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5''-Benzylidene-5-chloro-1',1''-dimethyl-4'-phenyldispiro[indoline-3,2'-pyrrolidine-3',3''-piperidine]-2,4''-dione

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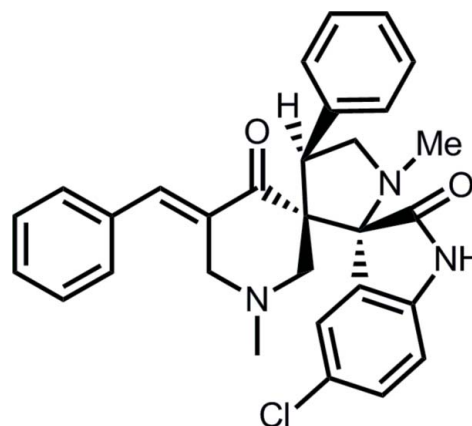
Received 2 December 2013; accepted 2 December 2013

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.052; wR factor = 0.137; data-to-parameter ratio = 17.9.

The title compound, $\text{C}_{30}\text{H}_{28}\text{ClN}_3\text{O}_2$, features two spiro links, one connecting the piperidine and pyrrolidine rings, and the other connecting the pyrrolidine ring and indole residue. The configuration about the ethene bond is *E*. The piperidine ring adopts a half-chair conformation where the C atom connected to the spiro-C atom lies 0.713 (3) Å out of the plane of the remaining five atoms (r.m.s. deviation = 0.086 Å). The pyrrolidine ring has an envelope conformation with the flap atom being the methylene C atom. Centrosymmetric eight-membered $\{\cdots\text{HNCO}\}_2$ amide synthons feature in the crystal packing. These are consolidated into a three-dimensional architecture by phenyl-pyrrolidine $\text{C}-\text{H}\cdots\text{N}$ and chlorobenzene-pyrrolidine-bound phenyl $\text{C}-\text{H}\cdots\pi$ interactions.

Related literature

For the biological activity of related spiro-pyrrolidine analogues, see: Girgis *et al.* (2012); Kumar *et al.* (2008). For related structural studies, see: Moustafa *et al.* (2012). For the synthesis of the precursor molecule, see: Al-Omary *et al.* (2012).



Experimental

Crystal data

$\text{C}_{30}\text{H}_{28}\text{ClN}_3\text{O}_2$ $V = 2562.20$ (14) Å³
 $M_r = 498.00$ $Z = 4$
 Monoclinic, $P2_1/n$ Mo $K\alpha$ radiation
 $a = 10.5028$ (3) Å $\mu = 0.18$ mm⁻¹
 $b = 20.4117$ (6) Å $T = 293$ K
 $c = 11.9951$ (4) Å $0.52 \times 0.22 \times 0.15$ mm
 $\beta = 94.877$ (1)°

Data collection

Nonius 590 KappaCCD diffractometer 5842 independent reflections
 10395 measured reflections 2547 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.066$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$ 327 parameters
 $wR(F^2) = 0.137$ H-atom parameters constrained
 $S = 0.94$ $\Delta\rho_{\text{max}} = 0.18$ e Å⁻³
 5842 reflections $\Delta\rho_{\text{min}} = -0.35$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the $\text{C}25-\text{C}30$ ring.

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N}3-\text{H}3n\cdots\text{O}2^i$	0.86	2.01	2.854 (3)	165
$\text{C}14-\text{H}14\cdots\text{N}2^{ii}$	0.93	2.58	3.480 (4)	163
$\text{C}20-\text{H}20\cdots\text{Cg}1^{iii}$	0.93	2.70	3.268 (3)	121

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

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, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *pubCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5367).

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supplementary materials

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5''-Benzylidene-5-chloro-1',1''-dimethyl-4'-phenyldispiro[indoline-3,2'-pyrrolidine-3',3''-piperidine]-2,4''-dione

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1. Introduction

2. Experimental

2.1. Synthesis and crystallization

A mixture of equimolar amounts of 3*E*,5*E*-1-methyl-3,5-bis(phenylmethylidene)-4-piperidone (5 mmol), prepared by a literature procedure (Al-Omary *et al.*, 2012), 5-chloroisatin and sarcosine in absolute ethanol (25 ml) was boiled under reflux (TLC monitoring). The separated solid was collected and crystallized from *n*-butanol affording (I). Reaction time 9 h. Colourless crystals. *M.pt.*: 512–514 K. Yield 88%. Anal. Calcd. for C₃₀H₂₈ClN₃O₂ (498.03): C, 72.35; H, 5.67; N, 8.44. Found: C, 72.56; H, 5.81; N, 8.67. IR: $\nu_{\max}/\text{cm}^{-1}$: 3168 (N—H); 1688 (C=O); 1597, 1457 (C=C).

2.2. Refinement

The C-bound H atoms were geometrically placed (C—H = 0.93–0.98 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}(\text{C})$. The N-bound H-atoms were treated similarly with N—H = 0.86 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$.

