

11-Methyl-2,3-benzodipyrin-1-one

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Received 4 August 2004

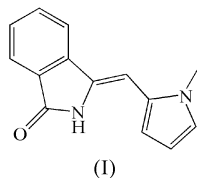
Accepted 21 October 2004

Online 23 November 2004

The title compound {alternative names: 11-methyl-2,3-benzopyrromethenone and 3-[(1-methylpyrrol-2-yl)methylidene]-2,3-dihydro-1*H*-isoindol-1-one}, C₁₄H₁₂N₂O, was prepared by the base-catalysed condensation of phthalimidine with 2-formyl-1-methylpyrrole; yellow orthorhombic crystals, space group *Pbca*, were obtained from ethanol. The molecule is almost planar, having *Z*(−)antiperiplanar geometry. The molecules are arranged in pairs with intermolecular hydrogen bonding between lactam functions. Comparison with literature values for polyalkyldipyrin-1-ones shows that, apart from the local constraints of the benzene ring, the fused benzo ring has little effect on the molecular dimensions of the dipyrin-1-one skeleton.

Comment

Bilirubin, which adopts a ridge-tile structure with extensive intramolecular hydrogen bonding in the crystal (Bonnett *et al.*, 1978), contains two inequivalent dipyrin-1-one (pyrromethenone) units. Such units also occur in a variety of other plant and animal linear tetrapyrroles. Hence, the crystal structures of dipyrin-1-ones have attracted some attention [for a review, see Sheldrick (1983)]. We have also had an interest in dipyrin-1-ones as potential building blocks for the synthesis of benzoporphyrins (Bonnett & McManus, 1996; Valles *et al.*, 1996).



This report concerns the 2,3-benzodipyrin-1-one system, as the 11-methyl derivative, (I). Few representatives of this system have been described (Swanson, 1991; Boiadjev & Lightner, 2003*a*), and we have found no previous X-ray crystal

structure determination in this series. However, X-ray analyses have been reported for two distantly related structures, namely 3-[(pyrrol-2-yl)methylidene]indolin-2-one, derived from 2-oxindole (Boiadjev & Lightner, 2003*b*), and 3-benzylideneisoindolin-1-one (Mukherjee *et al.*, 2000). The photophysical properties of (I) in organic solvents and in micellar preparations have been reported (Gerhardt *et al.*, 2003), but the compound was there formulated with the *Z*-*syn* geometry.

The molecular structure of (I) is shown in Fig. 1. X-Ray analysis shows that the molecule has a 4-*Z*-antiperiplanar geometry, the chromophore being essentially planar, with N10–C4–C5–C6 and C4–C5–C6–N11 torsion angles of –2.0 (3) and 178.05 (16)°, respectively. Cullen *et al.* (1979) reported a similar geometry for 11-methyl-2,3-dimethyldipyrin-1-one and, as with that compound, the molecules

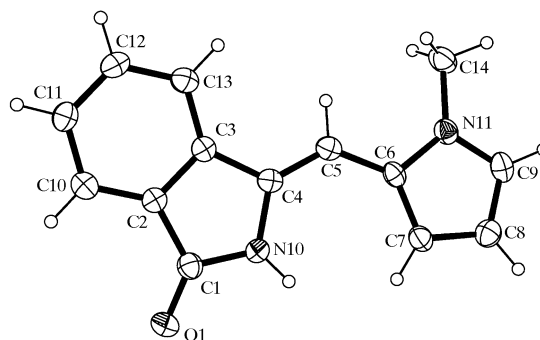


Figure 1

The molecular structure of (I). Ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

