Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N′-[(*E*)-4-Hydroxy-3-methoxybenzylidene]pyridine-4-carbohydrazide

Zahid Shafiq,[a] Muhammad Yaqub,[b] M. Nawaz Tahir,[c]* Abid Hussain[b] and M. Saeed Iqbal[d]

[a]Department of Chemistry, Bahauddin Zakariya University, Multan-60800, Pakistan, [b]Department of Chemistry, Bahauddin Zakariya University, Multan 60800, Pakistan, [c]Department of Physics, University of Sargodha, Sargodha, Pakistan, and [d]Department of Chemistry, Government College University, Lahore, Pakistan
Correspondence e-mail: dmntahir_uos@yahoo.com

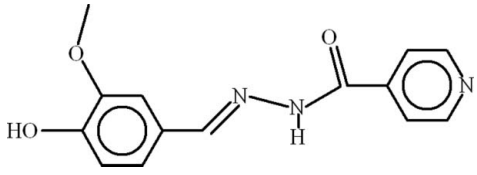
Received 23 October 2009; accepted 23 October 2009

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.086; data-to-parameter ratio = 8.8.

In the title compound, $C_{14}H_{13}N_3O_3$, the two six-membered rings are oriented at a dihedral angle of 15.17 (11)° and an intramolecular O—H···O hydrogen bond occurs. In the crystal, molecules interact by way of N—H···O, O—H···N and C—H···O hydrogen bonds, thereby generating S(5) chain and $R_2^1(7)$ ring motifs.

Related literature

For related structures, see: Liu & Shi (2007); Shi *et al.* (2007); Shafiq *et al.* (2009). For graph-set theory, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{14}H_{13}N_3O_3$
M_r = 271.27
Monoclinic, *Cc*
a = 14.8543 (10) Å
b = 12.4943 (9) Å
c = 7.7162 (5) Å
β = 116.716 (2)°
V = 1279.20 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 K
0.32 × 0.14 × 0.10 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.973, T_{max} = 0.984
7060 measured reflections
1613 independent reflections
1431 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.086
S = 1.04
1613 reflections
183 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2*B*···O3	0.82	2.25	2.694 (2)	114
N2—H2*A*···O1^{i}	0.86	2.25	3.089 (2)	164
O2—H2*B*···N1^{ii}	0.82	1.96	2.703 (3)	150
C5—H5···O1^{i}	0.93	2.55	3.410 (3)	153

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

AH gratefully acknowledges the Higher Education Commission, Islamabad, Pakistan, for providing him with a Scholaship under the Indigenous PhD Program (PIN 063–121531-PS3–127).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5177).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555-1573.
Bruker (2005). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Liu, X. & Shi, X.-F. (2007). *Acta Cryst.* E**63**, o4807.
Shafiq, Z., Yaqub, M., Tahir, M. N., Hussain, A. & Iqbal, M. S. (2009). *Acta Cryst.* E**65**, o2898.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Shi, X.-F., He, L., Ma, G.-Z. & Yuan, C.-C. (2007). *Acta Cryst.* E**63**, o1119–o1120.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supporting information

Acta Cryst. (2009). E**65**, o2899 [https://doi.org/10.1107/S1600536809044134]

N′-[(*E*)-4-Hydroxy-3-methoxybenzylidene]pyridine-4-carbohydrazide

Zahid Shafiq, Muhammad Yaqub, M. Nawaz Tahir, Abid Hussain and M. Saeed Iqbal

S1. Comment

We have reported the crystal structures of (II) *N*′-[(*E*)-(4-Hydroxy-3-methoxyphenyl)methylidene]benzohydrazide (Shafiq *et al.*, 2009). The title compound (I, Fig. 1), has been prepared in continuation of synthesizing hydrazide derivatives.

The crystal structure of (III) *N*′-(4-Hydroxy-3-methoxybenzylidene)isonicotinohydrazide monohydrate (Shi *et al.*, 2007) and (IV) *N*′-(4-Hydroxy-3-methoxybenzylidene)isonicotinohydrazide methanol solvate (Liu & Shi, 2007) have also been reported. The title compound differs from (III) and (IV) as there is no solvate.

