

2-[(*E*)-(2-Morpholinoethyl)iminiomethyl]-4-nitro-1-oxocyclohexadienide

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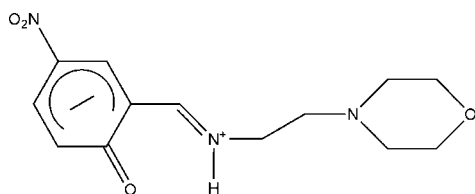
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.040; wR factor = 0.110; data-to-parameter ratio = 16.6.

The molecule of the title compound, $\text{C}_{13}\text{H}_{17}\text{N}_3\text{O}_4$, exists as a zwitterion, with the H atom of the phenol group being transferred to the imine N atom. The $\text{C}=\text{O}$, $\text{C}_{\text{Ar}}-\text{C}_{\text{Ar}}$ and $\text{C}-\text{N}$ bond lengths are in agreement with the oxocyclohexadienide-iminium zwitterionic form. A strong intramolecular $\text{N}^+-\text{H}\cdots\text{O}$ hydrogen bond generates an $S(6)$ ring motif. The morpholine ring adopts a chair conformation. In the crystal, molecules are linked into centrosymmetric dimers by intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds. In addition, $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds and very weak $\text{C}-\text{H}\cdots\pi$ interactions are observed.

Related literature

For general background, photochromic and thermochromic characteristics of Schiff base compounds, see: Calligaris *et al.* (1972); Cohen *et al.* (1964); Hadjoudis *et al.* (1987); Karabiyik *et al.* (2008). For related structures, see: Butt *et al.* (1987); Petek *et al.* (2006); Krygowski & Stepien (2005); Santos-Contreras *et al.* (2009). For graph-set analysis of hydrogen bonds, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{17}\text{N}_3\text{O}_4$
 $M_r = 279.30$
 Triclinic, $P\bar{1}$
 $a = 5.3520$ (4) Å

$b = 10.8972$ (9) Å
 $c = 12.4537$ (9) Å
 $\alpha = 102.329$ (7)°
 $\beta = 97.143$ (6)°

$\gamma = 104.173$ (9)°
 $V = 675.91$ (10) Å³
 $Z = 2$
 Mo $K\alpha$ radiation

$\mu = 0.10$ mm⁻¹
 $T = 296$ K
 $0.75 \times 0.70 \times 0.40$ mm

Data collection

Stoe IPDSII diffractometer
 Absorption correction: none
 11340 measured reflections

3094 independent reflections
 2664 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.039$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.110$
 $S = 1.06$
 3094 reflections
 186 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.26$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.17$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H1}\cdots\text{O1}$	0.89 (2)	1.99 (2)	2.6760 (14)	133 (2)
$\text{N2}-\text{H1}\cdots\text{O1}^{\text{i}}$	0.89 (2)	2.24 (2)	2.9587 (14)	138 (2)
$\text{C4}-\text{H4}\cdots\text{O4}^{\text{ii}}$	0.93	2.47	3.3547 (16)	160
$\text{C7}-\text{H7}\cdots\text{O3}^{\text{iii}}$	0.93	2.43	3.3020 (15)	157
$\text{C13}-\text{H13B}\cdots\text{Cg2}^{\text{iv}}$	0.97	2.99	3.9254	162

Symmetry codes: (i) $-x, -y + 1, -z + 1$; (ii) $x, y + 1, z + 1$; (iii) $-x + 2, -y + 2, -z + 1$; (iv) $-x + 1, -y + 1, -z + 1$. Cg2 is the centroid of the C1-C6 ring.

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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2842).