3. Results and discussion

In connection with on-going studies of spiro-pyrrolidine derivatives (Girgis *et al.* 2012; Moustafa *et al.* 2012), the title compound, (I), was synthesised and characterised crystallographically. These compounds have biological activity and the structure of the skeltal structure is well established (Kumar *et al.* 2008).

There are two spiro links in the molecule, Fig. 1, i) where the piperidine and pyrrolidine rings are connected at C1, and ii) where the pyrrolidine ring and indole residue are connected at C6. The phenylmethylidene functional group is connected to the piperidine ring at position C4 while the pyrrolidine-bound aryl ring is attached at C8. The conformation about the C4=C11 double bond is *E*. The sum of the angles around the piperidine-N1 atom is approximately 333° confirming its *sp*³ character. The piperidine ring adopts a half-chair conformation where the C2 atom lies 0.713 (3) Å out of the plane of the remaining five atoms (r.m.s. deviation = 0.086 Å). The C6 and C8 atoms occupy axial and equatorial positions with respect to the piperidine ring, the phenylmethylidene residue occupies an equatorial position, and the N-bound methyl substituent is equatorial. The pyrrolidine ring has an envelope conformation with the flap atom being the C7 atom which lies 0.648 (3) Å out of the plane of the remaining four atoms (r.m.s. deviation = 0.026 Å). Finally, the indole fused ring system is planar with a r.m.s. deviation = 0.051 Å.

The most prominent feature of the crystal packing of is the formation of centrosymmetric eight-membered { $\cdots\text{HNCO}$ }₂ synthons owing to the self-association of molecules *via* hydrogen bonding between amide groups, Table 1. The dimers are connected into a supramolecular chain parallel to the *b* axis by phenyl-C—H \cdots N (pyrrolidine) interactions and these are consolidated into a three-dimensional architecture by (chlorobenzene)C—H \cdots π (pyrrolidine-bound phenyl), edge-to-

face, interactions; a view of the unit cell contents is shown in Fig. 2.

Computing details

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); 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, 2012) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

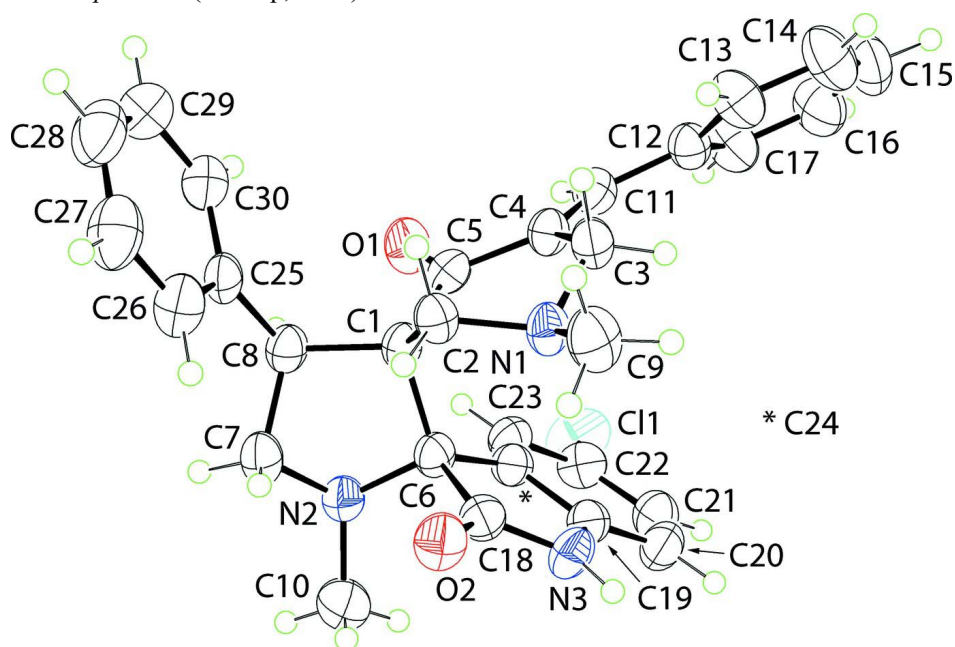
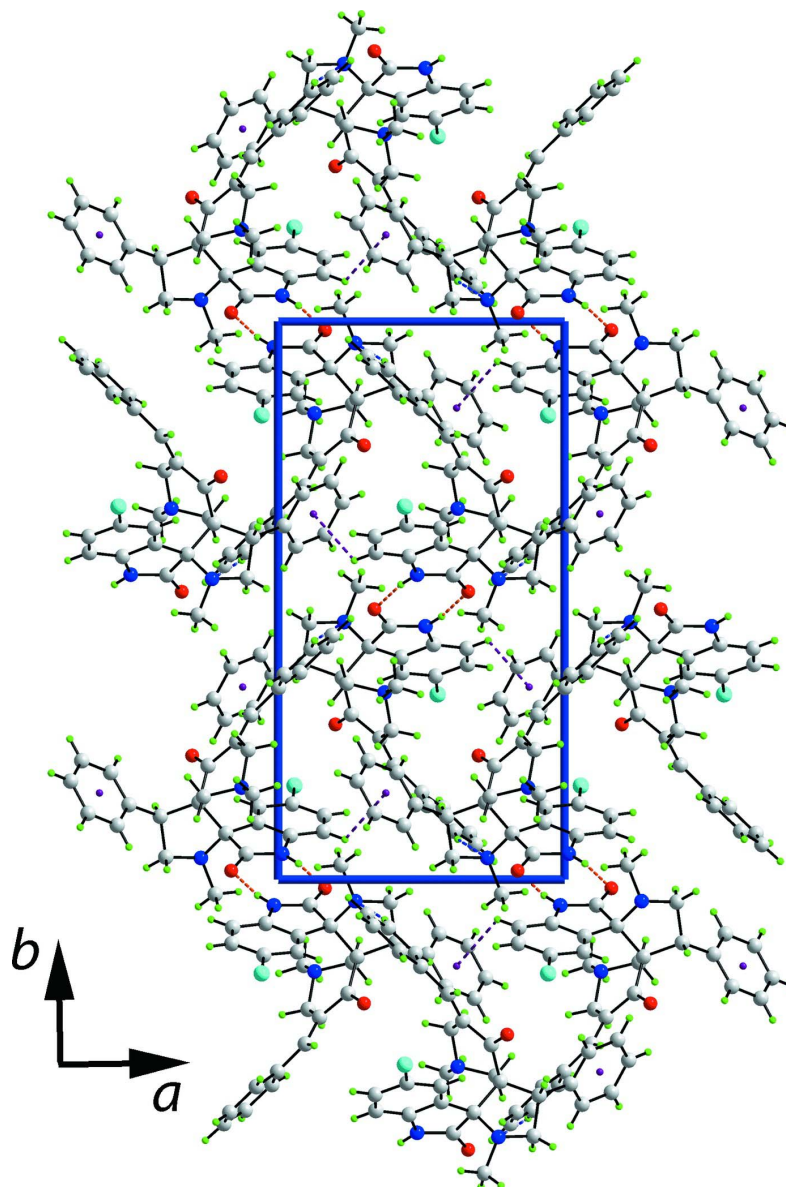


