipke, C., Tokes, Z. A. & Lien, E. J. (2002). *J. Med. Chem.* **45**, 410–419.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supporting information

Acta Cryst. (2011). E67, o2189 [doi:10.1107/S1600536811029850]

***N'*-(3,4-Dimethoxybenzylidene)-3,5-dihydroxybenzohydrazide methanol monosolvate**

Qi-Hui Zhang, Lian-Di Zhou, Chuan-Xun Li, Shan-Shan Huang and Bao-Jing Zhang

S1. Comment

Schiff bases containing the C=N fragments arouses considerable interest due to their antifungal, anticancer and antibacterial activities (Brückner *et al.*, 2000; Harrop *et al.*, 2003; Ren *et al.*, 2002). In continuation of our structural studies of Schiff base compounds (Deng *et al.*, 2009; Huang *et al.*, 2008) we present here the crystal structure of the title compound, (I).

In (I) (Fig. 1), the C7—O5 and C8—N2 bond lengths are 1.228 (3) and 1.274 (3) Å, respectively, corresponding to those reported for the related compounds (Deng *et al.*, 2009; Huang *et al.*, 2008). The solvent molecule is linked to the Schiff base molecule *via* O—H···O hydrogen bond (Table 1).

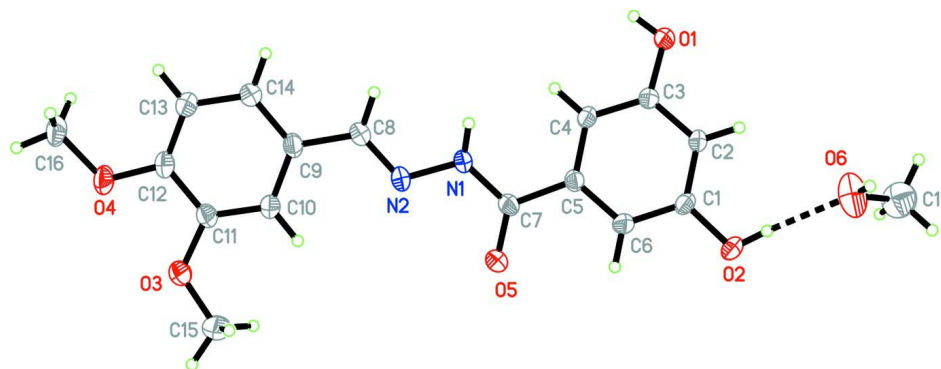
In the crystal structure, intermolecular O—H···O hydrogen bonds (Table 1) link all moieties into corrugated sheets parallel to (101) plane.

S2. Experimental

3,5-Dihydroxybenzhydrazide (0.1 mmol, 16.8 mg) and 3,4-dimethoxybenzaldehyde (0.1 mmol, 16.6 mg) were dissolved in a methanol solution (10 ml). The mixture was stirred at room temperature for 20 minutes and filtered. After keeping the filtrate in air for three days, colourless block-like crystals were formed.

S3. Refinement

The H18 atom bonded to N1 was located in a difference map and refined isotropically, Other H atoms were placed in geometrically idealized positions (C—H 0.93-0.97 Å; O—H 0.82 Å), and allowed to ride on their parent atoms, with $U_{\text{iso}}(\text{H})=1.2-1.5 U_{\text{eq}}$ of the parent atom.

**Figure 1**

The molecular structure of (I) showing the atomic numbering and 30% probability displacement ellipsoids. Dashed line denotes hydrogen bond.

N'-(3,4-Dimethoxybenzylidene)-3,5-dihydroxybenzohydrazide methanol monosolvate

Crystal data

$C_{16}H_{16}N_2O_5 \cdot CH_4O$

$M_r = 348.35$

Monoclinic, $P2_1/c$

Hall symbol: $-P2_1/c$

$a = 12.467$ (3) Å

$b = 12.201$ (3) Å

$c = 11.149$ (3) Å

$\beta = 91.191$ (3)°

$V = 1695.5$ (7) Å³

$Z = 4$

$F(000) = 736$

$D_x = 1.365$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1340 reflections

$\theta = 2.3$ – 21.1 °

$\mu = 0.10$ mm⁻¹

$T = 295$ K

Block, colourless

$0.22 \times 0.18 \times 0.15$ mm

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

phi and ω scans

8351 measured reflections

2984 independent reflections

1794 reflections with $I > 2\sigma(I)$

$R_{int} = 0.041$

$\theta_{max} = 25.0$ °, $\theta_{min} = 2.3$ °

$h = -14 \rightarrow 11$

$k = -14 \rightarrow 12$

$l = -12 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.055$

$wR(F^2) = 0.162$

$S = 1.00$

2984 reflections

236 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0897P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.68$ e Å⁻³

