3-(2,4-Dichloroanilino)isobenzofuran-1(3H)-one[1]

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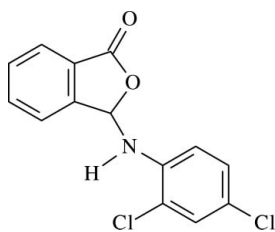Correspondence e-mail: orhanb@omu.edu.tr

In the molecule of the title compound, $C_{14}H_9Cl_2NO_2$, the essentially planar phthalide group is oriented at a dihedral angle of 63.23 (5)° with respect to the substituted aromatic ring. In the crystal structure, intermolecular C—H···O and N—H···O hydrogen bonds link the molecules, generating $R_4^4(21)$ ring motifs to form a three-dimensional network.

Related literature

For general background, see: Aoki *et al.* (1973, 1974); Tsi & Tan (1997); Roy & Sarkar (2005); Bellasio (1974, 1975). For related structures, see: Büyükgüngör & Odabaşoğlu (2006); Odabaşoğlu & Büyükgüngör (2006). For ring motif details, see: Bernstein *et al.* (1995); Etter (1990).

Experimental

Crystal data

$C_{14}H_9Cl_2NO_2$	V = 1327.56 (12) Å³
M_r = 294.12	Z = 4
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 7.7647 (4) Å	μ = 0.48 mm⁻¹
b = 23.9293 (12) Å	T = 296 K
c = 7.3261 (4) Å	0.62 × 0.40 × 0.29 mm
β = 102.768 (5)°	

Data collection

Stoe IPDSII diffractometer	7695 measured reflections
Absorption correction: integration	2500 independent reflections
(*X-RED32*; Stoe & Cie, 2002)	1860 reflections with $I > 2\sigma(I)$
T_{min} = 0.486, T_{max} = 0.854	R_{int} = 0.114

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048	H atoms treated by a mixture of
$wR(F^2)$ = 0.113	independent and constrained
S = 1.03	refinement
2500 reflections	$\Delta\rho_{max}$ = 0.39 e Å⁻³
176 parameters	$\Delta\rho_{min}$ = −0.38 e Å⁻³

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1ⁱ	0.86 (3)	2.36 (3)	3.176 (2)	159 (2)
C4—H4···O2ⁱⁱ	0.93	2.57	3.387 (3)	147

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); 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); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

References

Aoki, K., Furusho, T., Kimura, T., Satake, K. & Funayama, S. (1973). Jpn Patent No. 7 324 724.
Aoki, K., Furusho, T., Kimura, T., Satake, K. & Funayama, S. (1974). *Chem. Abstr.* **80**, 129246.
Bellasio, E. (1974). German Patent No. 2 422 193.
Bellasio, E. (1975). *Chem. Abstr.* **83**, 9788.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Büyükgüngör, O. & Odabaşoğlu, M. (2006). *Acta Cryst.* E**62**, o2003–o2004.
Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Odabaşoğlu, M. & Büyükgüngör, O. (2006). *Acta Cryst.* E**62**, o1879–o1881.
Roy, H. N. & Sarkar, M. S. (2005). *Synth. Commun.* **35**, 2177–2181.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Stoe & Cie (2002). *X-AREA* and *X-RED32* . Stoe & Cie, Darmstadt, Germany.
Tsi, D. & Tan, B. K. H. (1997). *Phytother. Res.* **11**, 576–582.

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[1] 3-Substituted phthalides. Part XXXV.

supporting information

3-(2,4-Dichloroanilino)isobenzofuran-1(3*H*)-one

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S1. Comment

Phthalides (isobenzofuranones) are five-membered lactones found in plants and they are known to show diverse biological activities, such as fungicidal, bactericidal, herbicidal, analgesic, pesticidal, hypotensive and vasorelaxant activities (Aoki *et al.*, 1973, 1974; Tsi & Tan, 1997; Roy & Sarkar, 2005). In addition, phthalide derivatives are useful in the treatment of circulatory and heart-related diseases (Bellasio, 1974, 1975). As part of our ongoing research on 3-substituted phthalides (Büyükgüngör & Odabaşoğlu, 2006; Odabaşoğlu & Büyükgüngör, 2006), the title compound, (I), has been synthesized and its crystal structure is reported here.

In the molecule of (I), (Fig. 1), rings A (C2–C7), B (C1/C2/C7/C8/O2) and C (C9–C14) are, of course, planar. The dihedral angles between them are A/B = 2.60 (5)°, A/C = 63.44 (4)° and B/C = 63.11 (5)°. So, rings A and B are also nearly coplanar. Ring C is oriented with respect to the coplanar ring system at a dihedral angle of 63.23 (5)°. The geometry of (I) does not show any significant difference from the average geometry found for 3-(4-chloro-anilino)isobenzofuran-1(3*H*)-one (Büyükgüngör & Odabaşoğlu, 2006).

