

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

3'-Benzyloxy-3-hydroxy-3,3'-bi-1H-indole-2,2'(3H,3'H)-dione monohydrate

P. Ramesh,^a S. S. Sundaresan,^b N. Vidhya Lakshmi,^c Paramasivan T. Perumal^c and M. N. Ponnuswamy^{b*}^aDepartment of Physics, Presidency College (Autonomous), Chennai 600 005, India,^bCentre of Advanced Study in Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, and ^cOrganic Chemistry Division, Central Leather Research Institute, Adyar, Chennai 600 020, India

Correspondence e-mail: mnpsy2004@yahoo.com

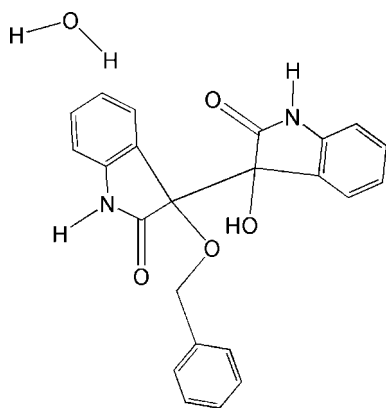
Received 28 February 2009; accepted 2 April 2009

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.032; wR factor = 0.084; data-to-parameter ratio = 11.4.

In the title compound, $\text{C}_{23}\text{H}_{18}\text{N}_2\text{O}_4 \cdot \text{H}_2\text{O}$, the two oxindole rings subtend a dihedral angle of $54.29(5)^\circ$. The crystal structure is stabilized by intermolecular $\text{N}-\text{H} \cdots \text{O}$, $\text{O}-\text{H} \cdots \text{O}$ and $\text{C}-\text{H} \cdots \pi$ interactions.

Related literature

For the biological activity and pharmaceutical applications of indole derivatives, see: Harris & Uhle (1960); Ho *et al.* (1986); Rajeswaran *et al.* (1999); Stevenson *et al.* (2000). For description of hydrogen-bond motifs, see Bernstein *et al.* (1995).



Experimental

Crystal data

$\text{C}_{23}\text{H}_{18}\text{N}_2\text{O}_4 \cdot \text{H}_2\text{O}$
 $M_r = 404.41$
 Triclinic, $P\bar{1}$

$a = 9.8243(3)$ Å
 $b = 9.9304(6)$ Å
 $c = 11.4460(5)$ Å

$\alpha = 107.517(2)^\circ$
 $\beta = 114.227(3)^\circ$
 $\gamma = 93.918(2)^\circ$
 $V = 947.17(8)$ Å³
 $Z = 2$

Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 293$ K
 $0.28 \times 0.25 \times 0.20$ mm

Data collection

Bruker Kappa APEXII area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 2001)
 $T_{\min} = 0.972$, $T_{\max} = 0.980$

16894 measured reflections
 3335 independent reflections
 3045 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.084$
 $S = 1.05$
 3335 reflections
 292 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.23$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.18$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{N1}-\text{H1} \cdots \text{O5}^{\text{i}}$	0.860 (18)	2.045 (18)	2.8858 (17)	165.6 (15)
$\text{O2}-\text{H2} \cdots \text{O3}^{\text{ii}}$	0.91 (2)	1.99 (2)	2.8463 (13)	155.9 (18)
$\text{O2}-\text{H2} \cdots \text{O2}^{\text{ii}}$	0.91 (2)	2.57 (2)	3.0274 (18)	111.9 (14)
$\text{O5}-\text{H5B} \cdots \text{O3}^{\text{iii}}$	0.96 (3)	1.97 (3)	2.8969 (17)	161 (3)
$\text{N12}-\text{H12} \cdots \text{O1}^{\text{iv}}$	0.877 (16)	2.044 (16)	2.8329 (14)	149.2 (14)
$\text{C17}-\text{H17} \cdots \text{Cg3}$	0.93	3.15	3.8226 (17)	130
$\text{C16}-\text{H16} \cdots \text{Cg5}^{\text{v}}$	0.93	2.75	3.5140 (19)	140

Symmetry codes: (i) $-x, -y, -z$; (ii) $-x+1, -y, -z+1$; (iii) $-x+1, -y, -z$; (iv) $-x+1, -y+1, -z+1$; (v) $-x, -y, -z$. Cg3 is the centroid of the C2–C7 ring and Cg5 is the centroid of the C20–C25 ring.