References

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Butt, G. L., Mackay, M. F. & Topsom, R. D. (1987). *Acta Cryst.* **C43**, 1092–1094.
- Calligaris, M., Nardin, G. & Randaccio, L. (1972). *Coord. Chem. Rev.* **7**, 385–403.
- Cohen, M. D., Schmidt, G. M. J. & Flavian, S. (1964). *J. Chem. Soc.* pp. 2041–2051.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Hadjoudis, E., Vitterakis, M., Moustakali, I. & Mavridis, I. (1987). *Tetrahedron*, **43**, 1345–1360.
- Karabiyik, H., Ocakiskeleli, N., Petek, H., Albayrak, Ç. & Ağar, E. (2008). *J. Mol. Struct.* **873**, 130–136.
- Krygowski, T. M. & Stepien, B. T. (2005). *Chem. Rev.* **105**, 3482–3512.
- Petek, H., Albayrak, Ç., Ağar, E. & Kalkan, H. (2006). *Acta Cryst.* **E62**, o3685–o3687.
- Santos-Contreras, R. J., Ramos-Organillo, A., García-Báez, E. V., Padilla-Martínez, I. I. & Martínez-Martínez, F. J. (2009). *Acta Cryst.* **C65**, o8–o10.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Stoe & Cie (2002). *X-Area* and *X-Red32*. Stoe & Cie, Darmstadt, Germany.

supplementary materials

Acta Cryst. (2009). E65, o1842 [doi:10.1107/S1600536809026191]

2-[(*E*)-(2-Morpholinoethyl)iminomethyl]-4-nitro-1-oxocyclohexadienide

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Comment

Schiff bases have been extensively used as ligands in the field of coordination chemistry (Calligaris *et al.*, 1972). Schiff base compounds can be classified by their photochromic and thermochromic characteristics (Cohen *et al.* 1964). These properties result from proton transfer from the hydroxyl O atom to the imine N atom (Hadjoudis *et al.*, 1987). Schiff bases exhibit two well-known tautomeric forms viz. OH and NH tautomers, and they also exist in zwitterionic form (Karabıyık *et al.*, 2008). Our investigations show that compound (I) exists in a zwitterionic form.

The molecular structure of (I) is shown in Fig.1. The C1—C7 [1.4241 (15) Å], C7=N2 [1.2894 (15) Å] and N2—C8 [1.4615 (14) Å] bond lengths agree with the corresponding distances in (*E*)-2-methoxy-6-[(2-morpholinoethylimino)methyl]phenolate [1.425 (2), 1.287 (2) and 1.464 (2) Å; Petek *et al.*, 2006]. The bonds lengths in the C1-C6 benzene ring show clear alternation in the delocalized C2-C5 portion. The nitro group is tilted out of the mean plane of adjacent ring by 7.02 (3)°, whereas the C3—N1 distance of 1.4398 (15) Å is in the characteristic range suggesting limited conjugation with the ring. Thus, the whole geometry is in the agreement with the predominance of the oxocyclohexadienide-iminum zwitterion bonding scheme (see scheme) (Krygowski & Stepień, 2005; Santos-Contreras *et al.*, 2009), in close agreement with the reported configurations of *p*-nitrophenolates of alkali metal cations (Butt *et al.*, 1987). The morpholine ring adopts a chair conformation. An intramolecular N—H···O hydrogen bond generates an S(6) ring motif (Bernstein *et al.*, 1995).

In the crystal structure of (I), the molecules form centrosymmetric dimers connected by intermolecular N—H···O hydrogen bonds (Fig. 2). In addition, C—H···O hydrogen bonds and very weak C—H··· π interactions between C13-H13B group and C1-C6 benzene ring are observed (Table 1).

Experimental

2-Hydroxy-5-nitrobenzaldehyde (0.0535 g, 0.32 mmol) in ethanol (20 ml) was added to 2-morpholinoethylamine (0.0417 g, 0.32 mmol) in ethanol (20 ml) and the reaction mixture was stirred for 1 h under reflux. Single crystals of the title compound suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution (yield % 65; m.p. 435-438 K).