In the crystal structure, intermolecular C-H···O and N-H···O hydrogen bonds (Table 1) link the molecules, generating $R_4^4(21)$ (Fig. 2) ring motifs (Bernstein *et al.*, 1995; Etter, 1990), to form a three-dimensional network, in which they may be effective in the stabilization of the structure.

S2. Experimental

The title compound was prepared according to the method described by Odabaşoğlu & Büyükgüngör (2006), using phthalaldehydic acid and 2,4-dichloroaniline as starting materials (yield; 80%). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol-DMF (1:1) solution at room temperature.

S3. Refinement

H atom (for NH) was located in difference synthesis and refined freely [N-H = 0.86 (3) Å and $U_{iso}(H) = 0.069 (7)$ Å^2]. The remaining H atoms were positioned geometrically, with C-H = 0.93 and 0.98 Å for aromatic and methine H, respectively, and constrained to ride on their parent atoms with $U_{iso}(H) = 1.2U_{eq}(C)$.
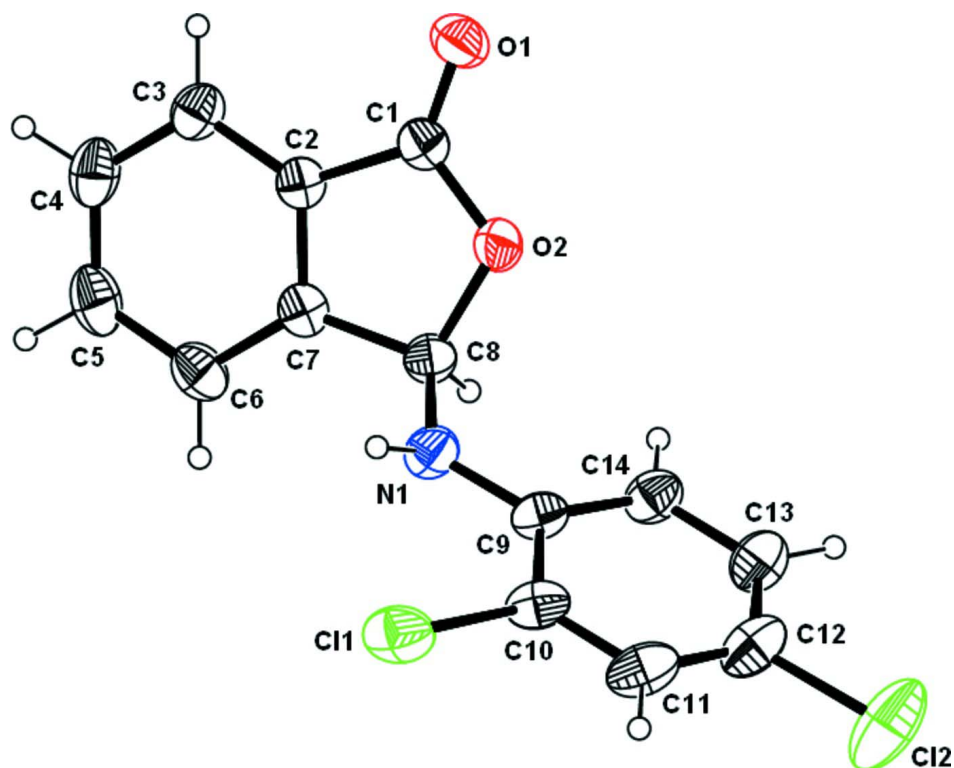
Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.
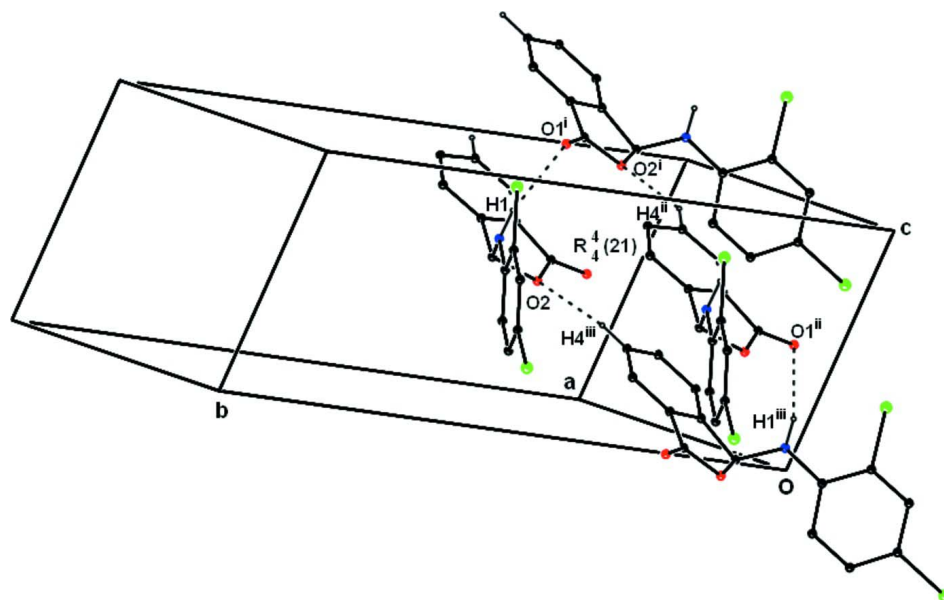