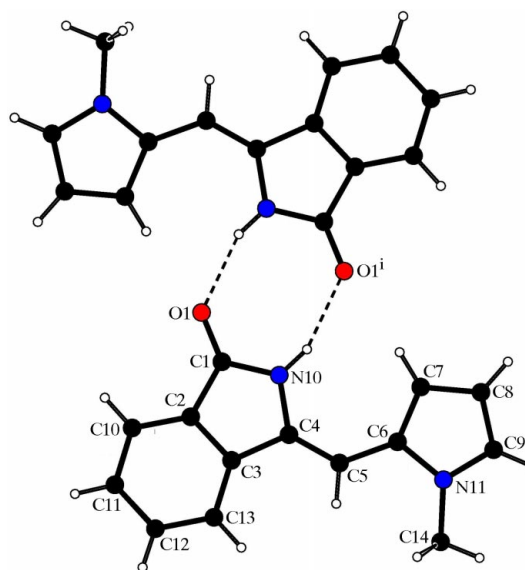
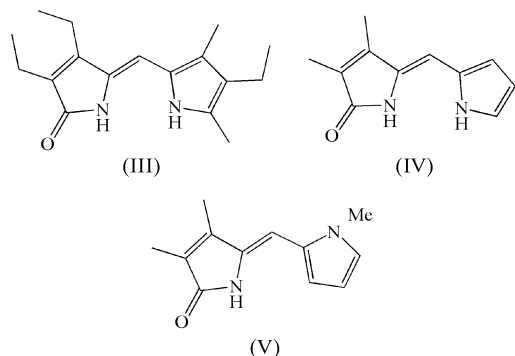


Figure 2

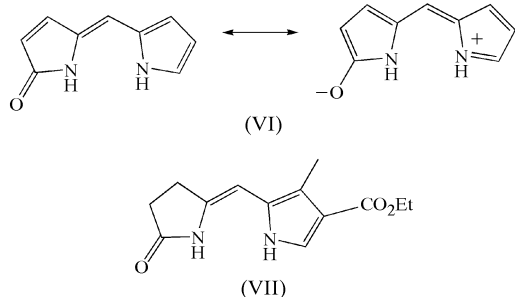
The dimeric assembly of (I), involving hydrogen-bonded (dashed lines) lactam groups. [Symmetry code: (i) $-x + 1, -y, -z$.]

in (I) are arranged in the crystal as dimers, with intermolecular hydrogen bonding between lactam groups (Table 1 and Fig. 2).



The bond lengths and angles observed here are similar to those observed in other 4-*Z*-dipyrrin-1-ones, namely (III) (Cullen *et al.*, 1977), (IV) and (V) (Cullen *et al.*, 1979), all of which have only alkyl substitution. The main difference, as might be anticipated, is in the region of the benzenoid ring; thus, the C2–C3 bond length in (I), at 1.391 (2) Å, is significantly longer than the average (1.325 Å) of that bond length for compounds (III)–(V). It may be noted that 4-*E*-dipyrrin-1-ones have also been prepared, typically by photoisomerization of the *Z* compounds, and X-ray structures are available (Sheldrick *et al.*, 1977; Hori *et al.*, 1981).

Other comparisons are made in Table 2. The bond lengths around the C5 bridge are of interest. The C4–C5 bond is slightly longer than expected for a double bond [although this discrepancy is marginal for (I), it still occurs], and the C5–C6 bond is slightly shorter than expected for a single bond. These changes are in accord with the pattern of delocalization shown in (VI). Cullen *et al.* (1979) have noted that the C1–N10 bond is significantly shorter than the C4–N10 bond, and this is also apparent in the 2,3-benzo derivative (I). We attribute this fact to the well known partial double-bond character of the C–N bond in amide functions. There is also a difference in length between the C9–N11 and C6–N11 bonds, the former being the shorter because of the delocalization represented in (VI). Although this delocalization can occur in (I) (although it is less pronounced because of the formal disruption of the benzenoid ring), it cannot occur in the *Z*-2,3-dihydro system (VII), and in this example the C9–N11 bond [1.386 (5) Å] is actually increased with respect to the C6–N11 bond



[1.373 (5) Å] (Gossauer *et al.*, 1976) because of delocalization to the 9-ethoxycarbonyl group.