In the title compound the pyridine ring A (C1–C3/N1/C4/C5) and the benzene ring of vanilline B (C8—C13) are planar with a maximum r. m. s. deviations of 0.0061 and 0.0122 Å respectively, from their mean square planes. The dihedral angle between A/B is 15.17 (11)°. The intramolecular H-bonding of O—H···O type completes S(5) ring motif (Bernstein *et al.*, 1995). There also exist $R_2^1(7)$ ring motif due to intermolecular H-bondings of C—H···O and N—H···O type (Table 1, Fig. 2). The molecules are stabilized in the form of two dimensional polymeric sheets owing to intermolecular H-bondings of O—H···N type (Fig. 2).

S2. Experimental

To a hot stirred solution of isoniazid (1.37 g, 0.01 mol) in ethanol (15 ml) was added vanillin (1.52 g, 0.01 mol). The resultant mixture was then heated under reflux. After an hour precipitates were formed. The reaction mixture was further heated about 30 min for the completion of the reaction which was monitored through TLC. The reaction mixture was cooled to room temperature, filtered and washed with hot ethanol. Yellow needles of (I) were obtained by recrystallization of the crude product in 1,4-dioxan:ethanol (1:1) after two days.

S3. Refinement

In the absence of significant anomalous dispersion effects, Friedel pairs were averaged before refinement.

The H-atoms were positioned geometrically (O–H = 0.82 Å, N–H = 0.86 Å, C–H = 0.93–0.96 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(carrier) or 1.5U_{eq}(methyl C).

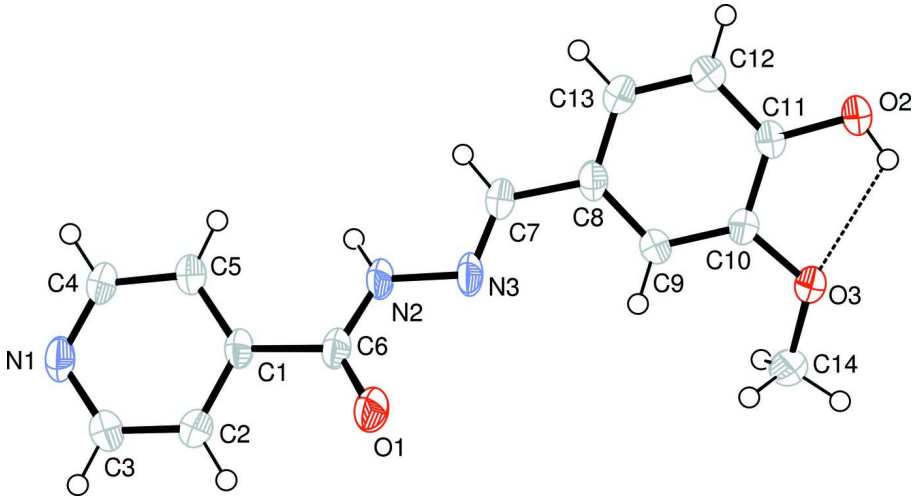


Figure 1

View of (I) with displacement ellipsoids drawn at the 50% probability level. H-atoms are shown by spheres of arbitrary radius. The dotted line represent the intramolecular H-bondings.