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); 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 (Farrugia, 1997); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2009).

PR thanks Dr Babu Varghese, SAIF, IIT-Madras, India, for his help with the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2889).

References

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N. L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
 Bruker (2004). APEX2 and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Harris, L. S. & Uhle, F. C. (1960). *J. Pharmacol. Exp. Ther.* **128**, 353–363.
 Ho, C. Y., Haegman, W. E. & Perisco, F. (1986). *J. Med. Chem.* **29**, 118–121.
 Rajeswaran, W. G., Labroo, R. B., Cohen, L. A. & King, M. M. (1999). *J. Org. Chem.* **64**, 1369–1371.
 Sheldrick, G. M. (2001). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
 Stevenson, G. I., Smith, A. L., Lewis, S. G., Neduvellil, J. G., Patel, S., Marwood, R. & Castro, J. L. (2000). *Bioorg. Med. Chem. Lett.* **10**, 2697–2704.

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Acta Cryst. (2009). E65, o994 [doi:10.1107/S1600536809012380]

3'-Benzyloxy-3-hydroxy-3,3'-bi-1*H*-indole-2,2'(3*H*,3'*H*)-dione monohydrate

P. Ramesh, S. S. Sundaresan, N. V. Lakshmi, P. T. Perumal and M. N. Ponnuswamy

Comment

Indole derivatives are used as bioactive drugs (Stevenson *et al.*, 2000) and they exhibit anti-allergic, central nervous system depressant and muscle relaxant properties (Harris & Uhle 1960; Ho *et al.*, 1986). Indoles have been proved to display high aldose reductase inhibitory activity (Rajeswaran *et al.*, 1999). Against this background and to ascertain the molecular conformation, the structure determination of the title compound has been carried out.

The ORTEP plot of the molecule is shown in Fig. 1. The oxindole rings enclose a dihedral angle of 54.29 (5)°. In the benzene ring of the indole ring system, the endocyclic angles at C3 and C14 are contracted to 117.24 (25)° and 116.90 (14)°, while those at C2 and C13 are expanded to 122.20 (14)° and 122.69 (13)°, respectively. This would appear to be a real effect caused by the fusion of the pyrrole with benzene ring resulting in an angular distortion. The sum of the bond angles around the hetero nitrogen atom in the oxindole ring systems are equal to N1 [359.76 (11)°] and N12 [359.93 (10)°], is in accordance with the sp^2 hybridization.

The packing of the molecules in the crystal structure is stabilized through N-H...O, O-H...O and C-H... π interactions. Atom N12 (*x*, *y*, *z*) donates a proton to O1 (-*x*+1, -*y*+1, -*z*+1) and forms a dimer with graph-set motif of $R^2_2(14)$ (Bernstein *et al.*, 1995). The intermolecular N1-H1...O5 and O5-H5B...O3 hydrogen bonds form a one dimensional chain running along *a*-axis. The indole ring interacts with the other indole moiety through a weak intra C-H... π interaction involving atom C17, the separation between H17 and the centroid of the C2/C3/C4/C5/C6/C7 (Cg3) ring being 3.15Å.

Experimental

To a refluxing solution of isatin (1 equivalent), Rh₂(OAc)₄ (0.01 equivalent) in dichloromethane, benzyl alcohol (1.2 equivalent) was added. After five minutes, 3-diazo-1,3-dihydro-indol-2-one in dichloromethane was added dropwise through a syringe pump. The reaction mixture was allowed to stir at 60°C for half-an-hour. The solid formed in the reaction was filtered and washed with methanol to obtain the pure product. The compound was recrystallized in ethanol.

Refinement

H atoms bonded to nitrogen and oxygen H atoms were freely refined. H atoms bonded to carbon were positioned geometrically (C—H=0.93–0.97 Å) and allowed to ride on their parent atoms with 1.2 U_{eq} (C).