Figure 2

A partial packing diagram of (I), showing the formation of $R_4^4(21)$ ring motif. Hydrogen bonds are shown as dashed lines [symmetry codes: (i) x, 1/2 - y, z + 1/2; (ii) x - 1, y, z; (iii) x - 1, 1/2 - y, 1/2 - z]. H atoms not involved in hydrogen bondings have been omitted for clarity.

3-(2,4-dichloroanilino)isobenzofuran-1(3H)-one

Crystal data

$C_{14}H_9Cl_2NO_2$	$F(000) = 600$
$M_r = 294.12$	$D_x = 1.472$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 7695 reflections
$a = 7.7647$ (4) Å	$\theta = 1.7$–26.1°
$b = 23.9293$ (12) Å	$\mu = 0.48$ mm^{-1}
$c = 7.3261$ (4) Å	$T = 296$ K
$\beta = 102.768$ (5)°	Prism, colorless
$V = 1327.56$ (12) Å^3	$0.62 \times 0.40 \times 0.29$ mm
$Z = 4$	

Data collection

Stoe IPDS II diffractometer	$T_{min} = 0.486$, $T_{max} = 0.854$
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus	7695 measured reflections
	2500 independent reflections
Plane graphite monochromator	1860 reflections with $I > 2\sigma(I)$
Detector resolution: 6.67 pixels mm^{-1}	$R_{int} = 0.114$
w–scan rotation method	$\theta_{max} = 25.7°$, $\theta_{min} = 1.7°$
Absorption correction: integration	$h = -9 \rightarrow 9$
(*X-RED32*; Stoe & Cie, 2002)	$k = -28 \rightarrow 28$
	$l = -8 \rightarrow 8$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.048$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.113$	
$S = 1.03$	H atoms treated by a mixture of independent and constrained refinement
2500 reflections	
176 parameters	$w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 0.1182P]$
0 restraints	where $P = (F_o^2 + 2F_c^2)/3$
Primary atom site location: structure-invariant direct methods	$(\Delta/\sigma)_{max} < 0.001$
	$\Delta\rho_{max} = 0.39$ e Å^{-3}
	$\Delta\rho_{min} = -0.38$ 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 > 2sigma(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}
Cl1	0.54932 (11)	0.44014 (3)	0.87525 (9)	0.0799 (3)
Cl2	0.05955 (13)	0.48298 (5)	0.25003 (14)	0.1281 (5)
O1	0.7384 (2)	0.18458 (7)	0.5396 (2)	0.0666 (4)