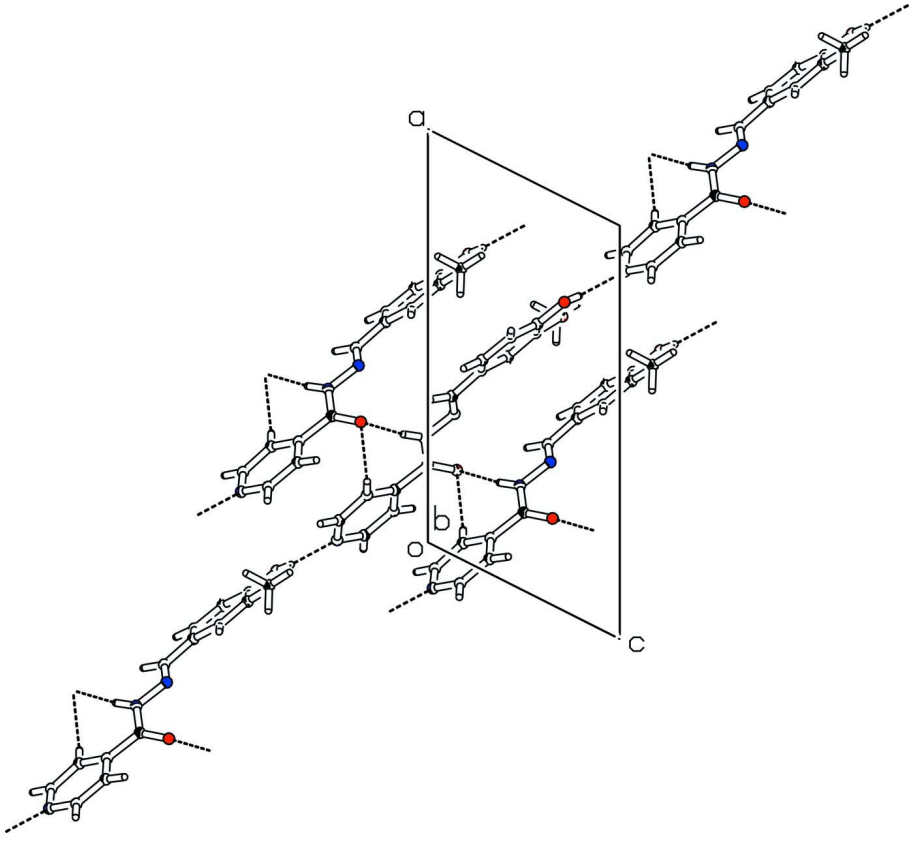


Figure 2

The partial packing of (I), which shows that molecules form two dimensional polymeric chains.

N′-[(*E*)-4-Hydroxy-3-methoxybenzylidene]pyridine-4-carbohydrazide

Crystal data

$C_{14}H_{13}N_3O_3$
$M_r = 271.27$
Monoclinic, *Cc*
Hall symbol: C -2yc
$a = 14.8543\ (10)$ Å
$b = 12.4943\ (9)$ Å
$c = 7.7162\ (5)$ Å
$\beta = 116.716\ (2)°$
$V = 1279.20\ (15)$ Å^3
$Z = 4$
$F(000) = 568$
$D_x = 1.409$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1613 reflections
$\theta = 2.2–28.7°$
$\mu = 0.10$ mm^{-1}
$T = 296$ K
Needle, yellow
0.32 × 0.14 × 0.10 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.40 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.973$, $T_{max} = 0.984$
7060 measured reflections
1613 independent reflections
1431 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 28.7°$, $\theta_{min} = 2.2°$
$h = -17\rightarrow19$
$k = -16\rightarrow16$
$l = -10\rightarrow5$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.086$
$S = 1.04$
1613 reflections
183 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.0491P)^2 + 0.2451P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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}
O1	0.20978 (12)	−0.14131 (15)	0.1499 (2)	0.0464 (5)
O2	0.74733 (14)	0.23723 (14)	0.7114 (3)	0.0495 (5)
O3	0.71022 (13)	0.02563 (13)	0.6995 (3)	0.0462 (5)
N1	−0.10456 (14)	−0.14377 (17)	−0.4734 (3)	0.0415 (6)
N2	0.24963 (13)	−0.01999 (16)	−0.0200 (2)	0.0357 (5)