Figures

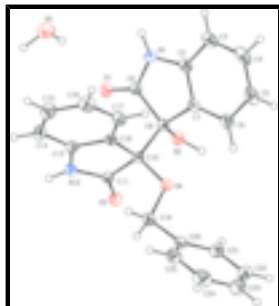


Fig. 1. Perspective view of the molecule with displacement ellipsoids drawn at the 50% probability level. The H atoms are shown as small circles of arbitrary radii.

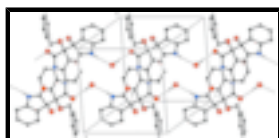


Fig. 2. The crystal packing of the molecules viewed down *c* axis. H atoms not involved in hydrogen bonding have been omitted for clarity.

3'-Benzyloxy-3-hydroxy-3,3'-bi-1*H*-indole-2,2'(3*H*,3'*H*)-dione monohydrate

Crystal data

$C_{23}H_{18}N_2O_4 \cdot H_2O$

$M_r = 404.41$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 9.8243$ (3) Å

$b = 9.9304$ (6) Å

$c = 11.4460$ (5) Å

$\alpha = 107.517$ (2)°

$\beta = 114.227$ (3)°

$\gamma = 93.918$ (2)°

$V = 947.17$ (8) Å³

$Z = 2$

$F_{000} = 424$

$D_x = 1.418$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 4635 reflections

$\theta = 2.1$ – 25.0 °

$\mu = 0.10$ mm⁻¹

$T = 293$ K

Block, colourless

$0.28 \times 0.25 \times 0.20$ mm

Data collection

Bruker Kappa APEXII area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ K

ω and φ scans

Absorption correction: Multi-scan (SADABS; Sheldrick, 2001)

$T_{\min} = 0.972$, $T_{\max} = 0.980$

16894 measured reflections

3335 independent reflections

3045 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\text{max}} = 25.0$ °

$\theta_{\text{min}} = 2.1$ °

$h = -11 \rightarrow 11$

$k = -11 \rightarrow 11$

$l = -13 \rightarrow 13$

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
$R[F^2 > 2\sigma(F^2)] = 0.032$	$w = 1/[\sigma^2(F_o^2) + (0.038P)^2 + 0.3004P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.084$	$(\Delta/\sigma)_{\max} = 0.003$
$S = 1.05$	$\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$
3335 reflections	$\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$
292 parameters	Extinction correction: SHELXL, $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.038 (3)
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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.33868 (11)	0.37105 (9)	0.58225 (10)	0.0399 (2)
O2	0.38716 (10)	0.07924 (10)	0.53420 (9)	0.0335 (2)
H2	0.381 (2)	-0.018 (2)	0.504 (2)	0.070 (6)*
O3	0.61939 (10)	0.21310 (10)	0.48237 (10)	0.0374 (2)
O4	0.33920 (10)	0.00510 (8)	0.25071 (9)	0.0302 (2)
O5	0.09912 (16)	-0.40448 (16)	-0.55511 (16)	0.0632 (3)
H5B	0.183 (4)	-0.346 (3)	-0.552 (3)	0.129 (10)*
H5A	0.064 (4)	-0.482 (4)	-0.622 (3)	0.135 (12)*
N1	0.09503 (13)	0.23295 (12)	0.47470 (12)	0.0349 (3)
H1	0.0522 (19)	0.2945 (18)	0.5098 (17)	0.048 (4)*
C2	0.02036 (14)	0.08934 (14)	0.38791 (13)	0.0320 (3)
C3	-0.12940 (15)	0.02332 (17)	0.34058 (15)	0.0435 (3)
H3	-0.1961	0.0735	0.3647	0.052*
C4	-0.17671 (17)	-0.12064 (19)	0.25573 (16)	0.0520 (4)
H4	-0.2777	-0.1681	0.2211	0.062*