O2	0.69942 (18)	0.27650 (6)	0.4919 (2)	0.0552 (4)
H1	0.719 (3)	0.3623 (11)	0.752 (4)	0.069 (7)*
N1	0.7106 (3)	0.36695 (8)	0.6346 (3)	0.0570 (5)
C1	0.7958 (3)	0.23169 (9)	0.5653 (3)	0.0502 (5)
C2	0.9681 (3)	0.25091 (9)	0.6711 (3)	0.0468 (5)
C3	1.1115 (3)	0.22004 (10)	0.7623 (3)	0.0574 (6)
H3	1.1072	0.1812	0.7669	0.069*
C4	1.2615 (3)	0.24907 (13)	0.8465 (3)	0.0685 (7)
H4	1.3608	0.2296	0.9088	0.082*
C5	1.2657 (3)	0.30675 (14)	0.8391 (3)	0.0722 (7)
H5	1.3685	0.3253	0.8971	0.087*
C6	1.1218 (3)	0.33757 (11)	0.7482 (3)	0.0643 (6)
H6	1.1255	0.3764	0.7449	0.077*
C7	0.9719 (3)	0.30836 (9)	0.6624 (3)	0.0497 (5)
C8	0.8012 (3)	0.32888 (9)	0.5450 (3)	0.0506 (5)
H8	0.8241	0.3464	0.4318	0.061*
C9	0.5585 (3)	0.39413 (9)	0.5407 (3)	0.0518 (5)
C10	0.4694 (3)	0.43014 (9)	0.6373 (3)	0.0583 (6)
C11	0.3179 (3)	0.45773 (11)	0.5500 (4)	0.0716 (7)
H11	0.2602	0.4814	0.6177	0.086*
C12	0.2532 (3)	0.44977 (12)	0.3615 (4)	0.0730 (7)
C13	0.3382 (3)	0.41567 (11)	0.2614 (3)	0.0685 (6)
H13	0.2942	0.4110	0.1336	0.082*
C14	0.4896 (3)	0.38808 (10)	0.3495 (3)	0.0597 (6)
H14	0.5468	0.3649	0.2799	0.072*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1151 (6)	0.0677 (4)	0.0658 (4)	0.0114 (4)	0.0389 (4)	−0.0076 (3)
Cl2	0.0912 (6)	0.1702 (11)	0.1280 (7)	0.0701 (6)	0.0349 (5)	0.0507 (7)
O1	0.0662 (10)	0.0565 (10)	0.0751 (10)	−0.0104 (8)	0.0116 (8)	−0.0096 (8)
O2	0.0437 (8)	0.0569 (9)	0.0613 (8)	0.0026 (7)	0.0036 (6)	−0.0052 (7)
N1	0.0652 (12)	0.0584 (11)	0.0487 (10)	0.0134 (9)	0.0153 (9)	−0.0014 (8)
C1	0.0471 (11)	0.0545 (13)	0.0509 (10)	−0.0004 (10)	0.0147 (9)	−0.0061 (9)
C2	0.0419 (10)	0.0541 (12)	0.0470 (10)	0.0009 (10)	0.0153 (8)	−0.0022 (8)
C3	0.0503 (12)	0.0652 (14)	0.0587 (12)	0.0101 (11)	0.0165 (10)	0.0023 (10)
C4	0.0434 (12)	0.098 (2)	0.0626 (13)	0.0103 (14)	0.0089 (10)	0.0015 (13)
C5	0.0426 (12)	0.103 (2)	0.0696 (14)	−0.0124 (14)	0.0087 (11)	−0.0137 (14)
C6	0.0552 (13)	0.0676 (15)	0.0710 (14)	−0.0150 (12)	0.0160 (11)	−0.0097 (11)
C7	0.0451 (11)	0.0574 (13)	0.0487 (10)	−0.0020 (10)	0.0152 (8)	−0.0032 (9)
C8	0.0511 (12)	0.0495 (12)	0.0527 (11)	0.0022 (10)	0.0147 (9)	−0.0008 (9)
C9	0.0580 (13)	0.0448 (11)	0.0569 (12)	0.0047 (10)	0.0219 (10)	0.0056 (9)
C10	0.0724 (15)	0.0471 (12)	0.0641 (13)	0.0061 (11)	0.0338 (12)	0.0062 (10)
C11	0.0778 (17)	0.0614 (14)	0.0886 (17)	0.0191 (14)	0.0463 (15)	0.0147 (13)
C12	0.0648 (15)	0.0764 (17)	0.0840 (17)	0.0203 (13)	0.0297 (14)	0.0266 (13)
C13	0.0671 (15)	0.0741 (16)	0.0656 (13)	0.0081 (13)	0.0175 (12)	0.0144 (12)
C14	0.0647 (14)	0.0591 (14)	0.0589 (12)	0.0133 (11)	0.0212 (11)	0.0046 (10)

Geometric parameters (Å, º)

