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N'-(*E*)-Furan-2-ylmethylidene]-4-hydroxybenzohydrazide

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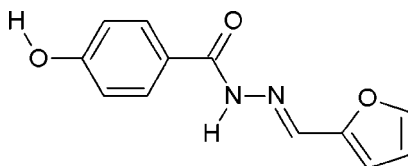
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.024; wR factor = 0.064; data-to-parameter ratio = 6.2.

The title compound, $\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_3$, exists in the *E* conformation. The five-membered ring and the phenyl rings form dihedral angles of 36.73 (10) and 12.22 (10) $^\circ$, respectively, with the central $\text{C}(=\text{O})\text{N}_2\text{C}$ unit. The crystal packing is dominated by strong $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds. Together with weaker $\text{C}-\text{H}\cdots\text{O}$ interactions, these establish a three-dimensional supramolecular network.

Related literature

For biological applications of benzohydrazones and derivatives, see: Sreeja *et al.* (2004); Rakha *et al.* (1996). For the synthesis of related compounds, see: Emmanuel *et al.* (2011). For a related structure, see: Datta *et al.* (2013).



Experimental

Crystal data

 $\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_3$ $M_r = 230.22$ Orthorhombic, $Pna2_1$ $a = 9.5934$ (3) Å $b = 11.1939$ (4) Å $c = 10.3332$ (3) Å $V = 1109.66$ (6) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.10$ mm⁻¹ $T = 298$ K $0.25 \times 0.20 \times 0.16$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Absorption correction: multi-scan (*SADABS*; Bruker, 2004) $T_{\min} = 0.975$, $T_{\max} = 0.984$

3425 measured reflections

1014 independent reflections

992 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.015$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.024$ $wR(F^2) = 0.064$ $S = 1.05$

1014 reflections

163 parameters

3 restraints

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.13$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.10$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, $^\circ$).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N}2-\text{H}2'\cdots\text{O}2^{\text{i}}$	0.88 (1)	2.09 (1)	2.9187 (19)	157 (2)
$\text{O}3-\text{H}3'\cdots\text{N}1^{\text{ii}}$	0.85 (1)	2.13 (1)	2.971 (2)	169 (3)
$\text{C}5-\text{H}5\cdots\text{O}2^{\text{i}}$	0.93	2.35	3.160 (2)	145
$\text{C}11-\text{H}11\cdots\text{O}3^{\text{iii}}$	0.93	2.42	3.202 (2)	142

Symmetry codes: (i) $x - \frac{1}{2}, -y + \frac{3}{2}, z$; (ii) $-x + 2, -y + 2, z - \frac{1}{2}$; (iii) $x + \frac{1}{2}, -y + \frac{5}{2}, z$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREP* (Bruker, 2004); 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, 2012) and *DIAMOND* (Brandenburg, 2010); software used to prepare material for publication: *SHELXL97* and *pubCIF* (Westrip, 2010).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: FJ2657).

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supporting information

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N'*-[(*E*)-Furan-2-ylmethylidene]-4-hydroxybenzohydrazide*Riya Datta, V. Ramya, M. Sithambaresan and M. R. Prathapachandra Kurup****S1. Comment**

Hydrazones and their derivatives show excellent biological activities (Sreeja *et al.*, 2004). The great potential applications of aryl- hydrazones as antineoplastic, antiviral and antiinflammatory agents, hammered on the investigations of their derivatives (Rakha *et al.*, 1996). As a continuous work on hydrazone compounds, a new hydrazone derivative, *N'*-[(*E*)-4,5-dihydrofuran-2-ylmethylidene]-4-hydroxybenzohydrazide, was prepared and structurally characterized. The *ORTEP* view of the title compound is shown in Fig. 1.