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C5	-0.07767 (19)	-0.19539 (17)	0.22128 (17)	0.0521 (4)
H5	-0.1129	-0.2921	0.1632	0.062*
C6	0.07409 (17)	-0.12838 (15)	0.27202 (14)	0.0407 (3)
H6	0.1416	-0.1794	0.2502	0.049*
C7	0.12222 (14)	0.01554 (13)	0.35540 (12)	0.0288 (3)
C8	0.27766 (13)	0.11694 (12)	0.43061 (12)	0.0262 (3)
C9	0.24378 (14)	0.25831 (13)	0.50590 (12)	0.0286 (3)
C10	0.33824 (13)	0.14368 (12)	0.33172 (12)	0.0255 (3)
C11	0.50226 (13)	0.24303 (12)	0.41187 (12)	0.0273 (3)
N12	0.49318 (12)	0.36457 (11)	0.38326 (11)	0.0305 (2)
H12	0.5721 (18)	0.4358 (17)	0.4159 (16)	0.042 (4)*
C13	0.34446 (14)	0.36220 (13)	0.29098 (12)	0.0289 (3)
C14	0.29438 (16)	0.46934 (14)	0.24218 (14)	0.0382 (3)
H14	0.3602	0.5570	0.2701	0.046*
C15	0.14143 (18)	0.44063 (16)	0.14958 (15)	0.0448 (4)
H15	0.1036	0.5108	0.1147	0.054*
C16	0.04399 (16)	0.31071 (17)	0.10790 (15)	0.0443 (4)
H16	-0.0580	0.2940	0.0447	0.053*
C17	0.09647 (15)	0.20413 (15)	0.15937 (13)	0.0359 (3)
H17	0.0307	0.1164	0.1314	0.043*
C18	0.24816 (14)	0.23153 (13)	0.25278 (12)	0.0277 (3)
C19	0.40617 (18)	0.00789 (15)	0.16166 (15)	0.0418 (3)
H19A	0.5160	0.0441	0.2151	0.050*
H19B	0.3649	0.0719	0.1131	0.050*
C20	0.37193 (14)	-0.14144 (13)	0.06125 (13)	0.0315 (3)
C21	0.39451 (15)	-0.25784 (15)	0.10387 (14)	0.0370 (3)
H21	0.4291	-0.2440	0.1963	0.044*
C22	0.36598 (19)	-0.39435 (16)	0.00986 (17)	0.0492 (4)
H22	0.3802	-0.4725	0.0390	0.059*
C23	0.3165 (2)	-0.41584 (17)	-0.12678 (18)	0.0573 (4)
H23	0.2985	-0.5080	-0.1897	0.069*
C24	0.2940 (2)	-0.30139 (19)	-0.16987 (16)	0.0566 (4)
H24	0.2607	-0.3157	-0.2623	0.068*
C25	0.32036 (17)	-0.16459 (17)	-0.07653 (15)	0.0440 (3)
H25	0.3033	-0.0874	-0.1067	0.053*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0466 (6)	0.0270 (5)	0.0427 (5)	0.0014 (4)	0.0243 (5)	0.0037 (4)
O2	0.0331 (5)	0.0333 (5)	0.0335 (5)	0.0095 (4)	0.0121 (4)	0.0154 (4)
O3	0.0245 (5)	0.0340 (5)	0.0483 (6)	0.0061 (4)	0.0110 (4)	0.0158 (4)
O4	0.0346 (5)	0.0230 (4)	0.0360 (5)	0.0038 (3)	0.0224 (4)	0.0061 (4)
O5	0.0581 (8)	0.0594 (8)	0.0800 (9)	0.0163 (6)	0.0439 (7)	0.0168 (7)
N1	0.0346 (6)	0.0360 (6)	0.0414 (6)	0.0138 (5)	0.0235 (5)	0.0135 (5)
C2	0.0298 (6)	0.0400 (7)	0.0316 (6)	0.0074 (5)	0.0149 (5)	0.0187 (6)
C3	0.0279 (7)	0.0621 (9)	0.0482 (8)	0.0095 (6)	0.0179 (6)	0.0292 (7)
C4	0.0302 (7)	0.0672 (11)	0.0510 (9)	-0.0090 (7)	0.0111 (7)	0.0258 (8)