$\Delta\rho_{min} = -0.32$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
H18	0.492 (3)	0.340 (3)	0.619 (3)	0.067 (11)*
N2	0.38492 (18)	0.42464 (18)	0.54230 (19)	0.0425 (6)
O5	0.44125 (17)	0.56294 (14)	0.72026 (18)	0.0515 (5)
O1	0.65292 (18)	0.18020 (14)	0.96344 (19)	0.0601 (7)
H1	0.6297	0.1410	0.9091	0.090*
O2	0.70583 (18)	0.55400 (15)	1.06156 (18)	0.0544 (6)
H2	0.7458	0.5258	1.1121	0.082*
N1	0.4573 (2)	0.3984 (2)	0.6318 (2)	0.0430 (6)
C8	0.3653 (2)	0.3502 (2)	0.4648 (2)	0.0421 (7)
H8	0.4011	0.2835	0.4716	0.051*
C9	0.2892 (2)	0.3658 (2)	0.3669 (2)	0.0402 (7)
C4	0.5705 (2)	0.3194 (2)	0.8382 (2)	0.0390 (7)
H4	0.5406	0.2673	0.7867	0.047*
C6	0.5969 (2)	0.5075 (2)	0.8954 (2)	0.0387 (7)
H6A	0.5834	0.5817	0.8830	0.046*
C7	0.4798 (2)	0.4703 (2)	0.7203 (2)	0.0373 (6)
O3	0.10563 (17)	0.57181 (16)	0.23529 (19)	0.0611 (6)
O4	0.06565 (17)	0.41280 (18)	0.09236 (18)	0.0589 (6)
C1	0.6621 (2)	0.4745 (2)	0.9900 (2)	0.0381 (7)
C2	0.6813 (2)	0.3649 (2)	1.0103 (2)	0.0411 (7)
H2A	0.7253	0.3430	1.0743	0.049*
C5	0.5515 (2)	0.43046 (19)	0.8190 (2)	0.0348 (6)
C14	0.2689 (2)	0.2816 (2)	0.2869 (2)	0.0450 (7)
H14	0.3055	0.2156	0.2958	0.054*
C11	0.1620 (2)	0.4774 (2)	0.2587 (2)	0.0423 (7)
C10	0.2352 (2)	0.4653 (2)	0.3514 (2)	0.0411 (7)
H10	0.2488	0.5232	0.4038	0.049*
C12	0.1404 (2)	0.3909 (2)	0.1795 (2)	0.0443 (7)
C3	0.6346 (2)	0.2874 (2)	0.9350 (2)	0.0400 (7)
C13	0.1950 (2)	0.2940 (2)	0.1939 (3)	0.0479 (8)
H13	0.1821	0.2365	0.1409	0.058*
C16	0.0472 (3)	0.3318 (3)	0.0018 (3)	0.0626 (9)
H16A	0.1120	0.3200	-0.0414	0.094*
H16B	-0.0083	0.3565	-0.0527	0.094*
H16C	0.0255	0.2644	0.0386	0.094*

