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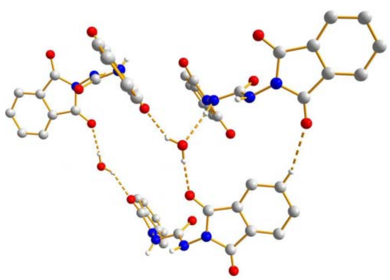


Crystal structure of 1,3-bis(1,3-dioxoisoindolin-1-yl)urea dihydrate: a urea-based anion receptor

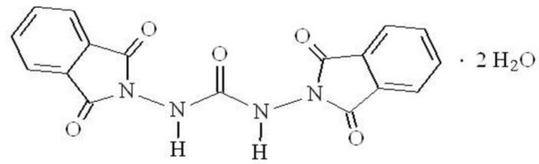
Felipe Medrano,* Sergio Lujano, Carolina Godoy-Alcántar and Hugo Tlahuext

Centro de Investigaciones Químicas, Universidad Autónoma del Estado de Morelos, Av. Universidad 1001 Col., Chamilpa, CP 62209, Cuernavaca, Mexico. *Correspondence e-mail: fmedrano@uaem.mx

The whole molecule of the title compound, $C_{17}H_{10}N_4O_5{\cdot}2H_2O$, is generated by twofold rotation symmetry and it crystallized as a dihydrate. The planes of the phthalimide moieties and the urea unit are almost normal to one another, with a dihedral angle of 78.62 (9)°. In the crystal, molecules are linked by N—H⋯O and O—H⋯O hydrogen bonds, forming a three-dimensional framework structure. The crystal packing also features C—H⋯O hydrogen bonds and slipped parallel π–π interactions [centroid–centroid distance = 3.6746 (15) Å] involving the benzene rings of neighbouring phthalimide moieties.

1. Chemical context

Hydrogen bonding and π–π interactions are two of the principal forces which determine structure, self-assembly and recognition in some chemical and biological systems (Lehn, 1990). A variety of urea-based anion receptors of varying complexity and sophistication have been synthesised (Amendola *et al.*, 2010). It has been shown that the efficiency of urea as a receptor subunit depends on the presence of two proximate polarised N—H fragments, capable of (i) chelating a spherical anion or (ii) donating two parallel hydrogen bonds to the O atoms of a carboxylate or of an inorganic oxoanion. A review of the biological activity of phthalimides has been published by Sharma *et al.* (2010) and a review of its the supramolecular chemistry by Barooah & Baruah (2007). Phthalimides and isoindolines have been shown to possess photophysical properties and have applications as colourimetric and other types of anion sensors (Griesbeck & Schieffer, 2003; Griesbeck *et al.*, 2007, 2010; Devaraj & Kandaswamy, 2013). In our ongoing research on 1,3-dioxoisoindolines as anion receptors (Lujano, 2012), we report herein on the synthesis and crystal structure of the title urea-based anion receptor.



2. Structural commentary

The molecular structure of the title compound is illustrated in Fig. 1. The molecule is located on a crystallographic twofold rotation axis that bisects the central C9═O3 bond. The planes of the phthalimide unit (N1/C1–C8) and the urea unit [N2—

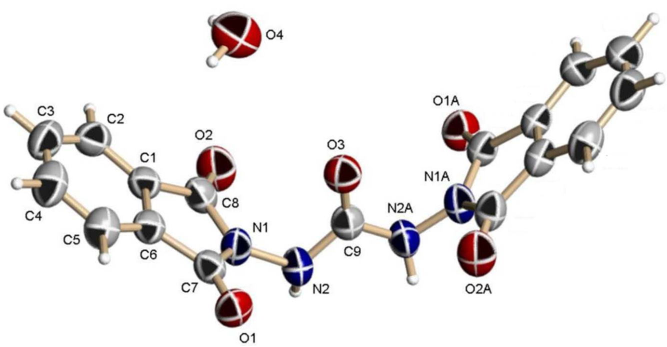


Figure 1
The molecular structure of the title molecule, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level. Atoms with the suffix A are generated by the symmetry operator ($-x$, y, $-z + \frac{1}{2}$) and the symmetry-related water molecule is not shown.

C9(=O3)—N2] are almost normal to one another, with a dihedral angle of 78.62 (9)°. The planes of the symmetry-related phthalimide moieties [N1/C1–C8 and N1^{i}/C1^{i}–C8^{i}; symmetry code: (i) $-x$, y, $-z + \frac{1}{2}$] are inclined to one another by 73.53 (7)°.