C5	0.0502 (9)	0.0427 (8)	0.0482 (9)	-0.0148 (7)	0.0180 (7)	0.0082 (7)
C6	0.0428 (8)	0.0333 (7)	0.0433 (8)	-0.0011 (6)	0.0219 (7)	0.0089 (6)
C7	0.0282 (6)	0.0306 (6)	0.0293 (6)	0.0026 (5)	0.0139 (5)	0.0125 (5)
C8	0.0256 (6)	0.0250 (6)	0.0283 (6)	0.0057 (5)	0.0123 (5)	0.0096 (5)
C9	0.0332 (7)	0.0277 (6)	0.0296 (6)	0.0076 (5)	0.0174 (5)	0.0117 (5)
C10	0.0247 (6)	0.0217 (6)	0.0288 (6)	0.0041 (4)	0.0132 (5)	0.0064 (5)
C11	0.0257 (6)	0.0253 (6)	0.0304 (6)	0.0055 (5)	0.0145 (5)	0.0070 (5)
N12	0.0265 (5)	0.0251 (5)	0.0359 (6)	0.0003 (4)	0.0123 (5)	0.0096 (4)
C13	0.0311 (6)	0.0290 (6)	0.0279 (6)	0.0066 (5)	0.0152 (5)	0.0095 (5)
C14	0.0458 (8)	0.0319 (7)	0.0389 (7)	0.0085 (6)	0.0185 (6)	0.0164 (6)
C15	0.0505 (9)	0.0480 (8)	0.0432 (8)	0.0204 (7)	0.0189 (7)	0.0272 (7)
C16	0.0328 (7)	0.0612 (9)	0.0389 (8)	0.0134 (7)	0.0110 (6)	0.0247 (7)
C17	0.0290 (7)	0.0431 (7)	0.0334 (7)	0.0037 (6)	0.0119 (5)	0.0151 (6)
C18	0.0289 (6)	0.0293 (6)	0.0269 (6)	0.0061 (5)	0.0152 (5)	0.0092 (5)
C19	0.0520 (8)	0.0333 (7)	0.0501 (8)	0.0037 (6)	0.0375 (7)	0.0089 (6)
C20	0.0287 (6)	0.0326 (7)	0.0353 (7)	0.0053 (5)	0.0194 (5)	0.0084 (5)
C21	0.0376 (7)	0.0400 (7)	0.0350 (7)	0.0105 (6)	0.0179 (6)	0.0129 (6)
C22	0.0611 (10)	0.0361 (8)	0.0614 (10)	0.0175 (7)	0.0380 (8)	0.0161 (7)
C23	0.0735 (11)	0.0402 (8)	0.0540 (10)	0.0033 (8)	0.0412 (9)	-0.0039 (7)
C24	0.0673 (11)	0.0638 (11)	0.0328 (8)	0.0027 (8)	0.0273 (8)	0.0056 (7)
C25	0.0499 (9)	0.0494 (8)	0.0409 (8)	0.0117 (7)	0.0253 (7)	0.0201 (7)

Geometric parameters (Å, °)

O1—C9	1.2191 (15)	N12—C13	1.4016 (16)
O2—C8	1.4066 (14)	N12—H12	0.877 (16)
O2—H2	0.91 (2)	C13—C14	1.3747 (18)
O3—C11	1.2218 (15)	C13—C18	1.3861 (17)
O4—C10	1.4123 (14)	C14—C15	1.384 (2)
O4—C19	1.4273 (15)	C14—H14	0.9300
O5—H5B	0.96 (3)	C15—C16	1.376 (2)
O5—H5A	0.83 (3)	C15—H15	0.9300
N1—C9	1.3392 (17)	C16—C17	1.391 (2)
N1—C2	1.4017 (17)	C16—H16	0.9300
N1—H1	0.860 (18)	C17—C18	1.3783 (18)
C2—C3	1.3742 (19)	C17—H17	0.9300
C2—C7	1.3851 (18)	C19—C20	1.4906 (18)
C3—C4	1.381 (2)	C19—H19A	0.9700
C3—H3	0.9300	C19—H19B	0.9700
C4—C5	1.375 (2)	C20—C25	1.3808 (19)
C4—H4	0.9300	C20—C21	1.3816 (19)
C5—C6	1.387 (2)	C21—C22	1.377 (2)
C5—H5	0.9300	C21—H21	0.9300
C6—C7	1.3759 (18)	C22—C23	1.374 (2)
C6—H6	0.9300	C22—H22	0.9300
C7—C8	1.5086 (16)	C23—C24	1.366 (3)
C8—C9	1.5477 (16)	C23—H23	0.9300
C8—C10	1.5551 (16)	C24—C25	1.380 (2)
C10—C18	1.5114 (17)	C24—H24	0.9300