N3	0.34327 (13)	−0.00082 (17)	0.1376 (3)	0.0371 (5)
C1	0.08684 (14)	−0.10723 (18)	−0.1727 (3)	0.0302 (6)
C2	0.03464 (17)	−0.2001 (2)	−0.1794 (3)	0.0400 (7)
C3	−0.06004 (17)	−0.2154 (2)	−0.3320 (4)	0.0455 (8)
C4	−0.05475 (16)	−0.0540 (2)	−0.4634 (3)	0.0387 (6)
C5	0.04083 (16)	−0.03215 (18)	−0.3174 (3)	0.0342 (6)
C6	0.18841 (15)	−0.09215 (18)	−0.0012 (3)	0.0317 (6)
C7	0.38334 (15)	0.08629 (19)	0.1254 (3)	0.0355 (6)
C8	0.48092 (15)	0.12099 (18)	0.2766 (3)	0.0322 (6)
C9	0.54708 (16)	0.05029 (18)	0.4162 (3)	0.0338 (6)
C10	0.63784 (15)	0.08723 (18)	0.5602 (3)	0.0320 (6)
C11	0.66190 (15)	0.19614 (18)	0.5701 (3)	0.0318 (6)
C12	0.59690 (16)	0.26504 (19)	0.4295 (3)	0.0353 (6)
C13	0.50728 (15)	0.22751 (19)	0.2826 (3)	0.0360 (6)
C14	0.70271 (18)	−0.08742 (19)	0.6725 (4)	0.0424 (7)
H2	0.06282	−0.25144	−0.08249	0.0480*
H2A	0.23175	0.01414	−0.12720	0.0428*
H2B	0.77967	0.18945	0.78640	0.0594*
H3	−0.09420	−0.27850	−0.33636	0.0546*
H4	−0.08569	−0.00289	−0.55979	0.0465*
H5	0.07325	0.03162	−0.31695	0.0411*
H7	0.34970	0.12947	0.01696	0.0425*
H9	0.53013	−0.02163	0.41231	0.0405*
H12	0.61341	0.33710	0.43342	0.0424*
H13	0.46457	0.27431	0.18744	0.0432*
H14A	0.70409	−0.10498	0.55254	0.0636*
H14B	0.64062	−0.11205	0.66859	0.0636*
H14C	0.75836	−0.12140	0.77800	0.0636*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0372 (9)	0.0570 (11)	0.0298 (8)	−0.0070 (8)	0.0015 (7)	0.0075 (7)
O2	0.0341 (8)	0.0373 (9)	0.0468 (9)	−0.0080 (7)	−0.0086 (7)	0.0022 (8)
O3	0.0347 (8)	0.0346 (9)	0.0433 (9)	−0.0031 (7)	−0.0056 (7)	0.0066 (7)
N1	0.0238 (8)	0.0486 (12)	0.0369 (10)	−0.0013 (8)	0.0001 (7)	−0.0017 (8)
N2	0.0252 (9)	0.0448 (11)	0.0234 (8)	−0.0053 (8)	−0.0013 (7)	0.0011 (7)
N3	0.0233 (8)	0.0481 (11)	0.0253 (8)	−0.0042 (8)	−0.0021 (7)	−0.0012 (8)
C1	0.0226 (9)	0.0371 (11)	0.0235 (9)	−0.0011 (8)	0.0037 (8)	−0.0041 (8)
C2	0.0317 (12)	0.0392 (12)	0.0366 (11)	−0.0011 (9)	0.0042 (10)	0.0068 (10)
C3	0.0302 (12)	0.0441 (14)	0.0484 (13)	−0.0087 (10)	0.0054 (10)	0.0025 (11)
C4	0.0258 (10)	0.0455 (13)	0.0316 (10)	0.0017 (9)	0.0011 (8)	0.0037 (9)
C5	0.0260 (10)	0.0387 (12)	0.0295 (10)	−0.0028 (9)	0.0050 (8)	−0.0021 (9)
C6	0.0242 (9)	0.0376 (12)	0.0240 (9)	0.0021 (8)	0.0027 (8)	−0.0028 (8)
C7	0.0257 (10)	0.0421 (12)	0.0281 (10)	−0.0004 (9)	0.0028 (8)	0.0005 (9)
C8	0.0230 (9)	0.0403 (12)	0.0269 (9)	−0.0028 (9)	0.0055 (8)	−0.0015 (9)
C9	0.0280 (10)	0.0311 (11)	0.0327 (10)	−0.0059 (8)	0.0052 (8)	0.0006 (8)
C10	0.0258 (9)	0.0326 (11)	0.0291 (9)	−0.0010 (8)	0.0048 (8)	0.0027 (8)

C11	0.0242 (9)	0.0343 (11)	0.0296 (10)	−0.0037 (8)	0.0055 (8)	−0.0018 (8)
C12	0.0323 (11)	0.0304 (11)	0.0358 (11)	−0.0033 (9)	0.0087 (9)	−0.0007 (9)
C13	0.0295 (11)	0.0387 (12)	0.0305 (10)	0.0035 (9)	0.0053 (9)	0.0039 (9)
C14	0.0352 (12)	0.0342 (12)	0.0485 (13)	0.0013 (10)	0.0105 (10)	0.0065 (10)

Geometric parameters (Å, º)