3. Supramolecular features

In the crystal, molecules are linked by N—H···O and O—H···O hydrogen bonds, forming a three-dimensional framework structure (Table 1 and Fig. 2). The solvent water molecules, which occupy general positions, take part in the hydrogen-bonding network (Table 1 and Figs. 2 and 3). The O atom of the water molecules, O4, is an acceptor of one H atom and simultaneously a donor of their two H atoms and enclose $R_4^4(24)$ and $R_3^3(15)$ ring motifs (Table 1 and Fig. 3). The crystal packing is reinforced by C—H···O hydrogen bonds, and slipped parallel π–π interactions (Fig. 4) involving benzene rings of neighbouring phthalimide moieties [$Cg\cdots Cg^{i}$ = 3.6746 (15) Å; normal distance = 3.3931 (9) Å; slippage = 1.411 Å; *Cg* is the centroid of the C1–C6 ring; symmetry code: (i) $-x + \frac{3}{2}$, $-y + \frac{1}{2}$, $-z + 2$].

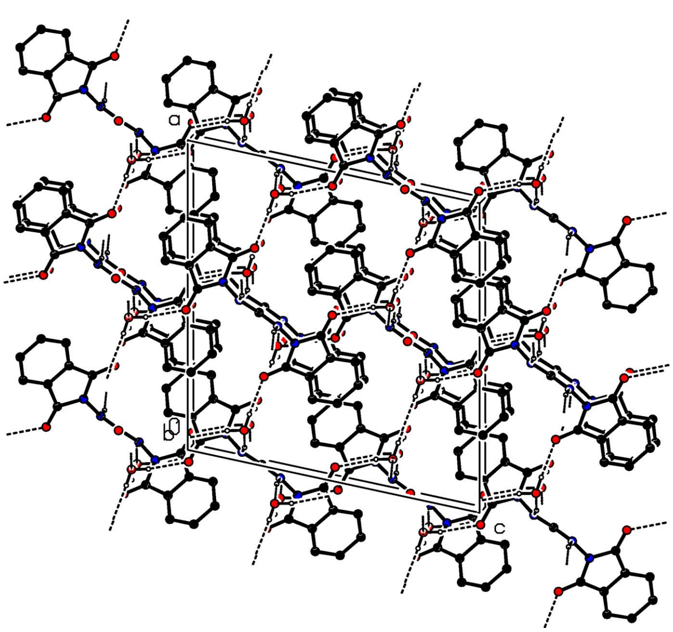


Figure 2
A view along the *b* axis of the crystal packing of the title compound. Hydrogen bonds are shown as dashed lines (see Table 1 for details. C-bound H atoms have been omitted for clarity.

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O4^{i}	0.87 (2)	1.96 (2)	2.811 (3)	167 (2)
O4—H4A···O1^{ii}	0.85 (1)	2.11 (1)	2.891 (3)	154 (3)
O4—H4B···O2^{iii}	0.85 (2)	2.01 (2)	2.850 (3)	175 (3)
C3—H3···O1^{iv}	0.93	2.56	3.447 (3)	160

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

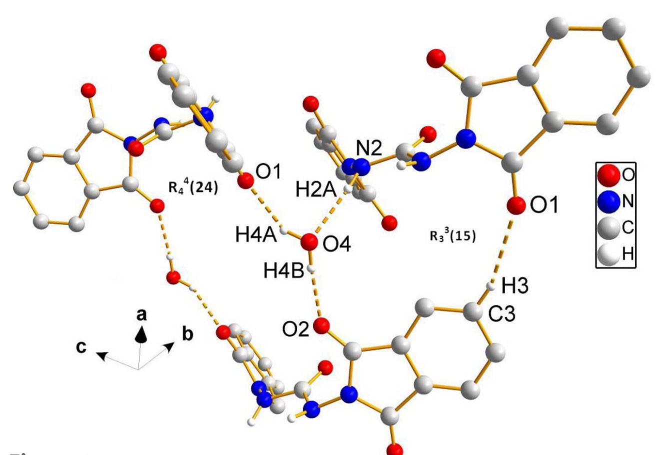


Figure 3
A view of the crystal packing of the title compound. The hydrogen bonds (dashed lines; see Table 1 for details) enclose $R_4^4(24)$ and $R_3^3(15)$ ring motifs.