supplementary materials

C10—C11	1.5608 (16)	C25—H25	0.9300
C11—N12	1.3418 (16)		
C8—O2—H2	110.3 (12)	C13—N12—H12	123.8 (10)
C10—O4—C19	113.84 (9)	C14—C13—C18	122.68 (12)
H5B—O5—H5A	112 (3)	C14—C13—N12	127.59 (12)
C9—N1—C2	111.86 (11)	C18—C13—N12	109.73 (11)
C9—N1—H1	124.2 (11)	C13—C14—C15	116.90 (13)
C2—N1—H1	123.7 (11)	C13—C14—H14	121.5
C3—C2—C7	122.20 (13)	C15—C14—H14	121.5
C3—C2—N1	127.96 (13)	C16—C15—C14	121.59 (13)
C7—C2—N1	109.82 (11)	C16—C15—H15	119.2
C2—C3—C4	117.24 (14)	C14—C15—H15	119.2
C2—C3—H3	121.4	C15—C16—C17	120.67 (13)
C4—C3—H3	121.4	C15—C16—H16	119.7
C5—C4—C3	121.41 (13)	C17—C16—H16	119.7
C5—C4—H4	119.3	C18—C17—C16	118.49 (12)
C3—C4—H4	119.3	C18—C17—H17	120.8
C4—C5—C6	120.82 (14)	C16—C17—H17	120.8
C4—C5—H5	119.6	C17—C18—C13	119.65 (12)
C6—C5—H5	119.6	C17—C18—C10	131.70 (11)
C7—C6—C5	118.33 (14)	C13—C18—C10	108.63 (10)
C7—C6—H6	120.8	O4—C19—C20	109.22 (10)
C5—C6—H6	120.8	O4—C19—H19A	109.8
C6—C7—C2	119.98 (12)	C20—C19—H19A	109.8
C6—C7—C8	131.53 (12)	O4—C19—H19B	109.8
C2—C7—C8	108.41 (10)	C20—C19—H19B	109.8
O2—C8—C7	114.09 (10)	H19A—C19—H19B	108.3
O2—C8—C9	105.88 (9)	C25—C20—C21	118.92 (12)
C7—C8—C9	101.76 (9)	C25—C20—C19	119.93 (13)
O2—C8—C10	111.95 (9)	C21—C20—C19	121.13 (12)
C7—C8—C10	112.73 (9)	C22—C21—C20	120.20 (13)
C9—C8—C10	109.61 (9)	C22—C21—H21	119.9
O1—C9—N1	126.95 (12)	C20—C21—H21	119.9
O1—C9—C8	124.94 (11)	C23—C22—C21	120.39 (15)
N1—C9—C8	108.10 (10)	C23—C22—H22	119.8
O4—C10—C18	115.50 (9)	C21—C22—H22	119.8
O4—C10—C8	105.35 (9)	C24—C23—C22	119.82 (14)
C18—C10—C8	112.18 (9)	C24—C23—H23	120.1
O4—C10—C11	110.65 (9)	C22—C23—H23	120.1
C18—C10—C11	101.56 (9)	C23—C24—C25	120.11 (14)
C8—C10—C11	111.77 (9)	C23—C24—H24	119.9
O3—C11—N12	125.87 (11)	C25—C24—H24	119.9
O3—C11—C10	126.42 (11)	C24—C25—C20	120.55 (14)
N12—C11—C10	107.61 (10)	C24—C25—H25	119.7
C11—N12—C13	112.33 (10)	C20—C25—H25	119.7
C11—N12—H12	123.8 (10)		
C9—N1—C2—C3	176.59 (13)	O4—C10—C11—O3	-50.44 (16)
C9—N1—C2—C7	-2.02 (15)	C18—C10—C11—O3	-173.59 (12)