The compound crystallizes in orthorhombic space group *Pna*2₁. This molecule adopts an *E* configuration with respect to the C5=N1 bond and it exists in the amido form with a C6=O2 bond length of 1.232 (2) Å which is very close to the reported C=O bond length of similar structure (Datta *et al.*, 2013). The O2 and N1 atoms are in *Z* configuration with respect to C6–N2 having a torsion angle of 3.7 (3)°. The central C(=O)N₂C unit has dihedral angles of 36.73 (10) and 12.22 (10)°, respectively with the five-membered ring and the phenyl ring.

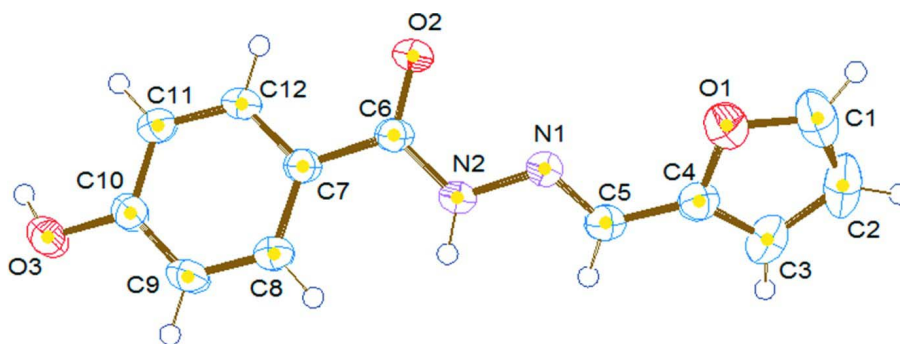
There are two classical intermolecular N2–H2'···O2 and O3–H3'···N1 hydrogen bond interactions (Fig. 2) between the neighbouring molecule with D···A distances of 2.9187 (19) and 2.971 (2) Å respectively (Table 1). Two weak C–H···O hydrogen bond interactions (Fig. 3) between the H atoms attached at the C5 & C11 and O2 & O3 atoms of neighbouring molecules with D···A distances of 3.160 (2) and 3.202 (2) Å respectively, also promote the classical hydrogen bond interactions forming a supramolecular three-dimensional-hydrogen bonding network in the lattice. Notwithstanding that there are very weak short ring interactions found in the crystal system, they are not significant to support the network since centroid-centroid distances are above 4 Å. Fig. 4 shows a packing diagram of the title compound viewed along *a* axis.

S2. Experimental

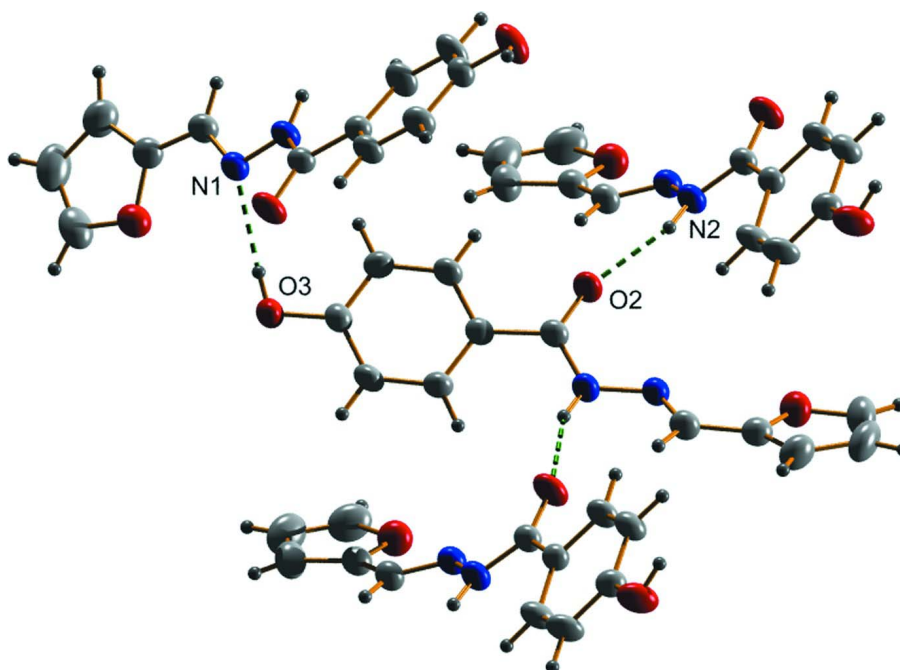
The title compound was prepared by adapting a reported procedure (Emmanuel *et al.*, 2011). A solution of furan-2-carbaldehyde (0.096 g, 1 mmol) in methanol/DMF 2:1 (10 ml) was mixed with a methanol/DMF solution (10 ml) of 4-hydroxybenzhydrazide (0.152 g, 1 mmol). The mixture was refluxed for 6 h and then cooled to room temperature. Light orange colored crystals were formed which were recrystallized in methanol/DMF (2:1 *v/v*). Block shaped crystals, suitable for SXR studies, were obtained after slow evaporation of the solution in air for a few days.