C15	0.1393 (3)	0.6683 (3)	0.2954 (3)	0.0755 (11)
H15A	0.1316	0.6589	0.3803	0.113*
H15B	0.0961	0.7289	0.2684	0.113*
H15C	0.2132	0.6825	0.2783	0.113*
C17	0.8698 (4)	0.4724 (4)	1.3338 (3)	0.0897 (13)
H17A	0.9159	0.5304	1.3616	0.135*
H17B	0.8985	0.4032	1.3598	0.135*
H17C	0.7995	0.4821	1.3658	0.135*
O6	0.8632 (2)	0.4745 (3)	1.2114 (3)	0.1058 (10)
H6	0.9237	0.4726	1.1841	0.159*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N2	0.0467 (14)	0.0457 (14)	0.0346 (13)	0.0035 (11)	-0.0118 (11)	0.0033 (11)
O5	0.0674 (14)	0.0377 (11)	0.0485 (12)	0.0063 (10)	-0.0173 (10)	0.0032 (9)
O1	0.0853 (17)	0.0318 (11)	0.0617 (14)	0.0043 (10)	-0.0389 (12)	-0.0004 (9)
O2	0.0710 (15)	0.0387 (11)	0.0526 (13)	-0.0065 (10)	-0.0229 (11)	-0.0088 (9)
N1	0.0514 (15)	0.0414 (14)	0.0354 (13)	0.0094 (12)	-0.0155 (11)	-0.0022 (11)
C8	0.0459 (17)	0.0409 (15)	0.0392 (16)	0.0054 (13)	-0.0074 (13)	0.0040 (13)
C9	0.0420 (17)	0.0450 (16)	0.0333 (15)	-0.0011 (13)	-0.0075 (13)	0.0039 (12)
C4	0.0471 (17)	0.0339 (14)	0.0356 (15)	-0.0042 (12)	-0.0097 (13)	-0.0023 (11)
C6	0.0468 (17)	0.0297 (14)	0.0394 (16)	-0.0025 (12)	-0.0043 (13)	0.0010 (12)
C7	0.0410 (16)	0.0354 (15)	0.0355 (15)	0.0004 (12)	-0.0028 (13)	0.0057 (12)
O3	0.0666 (15)	0.0524 (13)	0.0629 (14)	0.0191 (11)	-0.0278 (11)	-0.0073 (10)
O4	0.0559 (13)	0.0718 (14)	0.0479 (12)	0.0096 (11)	-0.0243 (10)	-0.0095 (10)
C1	0.0433 (16)	0.0349 (15)	0.0360 (15)	-0.0068 (12)	-0.0062 (13)	-0.0050 (12)
C2	0.0478 (17)	0.0369 (15)	0.0380 (15)	0.0007 (13)	-0.0156 (13)	-0.0012 (12)
C5	0.0363 (15)	0.0355 (14)	0.0324 (14)	-0.0020 (12)	-0.0049 (12)	0.0033 (11)
C14	0.0537 (18)	0.0400 (15)	0.0409 (16)	0.0041 (14)	-0.0065 (14)	-0.0002 (13)
C11	0.0410 (16)	0.0454 (16)	0.0402 (16)	0.0069 (13)	-0.0064 (13)	0.0011 (13)
C10	0.0430 (16)	0.0439 (16)	0.0361 (15)	0.0023 (13)	-0.0068 (13)	-0.0018 (12)
C12	0.0403 (17)	0.0546 (18)	0.0376 (16)	-0.0019 (14)	-0.0088 (13)	0.0007 (13)
C3	0.0464 (17)	0.0322 (15)	0.0407 (16)	-0.0013 (12)	-0.0113 (13)	0.0009 (12)
C13	0.0561 (19)	0.0460 (17)	0.0412 (17)	-0.0029 (14)	-0.0088 (15)	-0.0047 (13)
C16	0.069 (2)	0.071 (2)	0.0461 (18)	-0.0068 (18)	-0.0232 (17)	-0.0041 (16)
C15	0.091 (3)	0.051 (2)	0.083 (3)	0.0223 (19)	-0.022 (2)	-0.0127 (19)
C17	0.108 (4)	0.100 (3)	0.061 (3)	-0.003 (3)	0.011 (2)	-0.002 (2)
O6	0.077 (2)	0.151 (3)	0.088 (2)	0.007 (2)	-0.0210 (16)	0.011 (2)

Geometric parameters (Å, °)

N2—C8	1.274 (3)	O4—C16	1.429 (3)
N2—N1	1.370 (3)	C1—C2	1.376 (4)
O5—C7	1.228 (3)	C2—C3	1.384 (4)
O1—C3	1.364 (3)	C2—H2A	0.9300
O1—H1	0.8200	C14—C13	1.381 (4)
O2—C1	1.362 (3)	C14—H14	0.9300