Experimental

Compound (I) was prepared as follows (Swanson, 1991). A solution of phthalimidine (isoindol-1-one, 0.63 g) and 2-formyl-1-methylpyrrole (0.51 g) in ethanol (25 ml) was treated with aqueous sodium hydroxide (4 *M*, 20 ml) and heated under reflux for 7 h. The resulting yellow–orange solution was poured into ice-water. The bright-yellow precipitate was filtered off and washed with water to give a bright-yellow powder (0.30 g). Extraction of the filtrate with chloroform gave a further 0.05 g. The combined yellow solids were crystallized from ethanol to give (I) (0.19 g, 20%) as fine yellow needles (m.p. 471–475 K, with decomposition). Working on a larger scale allowed the yield to be increased to 40%. λ_{\max} (MeOH): 386 nm (ϵ 21 200 M⁻¹ cm⁻¹). ν_{\max} (KBr): 3400–3200, 1680, 1610, 1470, 1430, 1320 cm⁻¹. Analysis calculated for C₁₄H₁₂N₂O: C 75.00, H 5.36, N 12.50%; found: C 74.83, H 5.32, N 12.45%. Single crystals suitable for X-ray analysis were grown from ethanol.

Crystal data

C ₁₄ H ₁₂ N ₂ O	Mo <i>K</i> α radiation
<i>M_r</i> = 224.26	Cell parameters from 2787 reflections
Orthorhombic, <i>Pbca</i>	θ = 2.9–27.5°
<i>a</i> = 19.5886 (14) Å	μ = 0.08 mm ⁻¹
<i>b</i> = 13.8924 (9) Å	<i>T</i> = 120 (2) K
<i>c</i> = 8.3714 (3) Å	Slab, yellow
<i>V</i> = 2278.1 (2) Å ³	0.26 × 0.14 × 0.05 mm
<i>Z</i> = 8	
<i>D_x</i> = 1.308 Mg m ⁻³	

Data collection

Bruker–Nonius FR591 rotating-anode diffractometer	1530 reflections with <i>I</i> > 2σ(<i>I</i>)
φ and ω scans	<i>R_{int}</i> = 0.074
Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 2003)	θ_{\max} = 27.5°
<i>T_{min}</i> = 0.978, <i>T_{max}</i> = 0.996	<i>h</i> = -18 → 25
11 508 measured reflections	<i>k</i> = -14 → 18
2597 independent reflections	<i>l</i> = -10 → 8

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.0647P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.054$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.130$	(Δ/σ) _{max} < 0.001
<i>S</i> = 1.00	$\Delta\rho_{\max} = 0.22 \text{ e } \text{Å}^{-3}$
2597 reflections	$\Delta\rho_{\min} = -0.21 \text{ e } \text{Å}^{-3}$
155 parameters	
H-atom parameters constrained	

Table 1

Hydrogen-bonding 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>
N10–H1...O1 ⁱ	0.88	2.04	2.8747 (19)	157

Symmetry code: (i) 1 – *x*, –*y*, –*z*.

H atoms were treated as riding atoms (C–H = 0.95 and 0.98 Å, and N–H = 0.88 Å).

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 1998); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Table 2
Comparison of selected bond lengths (Å) in some dipyrin-1-ones.

Compound	(III)	(IV)	(V)	(I)
Geometry	<i>Z-syn</i>	<i>Z-syn</i>	<i>Z-anti</i>	<i>Z-anti</i>
C4—C5	1.347 (10)	1.354 (1)	1.350 (3)	1.348 (2)
C5—C6	1.405 (10)	1.431 (1)	1.445 (3)	1.435 (2)
C1—N10 (N1)	1.380 (10)	1.353 (1)	1.376 (3)	1.370 (2)
C4—N10 (N1)	1.401 (10)	1.396 (1)	1.387 (3)	1.405 (2)
C9—N11 (N2)	1.362 (9)	1.354 (1)	1.362 (3)	1.359 (2)
C6—N11 (N2)	1.384 (9)	1.375 (1)	1.391 (3)	1.387 (2)
Reference	Cullen <i>et al.</i> (1977)	Cullen <i>et al.</i> (1979)	Cullen <i>et al.</i> (1979)	Present work

We thank the EPSRC National Crystallography Service (Southampton University) for data collection, and financial support from the SERC is acknowledged.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FA1085). Services for accessing these data are described at the back of the journal.