S3. Refinement

The atoms H2' and H3' were located from a difference Fourier map and N2–H2' and O3–H3' distances are restrained to 0.88±0.01 and 0.84±0.01 Å respectively. All the other H atoms on C were placed in calculated positions, guided by difference maps, with C–H bond distances 0.93 Å. H atoms were assigned as $U_{iso}(H)=1.2U_{eq}(carrier)$.

**Figure 1**

ORTEP view of the title compound drawn with 50% probability displacement ellipsoids for the non-H atoms.

**Figure 2**

Classical hydrogen-bonding interactions in the crystal structure of C₁₂H₁₀N₂O₃.

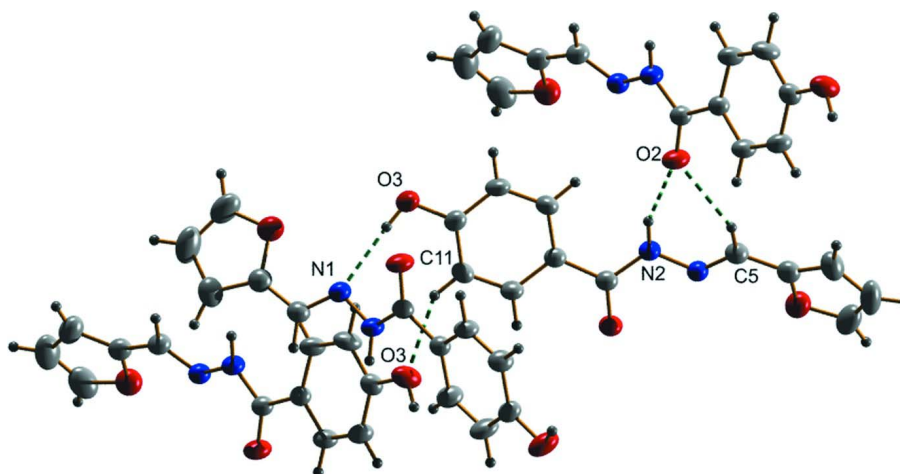


Figure 3
Hydrogen-bonding interactions in the crystal structure of $C_{12}H_{10}N_2O_3$.