C1—O1	1.212 (3)	C8—O2	1.487 (3)
C1—O2	1.349 (3)	C8—H8	0.9800
C1—C2	1.465 (3)	C9—N1	1.390 (3)
C2—C7	1.377 (3)	C9—C14	1.392 (3)
C2—C3	1.379 (3)	C9—C10	1.392 (3)
C3—C4	1.379 (3)	C10—C11	1.376 (3)
C3—H3	0.9300	C10—Cl1	1.733 (2)
C4—C5	1.382 (4)	C11—C12	1.375 (4)
C4—H4	0.9300	C11—H11	0.9300
C5—C6	1.381 (4)	C12—C13	1.362 (4)
C5—H5	0.9300	C12—Cl2	1.737 (3)
C6—C7	1.384 (3)	C13—C14	1.378 (3)
C6—H6	0.9300	C13—H13	0.9300
C7—C8	1.494 (3)	C14—H14	0.9300
C8—N1	1.400 (3)	N1—H1	0.86 (3)
O1—C1—O2	121.7 (2)	O2—C8—H8	108.9
O1—C1—C2	129.5 (2)	C7—C8—H8	108.9
O2—C1—C2	108.85 (18)	N1—C9—C14	123.02 (19)
C7—C2—C3	122.3 (2)	N1—C9—C10	120.1 (2)
C7—C2—C1	108.35 (18)	C14—C9—C10	116.9 (2)
C3—C2—C1	129.3 (2)	C11—C10—C9	122.0 (2)
C4—C3—C2	117.3 (2)	C11—C10—Cl1	118.71 (17)
C4—C3—H3	121.4	C9—C10—Cl1	119.26 (18)
C2—C3—H3	121.4	C12—C11—C10	119.0 (2)
C3—C4—C5	120.7 (2)	C12—C11—H11	120.5
C3—C4—H4	119.6	C10—C11—H11	120.5
C5—C4—H4	119.6	C13—C12—C11	120.7 (2)
C6—C5—C4	121.9 (2)	C13—C12—Cl2	119.6 (2)
C6—C5—H5	119.1	C11—C12—Cl2	119.7 (2)
C4—C5—H5	119.1	C12—C13—C14	120.0 (2)
C5—C6—C7	117.3 (2)	C12—C13—H13	120.0
C5—C6—H6	121.3	C14—C13—H13	120.0
C7—C6—H6	121.3	C13—C14—C9	121.3 (2)
C2—C7—C6	120.5 (2)	C13—C14—H14	119.3
C2—C7—C8	109.26 (18)	C9—C14—H14	119.3
C6—C7—C8	130.2 (2)	C9—N1—C8	122.17 (18)
N1—C8—O2	112.22 (16)	C9—N1—H1	115.0 (17)
N1—C8—C7	114.69 (17)	C8—N1—H1	117.1 (17)
O2—C8—C7	103.07 (16)	C1—O2—C8	110.45 (15)
N1—C8—H8	108.9		
O1—C1—C2—C7	−179.3 (2)	C14—C9—C10—C11	1.4 (3)
O2—C1—C2—C7	1.0 (2)	N1—C9—C10—Cl1	−0.5 (3)
O1—C1—C2—C3	2.9 (3)	C14—C9—C10—Cl1	−179.37 (17)
O2—C1—C2—C3	−176.77 (17)	C9—C10—C11—C12	−0.4 (4)

C7—C2—C3—C4	0.2 (3)	Cl1—C10—C11—C12	−179.66 (19)
C1—C2—C3—C4	177.70 (18)	C10—C11—C12—C13	−0.8 (4)
C2—C3—C4—C5	0.2 (3)	C10—C11—C12—Cl2	178.56 (19)
C3—C4—C5—C6	0.0 (4)	C11—C12—C13—C14	0.9 (4)
C4—C5—C6—C7	−0.5 (3)	Cl2—C12—C13—C14	−178.4 (2)
C3—C2—C7—C6	−0.8 (3)	C12—C13—C14—C9	0.1 (4)
C1—C2—C7—C6	−178.75 (17)	N1—C9—C14—C13	179.9 (2)
C3—C2—C7—C8	176.66 (16)	C10—C9—C14—C13	−1.2 (3)
C1—C2—C7—C8	−1.3 (2)	C14—C9—N1—C8	−3.5 (3)
C5—C6—C7—C2	0.9 (3)	C10—C9—N1—C8	177.7 (2)
C5—C6—C7—C8	−175.9 (2)	O2—C8—N1—C9	−69.0 (2)
C2—C7—C8—N1	123.40 (19)	C7—C8—N1—C9	173.83 (19)
C6—C7—C8—N1	−59.5 (3)	O1—C1—O2—C8	179.98 (18)
C2—C7—C8—O2	1.11 (19)	C2—C1—O2—C8	−0.3 (2)
C6—C7—C8—O2	178.20 (19)	N1—C8—O2—C1	−124.39 (18)
N1—C9—C10—C11	−179.7 (2)	C7—C8—O2—C1	−0.46 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1[i]	0.86 (3)	2.36 (3)	3.176 (2)	159 (2)
C4—H4···O2[ii]	0.93	2.57	3.387 (3)	147

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