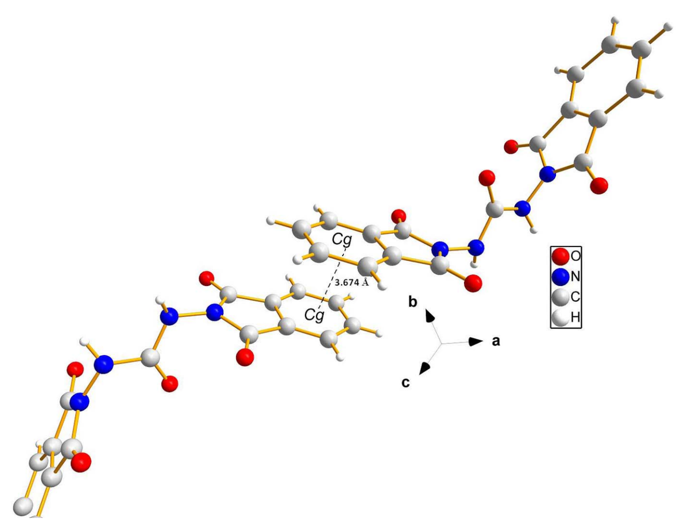


Figure 4
Two molecules of the title compound showing the offset π–π interactions involving the benzene rings of neighbouring phthalimide moieties (dashed line).

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{10}N_4O_5\cdot 2H_2O$
M_r	386.32
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	293
a, *b*, *c* (Å)	15.268 (3), 7.8053 (16), 14.729 (3)
β (°)	102.097 (3)
V ($Å^3$)	1716.3 (6)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	0.12
Crystal size (mm)	0.40 × 0.32 × 0.23
Data collection	
Diffractometer	Bruker SMART CCD area detector
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2003)
T_{min}, T_{max}	0.954, 0.973
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7038, 1529, 1414
R_{int}	0.035
$(\sin \theta/\lambda)_{max}$ ($Å^{-1}$)	0.597
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.052, 0.130, 1.12
No. of reflections	1529
No. of parameters	141
No. of restraints	4
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e $Å^{-3}$)	0.37, −0.25

Computer programs: *SMART* and *SAINT-Plus* (Bruker, 2001), *SHELXS97*, *SHELXL97* and *SHELXTL-NT* (Sheldrick, 2008), *DIAMOND* (Brandenburg, 1997), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

4. Synthesis and crystallization

Carbohydrazide (0.5 g, 5.5 mmol) and phthalic anhydride (1.64 g, 11 mmol) were dissolved in dimethyl sulfoxide (15 ml) and refluxed for 6 h at 323 K. The solvent was removed under reduced pressure in a rotatory evaporator and the pale-yellow solid residue was washed with water and dried under vacuum. The product was recrystallized from water/ethanol (30:70 *v*/*v*) to give colourless prismatic crystals suitable for X-ray diffraction analysis (m.p. 491–493 K). ^{1}H NMR (200 MHz, DMSO-d_6, Me_4Si): δ 9.25 (2H, N—H), 7.80 (8H, Ar). ^{13}C NMR (50 MHz, DMSO-d_6, Me_4Si): δ 165.2 (C7, C8, C7′, C8′), 154.7 (C9), 135.0 (C5, C2, C5′, C2′), 129.4 (C1, C6, C1′, C6′), 123.5 (C3, C4, C3′, C4′). MS (FAB$^+$): *m*/*z* (%) 349 (*M*—H, 25).

5. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 2. The NH group and water molecule H atoms were located in a difference Fourier map and refined with distance restraints N—H = 0.86 (1) Å and O—H = 0.84 (1) Å, and with $U_{iso}(H) = 1.2U_{eq}(N)$ and $1.5U_{eq}(O)$. C-bound H atoms were positioned geometrically and constrained using a riding-model approximation, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Acknowledgements

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Crystal structure of 1,3-bis(1,3-dioxoisoindolin-1-yl)urea dihydrate: a urea-based anion receptor

Felipe Medrano, Sergio Lujano, Carolina Godoy-Alcántar and Hugo Tlahuext

Computing details

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL-NT* (Sheldrick, 2008), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