O2—H2	0.8200	C11—C10	1.372 (4)
N1—C7	1.345 (3)	C11—C12	1.399 (4)
N1—H18	0.85 (3)	C10—H10	0.9300
C8—C9	1.443 (4)	C12—C13	1.371 (4)
C8—H8	0.9300	C13—H13	0.9300
C9—C14	1.380 (4)	C16—H16A	0.9600
C9—C10	1.398 (4)	C16—H16B	0.9600
C4—C3	1.385 (4)	C16—H16C	0.9600
C4—C5	1.391 (3)	C15—H15A	0.9600
C4—H4	0.9300	C15—H15B	0.9600
C6—C1	1.379 (4)	C15—H15C	0.9600
C6—C5	1.382 (3)	C17—O6	1.366 (4)
C6—H6A	0.9300	C17—H17A	0.9600
C7—C5	1.486 (4)	C17—H17B	0.9600
O3—C11	1.371 (3)	C17—H17C	0.9600
O3—C15	1.414 (4)	O6—H6	0.8200
O4—C12	1.360 (3)		
C8—N2—N1	116.2 (2)	O3—C11—C10	124.3 (2)
C3—O1—H1	109.5	O3—C11—C12	115.0 (2)
C1—O2—H2	109.5	C10—C11—C12	120.7 (2)
C7—N1—N2	120.4 (2)	C11—C10—C9	119.9 (2)
C7—N1—H18	125 (2)	C11—C10—H10	120.1
N2—N1—H18	114 (2)	C9—C10—H10	120.1
N2—C8—C9	122.2 (3)	O4—C12—C13	125.8 (3)
N2—C8—H8	118.9	O4—C12—C11	115.0 (2)
C9—C8—H8	118.9	C13—C12—C11	119.2 (3)
C14—C9—C10	119.0 (2)	O1—C3—C2	116.6 (2)
C14—C9—C8	120.0 (2)	O1—C3—C4	122.8 (2)
C10—C9—C8	121.0 (2)	C2—C3—C4	120.6 (2)
C3—C4—C5	119.2 (2)	C12—C13—C14	120.3 (3)
C3—C4—H4	120.4	C12—C13—H13	119.9
C5—C4—H4	120.4	C14—C13—H13	119.9
C1—C6—C5	120.0 (2)	O4—C16—H16A	109.5
C1—C6—H6A	120.0	O4—C16—H16B	109.5
C5—C6—H6A	120.0	H16A—C16—H16B	109.5
O5—C7—N1	121.6 (2)	O4—C16—H16C	109.5
O5—C7—C5	122.1 (2)	H16A—C16—H16C	109.5
N1—C7—C5	116.3 (2)	H16B—C16—H16C	109.5
C11—O3—C15	117.6 (2)	O3—C15—H15A	109.5
C12—O4—C16	117.8 (2)	O3—C15—H15B	109.5
O2—C1—C2	122.0 (2)	H15A—C15—H15B	109.5
O2—C1—C6	117.5 (2)	O3—C15—H15C	109.5
C2—C1—C6	120.5 (2)	H15A—C15—H15C	109.5
C1—C2—C3	119.6 (2)	H15B—C15—H15C	109.5
C1—C2—H2A	120.2	O6—C17—H17A	109.5
C3—C2—H2A	120.2	O6—C17—H17B	109.5
C6—C5—C4	120.1 (2)	H17A—C17—H17B	109.5

C6—C5—C7	117.8 (2)	O6—C17—H17C	109.5
C4—C5—C7	122.1 (2)	H17A—C17—H17C	109.5
C9—C14—C13	120.9 (3)	H17B—C17—H17C	109.5
C9—C14—H14	119.5	C17—O6—H6	109.5
C13—C14—H14	119.5		
C8—N2—N1—C7	-177.3 (2)	C15—O3—C11—C10	12.5 (4)
N1—N2—C8—C9	178.8 (2)	C15—O3—C11—C12	-167.1 (3)
N2—C8—C9—C14	-178.5 (3)	O3—C11—C10—C9	-179.4 (3)
N2—C8—C9—C10	1.4 (4)	C12—C11—C10—C9	0.2 (4)
N2—N1—C7—O5	-4.0 (4)	C14—C9—C10—C11	1.0 (4)
N2—N1—C7—C5	174.5 (2)	C8—C9—C10—C11	-178.9 (3)
C5—C6—C1—O2	179.2 (2)	C16—O4—C12—C13	-5.1 (4)
C5—C6—C1—C2	-1.0 (4)	C16—O4—C12—C11	174.2 (3)
O2—C1—C2—C3	179.8 (3)	O3—C11—C12—O4	-1.0 (4)
C6—C1—C2—C3	0.1 (4)	C10—C11—C12—O4	179.4 (3)
C1—C6—C5—C4	0.8 (4)	O3—C11—C12—C13	178.3 (3)
C1—C6—C5—C7	178.6 (2)	C10—C11—C12—C13	-1.3 (4)
C3—C4—C5—C6	0.4 (4)	C1—C2—C3—O1	-177.1 (3)
C3—C4—C5—C7	-177.2 (2)	C1—C2—C3—C4	1.2 (4)
O5—C7—C5—C6	-16.5 (4)	C5—C4—C3—O1	176.7 (3)
N1—C7—C5—C6	164.9 (2)	C5—C4—C3—C2	-1.4 (4)
O5—C7—C5—C4	161.2 (3)	O4—C12—C13—C14	-179.6 (3)
N1—C7—C5—C4	-17.4 (4)	C11—C12—C13—C14	1.2 (4)
C10—C9—C14—C13	-1.1 (4)	C9—C14—C13—C12	0.1 (4)
C8—C9—C14—C13	178.7 (3)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H2 \cdots O6	0.82	1.92	2.728 (3)	168
O1—H1 \cdots O5 ⁱ	0.82	1.93	2.742 (3)	172
O6—H6 \cdots O4 ⁱⁱ	0.82	2.19	2.973 (4)	161

Symmetry codes: (i) $-x+1, y-1/2, -z+3/2$; (ii) $x+1, y, z+1$.