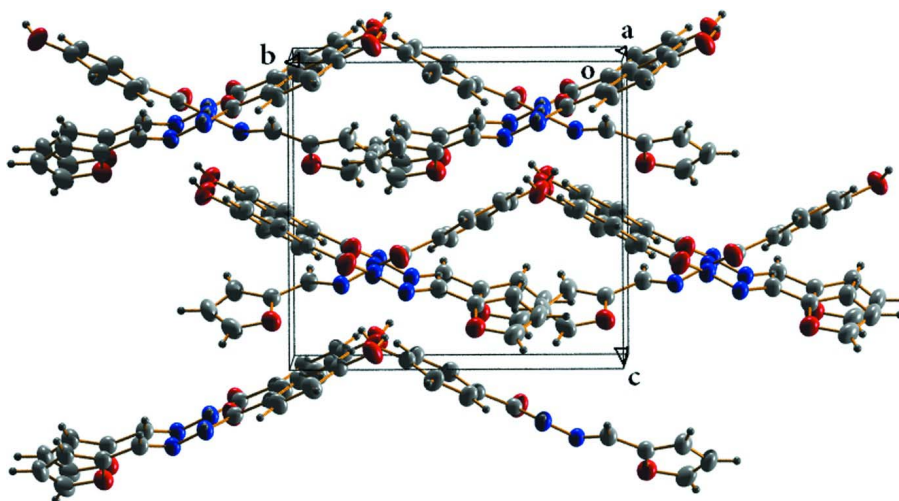


Figure 4
Packing diagram of the compound along the a axis.

N'-[(*E*)-Furan-2-ylmethylidene]-4-hydroxybenzohydrazide

Crystal data

$C_{12}H_{10}N_2O_3$

$M_r = 230.22$

Orthorhombic, $Pna2_1$

Hall symbol: $P\ 2c\ -2n$

$a = 9.5934\ (3)\ \text{\AA}$

$b = 11.1939\ (4)\ \text{\AA}$

$c = 10.3332\ (3)\ \text{\AA}$

$V = 1109.66\ (6)\ \text{\AA}^3$

$Z = 4$

$F(000) = 480$

$D_x = 1.378\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

$\theta = 2.7\text{--}28.4^\circ$

$\mu = 0.10\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Block, light orange

$0.25 \times 0.20 \times 0.16\ \text{mm}$

Data collection

Bruker APEXII CCD area-detector diffractometer	3425 measured reflections 1014 independent reflections
Radiation source: fine-focus sealed tube	992 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{\text{int}} = 0.015$
Detector resolution: 8.33 pixels mm^{-1}	$\theta_{\text{max}} = 25.0^\circ$, $\theta_{\text{min}} = 2.7^\circ$
phi and ω scans	$h = -11 \rightarrow 11$
Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 2004)	$k = -13 \rightarrow 9$
$T_{\text{min}} = 0.975$, $T_{\text{max}} = 0.984$	$l = -10 \rightarrow 12$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.024$	$w = 1/[\sigma^2(F_o^2) + (0.0363P)^2 + 0.1774P]$
$wR(F^2) = 0.064$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\text{max}} < 0.001$
1014 reflections	$\Delta\rho_{\text{max}} = 0.13 \text{ e } \text{\AA}^{-3}$
163 parameters	$\Delta\rho_{\text{min}} = -0.10 \text{ e } \text{\AA}^{-3}$
3 restraints	Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001x\text{Fc}^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.046 (5)
Secondary atom site location: difference Fourier map	

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>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	1.10537 (16)	0.44042 (12)	0.85962 (16)	0.0487 (4)
O2	1.15292 (13)	0.82491 (12)	0.64988 (16)	0.0454 (4)
O3	0.79142 (15)	1.25293 (13)	0.43449 (17)	0.0509 (4)
N1	1.00354 (16)	0.64857 (13)	0.74734 (17)	0.0346 (4)
N2	0.94150 (15)	0.74797 (14)	0.69195 (18)	0.0357 (4)
C1	1.1354 (3)	0.3230 (2)	0.8813 (3)	0.0609 (7)
H1	1.2119	0.2956	0.9279	0.073*
C2	1.0401 (3)	0.2529 (2)	0.8265 (3)	0.0655 (7)
H2	1.0390	0.1698	0.8271	0.079*
C3	0.9410 (3)	0.32850 (19)	0.7673 (3)	0.0528 (6)
H3	0.8614	0.3054	0.7224	0.063*
C4	0.9853 (2)	0.44092 (17)	0.7889 (2)	0.0394 (5)
C5	0.9330 (2)	0.55207 (17)	0.7401 (2)	0.0389 (5)
H5	0.8452	0.5539	0.7019	0.047*