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supporting information

Acta Cryst. (2004). C60, o890–o892 [doi:10.1107/S0108270104026708]

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Computing details

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 1998); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

3-[(1-methylpyrrol-2-yl)methylidene]-2,3-dihydro-1H-isoindol-2-one

Crystal data

$C_{14}H_{12}N_2O$

$M_r = 224.26$

Orthorhombic, *Pbca*

$a = 19.5886$ (14) Å

$b = 13.8924$ (9) Å

$c = 8.3714$ (3) Å

$V = 2278.1$ (2) Å³

$Z = 8$

$F(000) = 944$

$D_x = 1.308$ Mg m⁻³

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

Cell parameters from 2787 reflections

$\theta = 2.9$ – 27.5°

$\mu = 0.08$ mm⁻¹

$T = 120$ K

Slab, yellow

$0.26 \times 0.14 \times 0.05$ mm

Data collection

Bruker-Nonius 95mm CCD camera on κ -goniostat diffractometer

Radiation source: Bruker-Nonius FR591 rotating-anode

Graphite monochromator

Detector resolution: 9.091 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)

$T_{\min} = 0.978$, $T_{\max} = 0.996$

11508 measured reflections

2597 independent reflections

1530 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.074$

$\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 3.0^\circ$

$h = -18 \rightarrow 25$

$k = -14 \rightarrow 18$

$l = -10 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.054$

$wR(F^2) = 0.130$

$S = 1.00$

2597 reflections

155 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0647P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.22$ e Å⁻³

$\Delta\rho_{\min} = -0.21$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.45320 (10)	0.12758 (13)	-0.0406 (2)	0.0270 (5)
C2	0.42832 (9)	0.22602 (12)	-0.06893 (18)	0.0223 (4)
C3	0.47004 (9)	0.28845 (12)	0.01674 (17)	0.0223 (4)
C4	0.52300 (9)	0.23223 (12)	0.09729 (18)	0.0228 (4)
C5	0.57387 (9)	0.26733 (13)	0.18882 (18)	0.0241 (4)
H5	0.5749	0.3355	0.1974	0.029*
C6	0.62669 (9)	0.21829 (13)	0.27542 (17)	0.0228 (4)
C7	0.64078 (9)	0.12173 (13)	0.30170 (19)	0.0270 (5)
H7	0.6158	0.0688	0.2601	0.032*
C8	0.69855 (10)	0.11603 (14)	0.40061 (19)	0.0304 (5)
H8	0.7198	0.0588	0.4377	0.036*
C9	0.71863 (10)	0.20852 (13)	0.43374 (19)	0.0299 (5)
H9	0.7564	0.2264	0.4983	0.036*
C10	0.37414 (9)	0.25857 (14)	-0.16058 (19)	0.0271 (5)
H10	0.3464	0.2151	-0.2193	0.032*
C11	0.36179 (10)	0.35676 (14)	-0.1636 (2)	0.0297 (5)
H11	0.3254	0.3814	-0.2263	0.036*
C12	0.40241 (10)	0.41964 (13)	-0.0753 (2)	0.0308 (5)
H12	0.3926	0.4866	-0.0774	0.037*
C13	0.45682 (10)	0.38681 (13)	0.01553 (19)	0.0285 (5)
H13	0.4843	0.4302	0.0752	0.034*
C14	0.68193 (11)	0.37441 (14)	0.3662 (2)	0.0338 (5)
H14A	0.7192	0.3915	0.4391	0.051*
H14B	0.6391	0.4024	0.4052	0.051*
H14C	0.6919	0.3996	0.2594	0.051*
N10	0.50882 (8)	0.13602 (10)	0.05761 (16)	0.0258 (4)
H1	0.5329	0.0868	0.0920	0.031*
N11	0.67539 (8)	0.27026 (11)	0.35879 (14)	0.0258 (4)
O1	0.42990 (7)	0.05076 (9)	-0.09345 (14)	0.0373 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0277 (12)	0.0287 (11)	0.0245 (9)	0.0000 (9)	-0.0002 (8)	-0.0017 (8)
C2	0.0228 (11)	0.0244 (10)	0.0197 (8)	0.0005 (8)	0.0036 (7)	-0.0001 (7)
C3	0.0232 (11)	0.0256 (10)	0.0181 (8)	-0.0008 (8)	0.0035 (7)	0.0007 (7)