O1—C6	1.227 (3)	C8—C13	1.382 (3)
O2—C11	1.349 (3)	C8—C9	1.398 (3)
O3—C10	1.366 (3)	C9—C10	1.385 (3)
O3—C14	1.425 (3)	C10—C11	1.400 (3)
O2—H2B	0.8200	C11—C12	1.382 (3)
N1—C3	1.334 (3)	C12—C13	1.386 (3)
N1—C4	1.327 (3)	C2—H2	0.9300
N2—C6	1.334 (3)	C3—H3	0.9300
N2—N3	1.396 (3)	C4—H4	0.9300
N3—C7	1.264 (3)	C5—H5	0.9300
N2—H2A	0.8600	C7—H7	0.9300
C1—C5	1.381 (3)	C9—H9	0.9300
C1—C2	1.384 (3)	C12—H12	0.9300
C1—C6	1.506 (3)	C13—H13	0.9300
C2—C3	1.383 (4)	C14—H14A	0.9600
C4—C5	1.386 (3)	C14—H14B	0.9600
C7—C8	1.459 (3)	C14—H14C	0.9600
C10—O3—C14	117.5 (2)	O2—C11—C10	122.6 (2)
C11—O2—H2B	109.00	C10—C11—C12	119.5 (2)
C3—N1—C4	117.5 (2)	C11—C12—C13	120.5 (2)
N3—N2—C6	118.82 (17)	C8—C13—C12	120.5 (2)
N2—N3—C7	113.6 (2)	C1—C2—H2	120.00
C6—N2—H2A	121.00	C3—C2—H2	120.00
N3—N2—H2A	121.00	N1—C3—H3	119.00
C2—C1—C5	118.3 (2)	C2—C3—H3	119.00
C2—C1—C6	117.45 (19)	N1—C4—H4	118.00
C5—C1—C6	124.2 (2)	C5—C4—H4	118.00
C1—C2—C3	119.1 (2)	C1—C5—H5	121.00
N1—C3—C2	123.0 (2)	C4—C5—H5	121.00
N1—C4—C5	123.7 (2)	N3—C7—H7	119.00
C1—C5—C4	118.5 (2)	C8—C7—H7	119.00
O1—C6—N2	123.0 (2)	C8—C9—H9	120.00
N2—C6—C1	116.85 (18)	C10—C9—H9	120.00
O1—C6—C1	120.1 (2)	C11—C12—H12	120.00
N3—C7—C8	121.9 (2)	C13—C12—H12	120.00
C7—C8—C13	118.5 (2)	C8—C13—H13	120.00
C7—C8—C9	122.0 (2)	C12—C13—H13	120.00
C9—C8—C13	119.5 (2)	O3—C14—H14A	109.00
C8—C9—C10	120.1 (2)	O3—C14—H14B	109.00
O3—C10—C9	125.6 (2)	O3—C14—H14C	109.00

O3—C10—C11	114.4 (2)	H14A—C14—H14B	109.00
C9—C10—C11	120.0 (2)	H14A—C14—H14C	109.00
O2—C11—C12	118.0 (2)	H14B—C14—H14C	110.00
C14—O3—C10—C9	14.0 (4)	N1—C4—C5—C1	−0.7 (4)
C14—O3—C10—C11	−165.5 (2)	N3—C7—C8—C9	16.8 (4)
C4—N1—C3—C2	−0.6 (4)	N3—C7—C8—C13	−162.1 (2)
C3—N1—C4—C5	1.3 (4)	C7—C8—C9—C10	−178.3 (2)
C6—N2—N3—C7	−162.0 (2)	C13—C8—C9—C10	0.6 (4)
N3—N2—C6—O1	1.4 (3)	C7—C8—C13—C12	176.7 (2)
N3—N2—C6—C1	177.92 (19)	C9—C8—C13—C12	−2.3 (4)
N2—N3—C7—C8	179.5 (2)	C8—C9—C10—O3	−177.3 (2)
C5—C1—C2—C3	1.5 (4)	C8—C9—C10—C11	2.2 (4)
C6—C1—C2—C3	178.3 (2)	O3—C10—C11—O2	−3.1 (3)
C2—C1—C5—C4	−0.8 (3)	O3—C10—C11—C12	176.2 (2)
C6—C1—C5—C4	−177.4 (2)	C9—C10—C11—O2	177.4 (2)
C2—C1—C6—O1	−21.5 (3)	C9—C10—C11—C12	−3.4 (4)
C2—C1—C6—N2	161.9 (2)	O2—C11—C12—C13	−179.0 (2)
C5—C1—C6—O1	155.1 (2)	C10—C11—C12—C13	1.7 (4)
C5—C1—C6—N2	−21.5 (3)	C11—C12—C13—C8	1.1 (4)
C1—C2—C3—N1	−0.8 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*B*···O3	0.82	2.25	2.694 (2)	114
N2—H2*A*···O1^{i}	0.86	2.25	3.089 (2)	164
O2—H2*B*···N1^{ii}	0.82	1.96	2.703 (3)	150
C5—H5···O1^{i}	0.93	2.55	3.410 (3)	153

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