Refinement

Atom H1 was located in a difference map and refined freely. All other H atoms were placed in calculated positions and constrained to ride on their parent atoms, with C-H = 0.93–0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

Figures

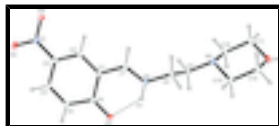


Fig. 1. The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

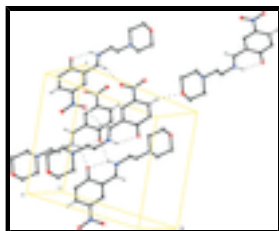


Fig. 2. A packing diagram for (I), showing O—H...O hydrogen-bonded (dashed lines) dimers. H atoms not involved in hydrogen bonding have been omitted for clarity.

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Crystal data

$C_{13}H_{17}N_3O_4$

$M_r = 279.30$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 5.3520$ (4) Å

$b = 10.8972$ (9) Å

$c = 12.4537$ (9) Å

$\alpha = 102.329$ (7)°

$\beta = 97.143$ (6)°

$\gamma = 104.173$ (9)°

$V = 675.91$ (10) Å³

$Z = 2$

$F_{000} = 296$

$D_x = 1.372$ Mg m⁻³

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

Cell parameters from 23053 reflections

$\theta = 1.7$ – 28.0 °

$\mu = 0.10$ mm⁻¹

$T = 296$ K

Prism, orange

$0.75 \times 0.70 \times 0.40$ mm

Data collection

Stoe IPDSII
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 6.67 pixels mm⁻¹

$T = 296$ K

ω scans

Absorption correction: none

11340 measured reflections

3094 independent reflections

2664 reflections with $I > 2\sigma(I)$

$R_{int} = 0.039$

$\theta_{max} = 27.6$ °

$\theta_{min} = 1.7$ °

$h = -6 \rightarrow 6$

$k = -14 \rightarrow 14$

$l = -16 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$	$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 0.1309P]$
$wR(F^2) = 0.110$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{\max} = 0.001$
3094 reflections	$\Delta\rho_{\max} = 0.26 \text{ e } \text{\AA}^{-3}$
186 parameters	$\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kF_c[1+0.001xF_c^2\lambda^3/\sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.077 (7)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\sigma(F^2)$ 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>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.4611 (2)	0.77887 (11)	0.53094 (9)	0.0335 (2)
C2	0.6439 (2)	0.90243 (11)	0.56526 (9)	0.0341 (2)
H2	0.7896	0.9204	0.5315	0.041*
C3	0.6067 (2)	0.99713 (10)	0.64924 (9)	0.0348 (3)
C4	0.3899 (3)	0.97257 (12)	0.70256 (10)	0.0396 (3)
H4	0.3692	1.0377	0.7597	0.048*
C5	0.2112 (3)	0.85348 (12)	0.67033 (11)	0.0426 (3)
H5	0.0698	0.8382	0.7069	0.051*
C6	0.2318 (2)	0.74989 (11)	0.58183 (10)	0.0363 (3)
C7	0.5074 (2)	0.68324 (11)	0.44412 (9)	0.0358 (3)
H7	0.6566	0.7074	0.4138	0.043*
C8	0.4146 (3)	0.46932 (12)	0.31724 (10)	0.0430 (3)
H8A	0.3937	0.3878	0.3393	0.052*
H8B	0.5956	0.5004	0.3093	0.052*
C9	0.2370 (3)	0.44450 (12)	0.20590 (10)	0.0437 (3)
H9A	0.0572	0.4050	0.2113	0.052*
H9B	0.2453	0.5268	0.1865	0.052*
C10	0.2038 (3)	0.36092 (15)	0.00679 (11)	0.0513 (3)
H10A	0.2527	0.4500	-0.0009	0.062*
H10B	0.0140	0.3307	-0.0047	0.062*
C11	0.3025 (3)	0.27445 (17)	-0.07913 (12)	0.0640 (4)
H11A	0.2304	0.2783	-0.1534	0.077*