1,3-Bis(1,3-dioxoisoindolin-1-yl)urea dihydrate

Crystal data

$C_{17}H_{10}N_4O_5 \cdot 2H_2O$
$M_r = 386.32$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 15.268$ (3) Å
$b = 7.8053$ (16) Å
$c = 14.729$ (3) Å
$\beta = 102.097$ (3)°
$V = 1716.3$ (6) Å^3
$Z = 4$
$F(000) = 800$
$D_x = 1.495$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5032 reflections
$\theta = 2.6$–28.1°
$\mu = 0.12$ mm^{-1}
$T = 293$ K
Prism, colourless
0.40 × 0.32 × 0.23 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3 pixels mm^{-1}
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.954$, $T_{max} = 0.973$
7038 measured reflections
1529 independent reflections
1414 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 25.1°$, $\theta_{min} = 2.7°$
$h = -17 \rightarrow 18$
$k = -9 \rightarrow 9$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.130$
$S = 1.12$
1529 reflections
141 parameters
4 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
$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 1.5592P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ 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}
O1	1.04237 (9)	0.1919 (2)	1.00070 (10)	0.0522 (4)
O2	0.79568 (10)	0.2183 (3)	0.76586 (10)	0.0646 (5)
O3	1.0000	0.3375 (3)	0.7500	0.0527 (6)
O4	0.89221 (13)	0.7577 (3)	0.80226 (14)	0.0780 (6)
N1	0.92631 (11)	0.1729 (2)	0.87283 (11)	0.0453 (5)
N2	0.97120 (12)	0.0845 (2)	0.81541 (12)	0.0477 (5)
C1	0.82495 (13)	0.3490 (3)	0.91942 (14)	0.0424 (5)
C2	0.75114 (14)	0.4447 (3)	0.92811 (17)	0.0528 (6)
H2	0.7014	0.4533	0.8795	0.063*
C3	0.75383 (16)	0.5273 (3)	1.01160 (18)	0.0589 (6)
H3	0.7050	0.5926	1.0193	0.071*
C4	0.82743 (16)	0.5149 (3)	1.08370 (19)	0.0611 (6)
H4	0.8270	0.5716	1.1392	0.073*
C5	0.90231 (15)	0.4194 (3)	1.07526 (16)	0.0515 (6)
C6	0.89956 (12)	0.3377 (2)	0.99210 (13)	0.0398 (5)
C7	0.96745 (13)	0.2286 (3)	0.96152 (13)	0.0392 (5)
C8	0.84162 (14)	0.2441 (3)	0.84167 (14)	0.0458 (5)
C9	1.0000	0.1824 (4)	0.7500	0.0416 (7)
H5	0.9532 (16)	0.412 (3)	1.1262 (17)	0.057 (6)*
H2A	0.9521 (17)	−0.0195 (17)	0.8048 (18)	0.068*
H4B	0.8366 (8)	0.741 (4)	0.784 (2)	0.085*
H4A	0.905 (2)	0.740 (4)	0.8601 (8)	0.085*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0329 (8)	0.0684 (10)	0.0520 (9)	0.0035 (7)	0.0018 (6)	0.0030 (7)
O2	0.0456 (9)	0.1021 (14)	0.0418 (9)	−0.0006 (9)	−0.0005 (7)	0.0028 (8)
O3	0.0514 (13)	0.0556 (14)	0.0510 (12)	0.000	0.0105 (10)	0.000
O4	0.0571 (11)	0.0918 (14)	0.0762 (13)	−0.0158 (10)	−0.0063 (10)	0.0090 (11)
N1	0.0363 (9)	0.0633 (11)	0.0374 (9)	0.0055 (8)	0.0104 (7)	0.0008 (8)

N2	0.0495 (10)	0.0558 (11)	0.0418 (9)	−0.0005 (9)	0.0185 (8)	−0.0018 (8)
C1	0.0345 (10)	0.0471 (11)	0.0467 (11)	−0.0009 (9)	0.0108 (8)	0.0104 (9)
C2	0.0372 (11)	0.0571 (13)	0.0648 (14)	0.0039 (10)	0.0122 (10)	0.0169 (11)
C3	0.0480 (13)	0.0474 (13)	0.0889 (18)	0.0033 (10)	0.0314 (13)	0.0017 (12)
C4	0.0578 (15)	0.0566 (14)	0.0747 (15)	−0.0090 (11)	0.0273 (12)	−0.0181 (12)
C5	0.0443 (12)	0.0560 (13)	0.0548 (13)	−0.0088 (10)	0.0117 (10)	−0.0102 (10)
C6	0.0323 (10)	0.0427 (10)	0.0452 (11)	−0.0050 (8)	0.0100 (8)	0.0044 (8)
C7	0.0325 (10)	0.0469 (11)	0.0379 (10)	−0.0038 (8)	0.0069 (8)	0.0056 (8)
C8	0.0358 (11)	0.0636 (13)	0.0376 (11)	−0.0025 (9)	0.0070 (9)	0.0094 (9)
C9	0.0323 (14)	0.0539 (18)	0.0371 (14)	0.000	0.0039 (11)	0.000