C6	1.02519 (18)	0.83589 (16)	0.64733 (19)	0.0332 (4)
C7	0.95845 (19)	0.94475 (16)	0.5952 (2)	0.0327 (4)
C8	0.81588 (19)	0.96915 (18)	0.6015 (2)	0.0422 (5)
H8	0.7566	0.9156	0.6428	0.051*
C9	0.7622 (2)	1.07164 (18)	0.5474 (2)	0.0464 (5)
H9	0.6670	1.0866	0.5525	0.056*
C10	0.8484 (2)	1.15259 (16)	0.48562 (19)	0.0363 (5)
C11	0.9907 (2)	1.13042 (17)	0.4805 (2)	0.0400 (5)
H11	1.0502	1.1850	0.4410	0.048*
C12	1.04335 (19)	1.02759 (17)	0.5340 (2)	0.0383 (5)
H12	1.1387	1.0131	0.5291	0.046*
H2'	0.8518 (11)	0.7474 (18)	0.676 (2)	0.041 (6)*
H3'	0.853 (2)	1.288 (2)	0.389 (2)	0.062 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0473 (8)	0.0475 (9)	0.0515 (9)	0.0037 (7)	-0.0047 (7)	0.0042 (8)
O2	0.0233 (6)	0.0449 (8)	0.0679 (10)	0.0008 (5)	-0.0012 (7)	0.0128 (8)
O3	0.0378 (8)	0.0491 (9)	0.0657 (11)	0.0131 (7)	0.0115 (9)	0.0196 (8)
N1	0.0280 (7)	0.0328 (8)	0.0428 (9)	0.0011 (6)	0.0003 (7)	0.0026 (7)
N2	0.0235 (7)	0.0349 (8)	0.0488 (10)	0.0005 (6)	-0.0024 (8)	0.0037 (7)
C1	0.0719 (16)	0.0551 (15)	0.0556 (14)	0.0205 (13)	0.0011 (14)	0.0138 (12)
C2	0.098 (2)	0.0364 (12)	0.0621 (15)	0.0059 (13)	0.0102 (16)	0.0116 (12)
C3	0.0609 (14)	0.0396 (11)	0.0578 (14)	-0.0100 (10)	0.0026 (12)	0.0031 (11)
C4	0.0360 (9)	0.0397 (10)	0.0426 (11)	-0.0017 (8)	0.0032 (9)	0.0006 (9)
C5	0.0302 (9)	0.0388 (10)	0.0478 (12)	-0.0020 (7)	-0.0008 (10)	-0.0004 (9)
C6	0.0261 (8)	0.0356 (9)	0.0380 (10)	-0.0006 (7)	-0.0014 (8)	-0.0012 (8)
C7	0.0271 (9)	0.0337 (9)	0.0372 (10)	-0.0006 (7)	-0.0001 (9)	-0.0020 (8)
C8	0.0283 (9)	0.0430 (10)	0.0554 (12)	0.0004 (8)	0.0087 (10)	0.0099 (11)
C9	0.0260 (9)	0.0519 (12)	0.0614 (13)	0.0090 (8)	0.0089 (11)	0.0101 (12)
C10	0.0328 (10)	0.0362 (10)	0.0399 (12)	0.0054 (8)	0.0017 (9)	0.0015 (9)
C11	0.0295 (10)	0.0416 (10)	0.0488 (13)	-0.0036 (8)	0.0037 (9)	0.0084 (10)
C12	0.0227 (9)	0.0419 (10)	0.0504 (13)	0.0010 (7)	0.0001 (9)	0.0044 (9)

Geometric parameters (Å, °)

O1—C4	1.364 (3)	C3—H3	0.9300
O1—C1	1.364 (3)	C4—C5	1.433 (3)
O2—C6	1.232 (2)	C5—H5	0.9300
O3—C10	1.356 (2)	C6—C7	1.478 (2)
O3—H3'	0.847 (10)	C7—C12	1.387 (2)
N1—C5	1.277 (2)	C7—C8	1.396 (3)
N1—N2	1.386 (2)	C8—C9	1.376 (3)
N2—C6	1.351 (2)	C8—H8	0.9300
N2—H2'	0.876 (10)	C9—C10	1.383 (3)
C1—C2	1.331 (4)	C9—H9	0.9300
C1—H1	0.9300	C10—C11	1.389 (3)