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H11B	0.4920	0.3062	-0.0681	0.077*
C12	0.3364 (4)	0.13651 (16)	0.03730 (15)	0.0665 (5)
H12A	0.5264	0.1645	0.0487	0.080*
H12B	0.2834	0.0467	0.0429	0.080*
C13	0.2450 (3)	0.22200 (13)	0.12725 (12)	0.0504 (3)
H13A	0.0561	0.1904	0.1194	0.060*
H13B	0.3244	0.2177	0.2002	0.060*
N1	0.7884 (2)	1.12600 (10)	0.68031 (9)	0.0410 (3)
N2	0.3567 (2)	0.56554 (9)	0.40467 (8)	0.0378 (2)
N3	0.3171 (2)	0.35764 (10)	0.11888 (8)	0.0422 (3)
O1	0.06162 (19)	0.64042 (9)	0.55030 (9)	0.0513 (3)
O2	0.7384 (2)	1.21314 (9)	0.74731 (9)	0.0600 (3)
O3	0.9854 (2)	1.14568 (9)	0.63771 (9)	0.0549 (3)
O4	0.2302 (3)	0.14272 (12)	-0.07115 (9)	0.0727 (4)
H1	0.214 (4)	0.5433 (18)	0.4335 (15)	0.065 (5)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0382 (6)	0.0302 (5)	0.0305 (5)	0.0089 (4)	0.0086 (4)	0.0041 (4)
C2	0.0370 (6)	0.0324 (5)	0.0316 (5)	0.0083 (4)	0.0096 (4)	0.0055 (4)
C3	0.0410 (6)	0.0286 (5)	0.0310 (5)	0.0073 (4)	0.0048 (4)	0.0035 (4)
C4	0.0493 (7)	0.0363 (6)	0.0335 (6)	0.0162 (5)	0.0120 (5)	0.0020 (4)
C5	0.0450 (7)	0.0419 (6)	0.0422 (6)	0.0125 (5)	0.0198 (5)	0.0065 (5)
C6	0.0388 (6)	0.0319 (5)	0.0372 (6)	0.0085 (4)	0.0102 (5)	0.0063 (4)
C7	0.0398 (6)	0.0336 (5)	0.0323 (5)	0.0088 (5)	0.0098 (4)	0.0049 (4)
C8	0.0508 (7)	0.0361 (6)	0.0374 (6)	0.0151 (5)	0.0086 (5)	-0.0038 (5)
C9	0.0545 (7)	0.0391 (6)	0.0375 (6)	0.0195 (5)	0.0105 (5)	0.0008 (5)
C10	0.0532 (8)	0.0582 (8)	0.0358 (7)	0.0119 (6)	0.0063 (6)	0.0030 (6)
C11	0.0646 (10)	0.0736 (10)	0.0367 (7)	0.0046 (8)	0.0132 (6)	-0.0071 (7)
C12	0.0776 (11)	0.0501 (8)	0.0648 (10)	0.0231 (8)	0.0203 (8)	-0.0105 (7)
C13	0.0625 (9)	0.0409 (7)	0.0457 (7)	0.0180 (6)	0.0147 (6)	-0.0003 (5)
N1	0.0491 (6)	0.0311 (5)	0.0371 (5)	0.0070 (4)	0.0045 (4)	0.0030 (4)
N2	0.0443 (6)	0.0317 (5)	0.0331 (5)	0.0083 (4)	0.0105 (4)	-0.0002 (4)
N3	0.0497 (6)	0.0406 (5)	0.0324 (5)	0.0146 (5)	0.0088 (4)	-0.0020 (4)
O1	0.0483 (5)	0.0358 (5)	0.0617 (6)	-0.0002 (4)	0.0219 (4)	0.0025 (4)
O2	0.0719 (7)	0.0337 (5)	0.0622 (6)	0.0106 (4)	0.0133 (5)	-0.0092 (4)
O3	0.0556 (6)	0.0417 (5)	0.0564 (6)	-0.0031 (4)	0.0169 (5)	0.0047 (4)
O4	0.0816 (8)	0.0612 (7)	0.0526 (6)	0.0055 (6)	0.0197 (6)	-0.0207 (5)