C4	0.0260 (11)	0.0226 (10)	0.0197 (8)	-0.0015 (8)	0.0041 (8)	-0.0004 (7)
C5	0.0274 (11)	0.0235 (10)	0.0213 (8)	-0.0033 (8)	0.0036 (8)	0.0003 (7)
C6	0.0229 (11)	0.0283 (11)	0.0172 (8)	-0.0052 (8)	0.0012 (7)	-0.0012 (7)
C7	0.0288 (12)	0.0285 (11)	0.0236 (9)	-0.0034 (9)	-0.0024 (8)	-0.0019 (8)
C8	0.0313 (12)	0.0316 (11)	0.0282 (9)	0.0017 (9)	0.0004 (8)	0.0015 (8)
C9	0.0248 (11)	0.0397 (12)	0.0252 (9)	-0.0040 (9)	-0.0037 (8)	0.0003 (8)
C10	0.0247 (11)	0.0307 (12)	0.0258 (9)	-0.0017 (9)	0.0033 (8)	-0.0036 (8)
C11	0.0252 (12)	0.0330 (12)	0.0308 (10)	0.0039 (9)	-0.0006 (8)	0.0019 (8)
C12	0.0342 (12)	0.0245 (11)	0.0336 (10)	0.0053 (9)	0.0013 (9)	0.0015 (9)
C13	0.0309 (12)	0.0255 (10)	0.0289 (9)	-0.0032 (9)	0.0010 (8)	-0.0008 (8)
C14	0.0366 (13)	0.0304 (12)	0.0342 (10)	-0.0100 (10)	-0.0039 (9)	-0.0025 (8)
N10	0.0277 (10)	0.0225 (9)	0.0271 (8)	0.0026 (7)	-0.0039 (7)	0.0000 (6)
N11	0.0258 (9)	0.0279 (9)	0.0236 (7)	-0.0054 (7)	-0.0010 (6)	0.0014 (6)
O1	0.0424 (9)	0.0246 (8)	0.0448 (7)	-0.0013 (7)	-0.0136 (7)	-0.0070 (6)

Geometric parameters (Å, °)

C1—O1	1.242 (2)	C8—H8	0.9500
C1—N10	1.370 (2)	C9—N11	1.359 (2)
C1—C2	1.471 (2)	C9—H9	0.9500
C2—C10	1.385 (2)	C10—C11	1.386 (2)
C2—C3	1.391 (2)	C10—H10	0.9500
C3—C13	1.391 (2)	C11—C12	1.394 (3)
C3—C4	1.463 (2)	C11—H11	0.9500
C4—C5	1.348 (2)	C12—C13	1.386 (3)
C4—N10	1.405 (2)	C12—H12	0.9500
C5—C6	1.435 (2)	C13—H13	0.9500
C5—H5	0.9500	C14—N11	1.454 (2)
C6—N11	1.385 (2)	C14—H14A	0.9800
C6—C7	1.387 (2)	C14—H14B	0.9800
C7—C8	1.404 (3)	C14—H14C	0.9800
C7—H7	0.9500	N10—H1	0.8800
C8—C9	1.372 (2)		
O1—C1—N10	125.42 (17)	C8—C9—H9	125.7
O1—C1—C2	128.28 (17)	C2—C10—C11	117.73 (17)
N10—C1—C2	106.30 (15)	C2—C10—H10	121.1
C10—C2—C3	122.15 (17)	C11—C10—H10	121.1
C10—C2—C1	130.29 (16)	C10—C11—C12	120.49 (18)
C3—C2—C1	107.57 (15)	C10—C11—H11	119.8
C13—C3—C2	119.96 (16)	C12—C11—H11	119.8
C13—C3—C4	131.29 (16)	C13—C12—C11	121.58 (18)
C2—C3—C4	108.75 (15)	C13—C12—H12	119.2
C5—C4—N10	128.65 (16)	C11—C12—H12	119.2
C5—C4—C3	126.37 (17)	C12—C13—C3	118.07 (17)
N10—C4—C3	104.99 (14)	C12—C13—H13	121.0
C4—C5—C6	130.40 (17)	C3—C13—H13	121.0
C4—C5—H5	114.8	N11—C14—H14A	109.5