C7—C2—C3—C4	-1.4 (2)	C8—C10—C11—O3	66.64 (15)
N1—C2—C3—C4	-179.82 (13)	O4—C10—C11—N12	126.13 (10)
C2—C3—C4—C5	0.7 (2)	C18—C10—C11—N12	2.99 (12)
C3—C4—C5—C6	0.6 (2)	C8—C10—C11—N12	-116.79 (11)
C4—C5—C6—C7	-1.3 (2)	O3—C11—N12—C13	175.33 (12)
C5—C6—C7—C2	0.7 (2)	C10—C11—N12—C13	-1.27 (13)
C5—C6—C7—C8	177.20 (13)	C11—N12—C13—C14	178.62 (12)
C3—C2—C7—C6	0.65 (19)	C11—N12—C13—C18	-1.22 (14)
N1—C2—C7—C6	179.35 (11)	C18—C13—C14—C15	-1.10 (19)
C3—C2—C7—C8	-176.58 (11)	N12—C13—C14—C15	179.07 (12)
N1—C2—C7—C8	2.12 (14)	C13—C14—C15—C16	-0.2 (2)
C6—C7—C8—O2	-64.69 (17)	C14—C15—C16—C17	0.8 (2)
C2—C7—C8—O2	112.10 (11)	C15—C16—C17—C18	-0.1 (2)
C6—C7—C8—C9	-178.23 (13)	C16—C17—C18—C13	-1.14 (19)
C2—C7—C8—C9	-1.44 (12)	C16—C17—C18—C10	176.85 (12)
C6—C7—C8—C10	64.46 (17)	C14—C13—C18—C17	1.79 (19)
C2—C7—C8—C10	-118.75 (11)	N12—C13—C18—C17	-178.35 (11)
C2—N1—C9—O1	-177.54 (12)	C14—C13—C18—C10	-176.62 (11)
C2—N1—C9—C8	1.01 (14)	N12—C13—C18—C10	3.23 (13)
O2—C8—C9—O1	59.33 (15)	O4—C10—C18—C17	58.38 (17)
C7—C8—C9—O1	178.85 (11)	C8—C10—C18—C17	-62.37 (16)
C10—C8—C9—O1	-61.60 (15)	C11—C10—C18—C17	178.15 (13)
O2—C8—C9—N1	-119.26 (10)	O4—C10—C18—C13	-123.47 (11)
C7—C8—C9—N1	0.27 (12)	C8—C10—C18—C13	115.79 (11)
C10—C8—C9—N1	119.82 (11)	C11—C10—C18—C13	-3.70 (12)
C19—O4—C10—C18	61.51 (14)	C10—O4—C19—C20	-168.10 (10)
C19—O4—C10—C8	-174.11 (10)	O4—C19—C20—C25	133.58 (13)
C19—O4—C10—C11	-53.14 (13)	O4—C19—C20—C21	-48.07 (17)
O2—C8—C10—O4	73.14 (11)	C25—C20—C21—C22	0.1 (2)
C7—C8—C10—O4	-57.10 (12)	C19—C20—C21—C22	-178.29 (13)
C9—C8—C10—O4	-169.68 (9)	C20—C21—C22—C23	0.7 (2)
O2—C8—C10—C18	-160.41 (9)	C21—C22—C23—C24	-0.7 (3)
C7—C8—C10—C18	69.34 (12)	C22—C23—C24—C25	-0.1 (3)
C9—C8—C10—C18	-43.23 (13)	C23—C24—C25—C20	1.0 (3)
O2—C8—C10—C11	-47.09 (13)	C21—C20—C25—C24	-0.9 (2)
C7—C8—C10—C11	-177.34 (9)	C19—C20—C25—C24	177.44 (14)
C9—C8—C10—C11	70.09 (12)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O5 ⁱ	0.860 (18)	2.045 (18)	2.8858 (17)	165.6 (15)
O2—H2...O3 ⁱⁱ	0.91 (2)	1.99 (2)	2.8463 (13)	155.9 (18)
O2—H2...O2 ⁱⁱ	0.91 (2)	2.57 (2)	3.0274 (18)	111.9 (14)
O5—H5B...O3 ⁱⁱⁱ	0.96 (3)	1.97 (3)	2.8969 (17)	161 (3)
N12—H12...O1 ^{iv}	0.877 (16)	2.044 (16)	2.8329 (14)	149.2 (14)
C17—H17...Cg3	0.93	3.15	3.8226 (17)	131
C16—H16...Cg5 ^v	0.93	2.75	3.5140 (19)	140