C2—C3	1.412 (4)	C11—C12	1.373 (3)
C2—H2	0.9300	C11—H11	0.9300
C3—C4	1.347 (3)	C12—H12	0.9300
C4—O1—C1	105.67 (19)	O2—C6—N2	120.73 (17)
C10—O3—H3'	108.7 (18)	O2—C6—C7	121.39 (16)
C5—N1—N2	115.31 (16)	N2—C6—C7	117.87 (15)
C6—N2—N1	118.08 (14)	C12—C7—C8	117.77 (17)
C6—N2—H2'	121.6 (14)	C12—C7—C6	117.59 (15)
N1—N2—H2'	119.6 (14)	C8—C7—C6	124.63 (17)
C2—C1—O1	110.7 (2)	C9—C8—C7	120.71 (19)
C2—C1—H1	124.7	C9—C8—H8	119.6
O1—C1—H1	124.7	C7—C8—H8	119.6
C1—C2—C3	107.1 (2)	C8—C9—C10	120.64 (17)
C1—C2—H2	126.5	C8—C9—H9	119.7
C3—C2—H2	126.5	C10—C9—H9	119.7
C4—C3—C2	106.0 (2)	O3—C10—C9	118.77 (17)
C4—C3—H3	127.0	O3—C10—C11	121.97 (18)
C2—C3—H3	127.0	C9—C10—C11	119.24 (18)
C3—C4—O1	110.59 (19)	C12—C11—C10	119.74 (18)
C3—C4—C5	129.9 (2)	C12—C11—H11	120.1
O1—C4—C5	119.19 (18)	C10—C11—H11	120.1
N1—C5—C4	121.89 (19)	C11—C12—C7	121.88 (16)
N1—C5—H5	119.1	C11—C12—H12	119.1
C4—C5—H5	119.1	C7—C12—H12	119.1
C5—N1—N2—C6	-152.8 (2)	N2—C6—C7—C12	-171.64 (19)
C4—O1—C1—C2	0.4 (3)	O2—C6—C7—C8	-172.9 (2)
O1—C1—C2—C3	-0.9 (3)	N2—C6—C7—C8	7.4 (3)
C1—C2—C3—C4	1.0 (3)	C12—C7—C8—C9	0.7 (3)
C2—C3—C4—O1	-0.8 (3)	C6—C7—C8—C9	-178.4 (2)
C2—C3—C4—C5	173.0 (2)	C7—C8—C9—C10	0.0 (4)
C1—O1—C4—C3	0.2 (3)	C8—C9—C10—O3	-179.5 (2)
C1—O1—C4—C5	-174.3 (2)	C8—C9—C10—C11	-1.1 (3)
N2—N1—C5—C4	177.85 (18)	O3—C10—C11—C12	179.84 (19)
C3—C4—C5—N1	-164.8 (3)	C9—C10—C11—C12	1.5 (3)
O1—C4—C5—N1	8.5 (3)	C10—C11—C12—C7	-0.8 (3)
N1—N2—C6—O2	3.7 (3)	C8—C7—C12—C11	-0.3 (3)
N1—N2—C6—C7	-176.57 (16)	C6—C7—C12—C11	178.9 (2)
O2—C6—C7—C12	8.1 (3)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2' \cdots O2 ⁱ	0.88 (1)	2.09 (1)	2.9187 (19)	157 (2)
O3—H3' \cdots N1 ⁱⁱ	0.85 (1)	2.13 (1)	2.971 (2)	169 (3)

C5—H5···O2 ⁱ	0.93	2.35	3.160 (2)	145
C11—H11···O3 ⁱⁱⁱ	0.93	2.42	3.202 (2)	142

Symmetry codes: (i) $x-1/2, -y+3/2, z$; (ii) $-x+2, -y+2, z-1/2$; (iii) $x+1/2, -y+5/2, z$.