Geometric parameters (Å, º)

O1—C7	1.203 (2)	C1—C8	1.472 (3)
O2—C8	1.205 (2)	C2—C3	1.381 (3)
O3—C9	1.210 (4)	C2—H2	0.9300
O4—H4B	0.846 (10)	C3—C4	1.378 (3)
O4—H4A	0.844 (10)	C3—H3	0.9300
N1—N2	1.380 (2)	C4—C5	1.392 (3)
N1—C8	1.394 (3)	C4—H4	0.9300
N1—C7	1.395 (3)	C5—C6	1.374 (3)
N2—C9	1.373 (2)	C5—H5	0.96 (2)
N2—H2A	0.865 (10)	C6—C7	1.483 (3)
C1—C2	1.380 (3)	C9—N2^{i}	1.373 (2)
C1—C6	1.393 (3)		
H4B—O4—H4A	108 (3)	C3—C4—H4	119.3
N2—N1—C8	122.91 (16)	C5—C4—H4	119.3
N2—N1—C7	123.07 (16)	C6—C5—C4	117.1 (2)
C8—N1—C7	112.88 (17)	C6—C5—H5	122.4 (14)
C9—N2—N1	115.14 (19)	C4—C5—H5	120.5 (14)
C9—N2—H2A	122.8 (18)	C5—C6—C1	121.5 (2)
N1—N2—H2A	112.8 (18)	C5—C6—C7	130.16 (19)
C2—C1—C6	121.0 (2)	C1—C6—C7	108.31 (17)
C2—C1—C8	130.59 (19)	O1—C7—N1	124.85 (19)
C6—C1—C8	108.40 (17)	O1—C7—C6	130.25 (19)
C1—C2—C3	117.6 (2)	N1—C7—C6	104.90 (16)
C1—C2—H2	121.2	O2—C8—N1	123.9 (2)
C3—C2—H2	121.2	O2—C8—C1	130.7 (2)
C4—C3—C2	121.4 (2)	N1—C8—C1	105.38 (16)
C4—C3—H3	119.3	O3—C9—N2^{i}	123.86 (13)
C2—C3—H3	119.3	O3—C9—N2	123.86 (13)
C3—C4—C5	121.4 (2)	N2^{i}—C9—N2	112.3 (3)
C8—N1—N2—C9	69.0 (2)	C8—N1—C7—C6	3.9 (2)
C7—N1—N2—C9	−97.9 (2)	C5—C6—C7—O1	−3.6 (4)
C6—C1—C2—C3	−0.5 (3)	C1—C6—C7—O1	176.4 (2)
C8—C1—C2—C3	178.3 (2)	C5—C6—C7—N1	176.7 (2)

C1—C2—C3—C4	0.0 (3)	C1—C6—C7—N1	−3.3 (2)
C2—C3—C4—C5	0.3 (4)	N2—N1—C8—O2	9.2 (3)
C3—C4—C5—C6	−0.2 (3)	C7—N1—C8—O2	177.3 (2)
C4—C5—C6—C1	−0.3 (3)	N2—N1—C8—C1	−171.03 (18)
C4—C5—C6—C7	179.7 (2)	C7—N1—C8—C1	−2.9 (2)
C2—C1—C6—C5	0.7 (3)	C2—C1—C8—O2	1.4 (4)
C8—C1—C6—C5	−178.36 (19)	C6—C1—C8—O2	−179.6 (2)
C2—C1—C6—C7	−179.32 (18)	C2—C1—C8—N1	−178.3 (2)
C8—C1—C6—C7	1.6 (2)	C6—C1—C8—N1	0.7 (2)
N2—N1—C7—O1	−7.7 (3)	N1—N2—C9—O3	11.43 (18)
C8—N1—C7—O1	−175.81 (19)	N1—N2—C9—N2^{i}	−168.57 (18)
N2—N1—C7—C6	171.95 (17)		

Symmetry code: (i) −*x*+2, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O4^{ii}	0.87 (2)	1.96 (2)	2.811 (3)	167 (2)
O4—H4*A*···O1^{iii}	0.85 (1)	2.11 (1)	2.891 (3)	154 (3)
O4—H4*B*···O2^{iv}	0.85 (2)	2.01 (2)	2.850 (3)	175 (3)
C3—H3···O1^{v}	0.93	2.56	3.447 (3)	160

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