supplementary materials

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

Fig. 1

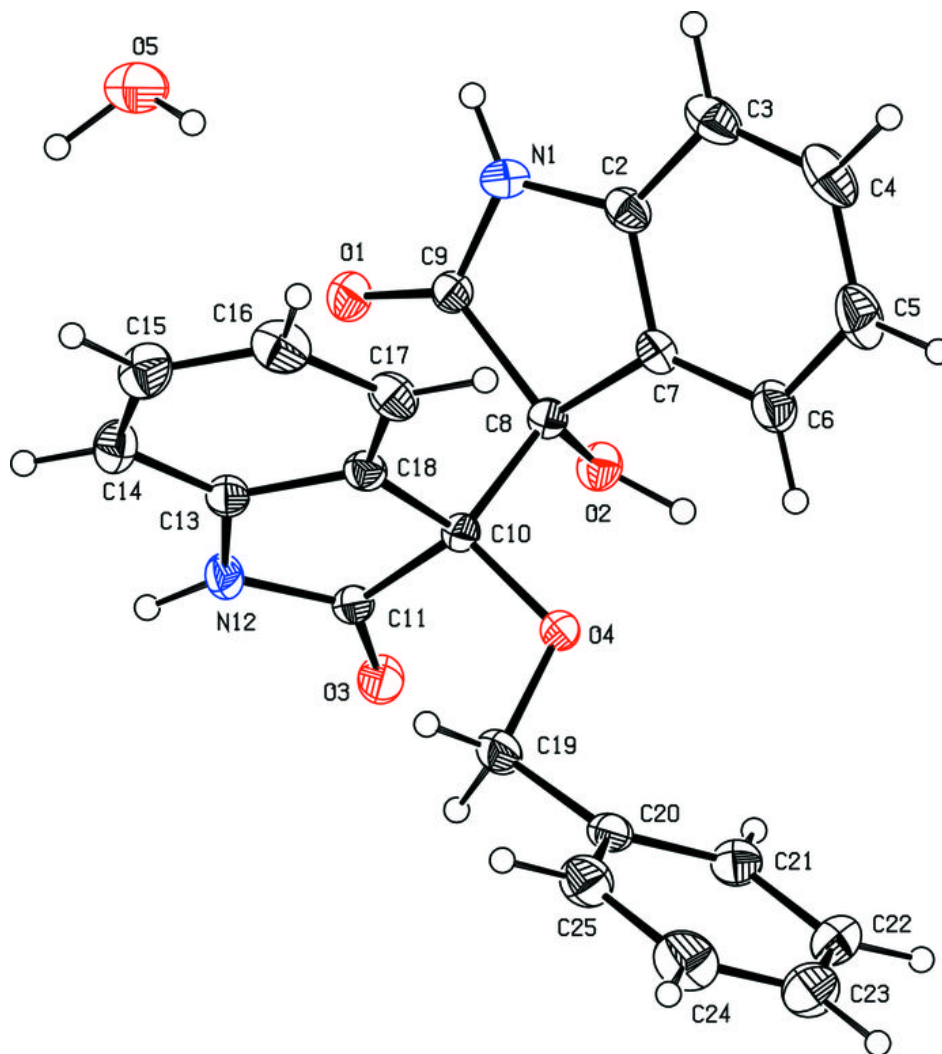


Fig. 2