Geometric parameters (\AA , $^\circ$)

C1—C2	1.3994 (16)	C9—H9A	0.97
C1—C7	1.4241 (15)	C9—H9B	0.97
C1—C6	1.4478 (16)	C10—N3	1.4625 (17)
C2—C3	1.3748 (15)	C10—C11	1.505 (2)
C2—H2	0.93	C10—H10A	0.97
C3—C4	1.4051 (17)	C10—H10B	0.97
C3—N1	1.4398 (15)	C11—O4	1.421 (2)

C4—C5	1.3543 (18)	C11—H11A	0.97
C4—H4	0.93	C11—H11B	0.97
C5—C6	1.4357 (16)	C12—O4	1.421 (2)
C5—H5	0.93	C12—C13	1.5086 (19)
C6—O1	1.2594 (14)	C12—H12A	0.97
C7—N2	1.2894 (15)	C12—H12B	0.97
C7—H7	0.93	C13—N3	1.4632 (18)
C8—N2	1.4615 (14)	C13—H13A	0.97
C8—C9	1.5117 (18)	C13—H13B	0.97
C8—H8A	0.97	N1—O2	1.2293 (14)
C8—H8B	0.97	N1—O3	1.2315 (14)
C9—N3	1.4596 (15)	N2—H1	0.887 (19)
C2—C1—C7	117.84 (10)	N3—C10—H10A	109.8
C2—C1—C6	120.88 (10)	C11—C10—H10A	109.8
C7—C1—C6	121.27 (10)	N3—C10—H10B	109.8
C3—C2—C1	119.54 (10)	C11—C10—H10B	109.8
C3—C2—H2	120.2	H10A—C10—H10B	108.2
C1—C2—H2	120.2	O4—C11—C10	111.17 (13)
C2—C3—C4	121.54 (10)	O4—C11—H11A	109.4
C2—C3—N1	119.24 (10)	C10—C11—H11A	109.4
C4—C3—N1	119.18 (10)	O4—C11—H11B	109.4
C5—C4—C3	119.65 (11)	C10—C11—H11B	109.4
C5—C4—H4	120.2	H11A—C11—H11B	108.0
C3—C4—H4	120.2	O4—C12—C13	111.48 (14)
C4—C5—C6	122.50 (11)	O4—C12—H12A	109.3
C4—C5—H5	118.8	C13—C12—H12A	109.3
C6—C5—H5	118.8	O4—C12—H12B	109.3
O1—C6—C5	122.23 (11)	C13—C12—H12B	109.3
O1—C6—C1	121.91 (10)	H12A—C12—H12B	108.0
C5—C6—C1	115.86 (10)	N3—C13—C12	110.35 (13)
N2—C7—C1	124.56 (11)	N3—C13—H13A	109.6
N2—C7—H7	117.7	C12—C13—H13A	109.6
C1—C7—H7	117.7	N3—C13—H13B	109.6
N2—C8—C9	111.99 (10)	C12—C13—H13B	109.6
N2—C8—H8A	109.2	H13A—C13—H13B	108.1
C9—C8—H8A	109.2	O2—N1—O3	122.35 (11)
N2—C8—H8B	109.2	O2—N1—C3	118.53 (11)
C9—C8—H8B	109.2	O3—N1—C3	119.11 (10)
H8A—C8—H8B	107.9	C7—N2—C8	122.84 (11)
N3—C9—C8	110.22 (10)	C7—N2—H1	117.3 (12)
N3—C9—H9A	109.6	C8—N2—H1	119.8 (12)
C8—C9—H9A	109.6	C9—N3—C10	111.82 (10)
N3—C9—H9B	109.6	C9—N3—C13	112.22 (10)
C8—C9—H9B	109.6	C10—N3—C13	108.56 (11)
H9A—C9—H9B	108.1	C12—O4—C11	109.63 (11)
N3—C10—C11	109.46 (13)		
C7—C1—C2—C3	-179.68 (10)	N3—C10—C11—O4	60.36 (17)
C6—C1—C2—C3	0.69 (17)	O4—C12—C13—N3	-57.64 (18)