C6—C5—H5	114.8	N11—C14—H14B	109.5
N11—C6—C7	106.68 (15)	H14A—C14—H14B	109.5
N11—C6—C5	120.24 (16)	N11—C14—H14C	109.5
C7—C6—C5	133.05 (16)	H14A—C14—H14C	109.5
C6—C7—C8	107.96 (16)	H14B—C14—H14C	109.5
C6—C7—H7	126.0	C1—N10—C4	112.37 (15)
C8—C7—H7	126.0	C1—N10—H1	123.8
C9—C8—C7	107.31 (17)	C4—N10—H1	123.8
C9—C8—H8	126.3	C9—N11—C6	109.45 (16)
C7—C8—H8	126.3	C9—N11—C14	123.60 (15)
N11—C9—C8	108.60 (16)	C6—N11—C14	126.95 (15)
N11—C9—H9	125.7		
O1—C1—C2—C10	-0.6 (3)	C7—C8—C9—N11	0.0 (2)
N10—C1—C2—C10	179.20 (16)	C3—C2—C10—C11	-0.7 (2)
O1—C1—C2—C3	179.22 (17)	C1—C2—C10—C11	179.10 (16)
N10—C1—C2—C3	-0.99 (18)	C2—C10—C11—C12	-0.8 (3)
C10—C2—C3—C13	1.8 (2)	C10—C11—C12—C13	1.2 (3)
C1—C2—C3—C13	-178.07 (14)	C11—C12—C13—C3	-0.1 (3)
C10—C2—C3—C4	-178.68 (15)	C2—C3—C13—C12	-1.3 (2)
C1—C2—C3—C4	1.49 (18)	C4—C3—C13—C12	179.23 (16)
C13—C3—C4—C5	-2.0 (3)	O1—C1—N10—C4	179.88 (17)
C2—C3—C4—C5	178.46 (16)	C2—C1—N10—C4	0.08 (19)
C13—C3—C4—N10	178.07 (17)	C5—C4—N10—C1	-179.07 (16)
C2—C3—C4—N10	-1.42 (17)	C3—C4—N10—C1	0.80 (18)
N10—C4—C5—C6	-2.0 (3)	C8—C9—N11—C6	-0.19 (19)
C3—C4—C5—C6	178.18 (16)	C8—C9—N11—C14	-179.25 (15)
C4—C5—C6—N11	178.05 (16)	C7—C6—N11—C9	0.35 (18)
C4—C5—C6—C7	-4.3 (3)	C5—C6—N11—C9	178.52 (14)
N11—C6—C7—C8	-0.37 (18)	C7—C6—N11—C14	179.38 (16)
C5—C6—C7—C8	-178.21 (17)	C5—C6—N11—C14	-2.4 (2)
C6—C7—C8—C9	0.3 (2)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N10—H1 \cdots O1 ⁱ	0.88	2.04	2.8747 (19)	157

Symmetry code: (i) $-x+1, -y, -z$.