supplementary materials

C1—C2—C3—C4	0.62 (18)	C2—C3—N1—O2	172.71 (11)
C1—C2—C3—N1	-176.92 (10)	C4—C3—N1—O2	-4.89 (17)
C2—C3—C4—C5	-0.61 (18)	C2—C3—N1—O3	-6.09 (17)
N1—C3—C4—C5	176.93 (11)	C4—C3—N1—O3	176.31 (11)
C3—C4—C5—C6	-0.7 (2)	C1—C7—N2—C8	-178.52 (11)
C4—C5—C6—O1	-178.16 (12)	C9—C8—N2—C7	-106.90 (14)
C4—C5—C6—C1	1.93 (19)	C8—C9—N3—C10	-163.51 (11)
C2—C1—C6—O1	178.21 (11)	C8—C9—N3—C13	74.22 (14)
C7—C1—C6—O1	-1.41 (18)	C11—C10—N3—C9	177.47 (12)
C2—C1—C6—C5	-1.89 (17)	C11—C10—N3—C13	-58.19 (15)
C7—C1—C6—C5	178.49 (11)	C12—C13—N3—C9	-178.84 (12)
C2—C1—C7—N2	179.62 (11)	C12—C13—N3—C10	57.05 (16)
C6—C1—C7—N2	-0.75 (18)	C13—C12—O4—C11	57.82 (18)
N2—C8—C9—N3	174.64 (11)	C10—C11—O4—C12	-59.34 (18)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H1 \cdots O1	0.89 (2)	1.99 (2)	2.6760 (14)	133 (2)
N2—H1 \cdots O1 ⁱ	0.89 (2)	2.24 (2)	2.9587 (14)	138 (2)
C4—H4 \cdots O4 ⁱⁱ	0.93	2.47	3.3547 (16)	160
C7—H7 \cdots O3 ⁱⁱⁱ	0.93	2.43	3.3020 (15)	157
C13—H13B \cdots Cg2 ^{iv}	0.97	2.99	3.9254	162

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

Fig. 1

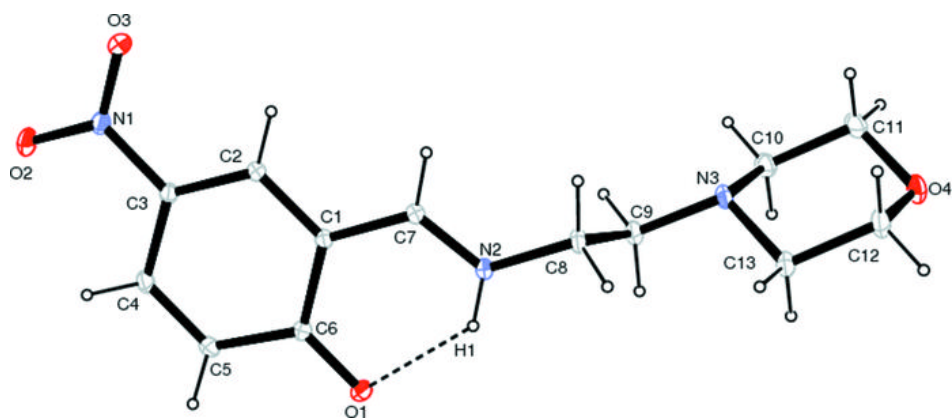


Fig. 2