Figure 1

The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

**Figure 2**

A view in projection down the c axis of the unit-cell contents for (I). The N—H \cdots O, C—H \cdots N and C—H \cdots π interactions are shown as orange, blue and purple dashed lines, respectively.

5''-Benzylidene-5-chloro-1',1''-dimethyl-4'-phenyldispiro[indoline-3,2'-pyrrolidine-3',3''-piperidine]-2,4''-dione

Crystal data

$C_{30}H_{28}ClN_3O_2$

$M_r = 498.00$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 10.5028(3) \text{ \AA}$

$b = 20.4117(6) \text{ \AA}$

$c = 11.9951(4) \text{ \AA}$

$\beta = 94.877(1)^\circ$

$V = 2562.20(14) \text{ \AA}^3$

$Z = 4$

$F(000) = 1048$

$D_x = 1.291 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 5029 reflections

$\theta = 2.9\text{--}27.5^\circ$

$\mu = 0.18 \text{ mm}^{-1}$

$T = 293$ K
Block, colourless

$0.52 \times 0.22 \times 0.15$ mm

Data collection

Nonius 590 KappaCCD
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
 φ and ω scans
10395 measured reflections
5842 independent reflections

2547 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.066$
 $\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 3.2^\circ$
 $h = -13 \rightarrow 13$
 $k = -24 \rightarrow 26$
 $l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.137$
 $S = 0.94$
5842 reflections
327 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
 $w = 1/[\sigma^2(F_o^2) + (0.0569P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.18 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.35 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	1.05716 (7)	0.16826 (4)	-0.06845 (7)	0.0777 (3)
O1	0.70008 (15)	0.22207 (8)	0.17686 (15)	0.0518 (5)
O2	0.83631 (15)	0.01572 (8)	0.45243 (14)	0.0501 (5)
N1	0.87248 (16)	0.16478 (9)	0.47693 (16)	0.0395 (5)
N2	0.73065 (16)	0.03868 (9)	0.21124 (16)	0.0425 (5)
N3	1.01454 (17)	0.04625 (9)	0.36752 (17)	0.0441 (5)
H3n	1.0711	0.0286	0.4142	0.053*
C1	0.72419 (19)	0.13839 (11)	0.31799 (19)	0.0354 (6)
C2	0.7398 (2)	0.14837 (11)	0.44474 (19)	0.0389 (6)
H2A	0.6845	0.1835	0.4659	0.047*
H2B	0.7165	0.1086	0.4825	0.047*
C3	0.8953 (2)	0.23259 (11)	0.4469 (2)	0.0425 (6)
H3A	0.9855	0.2423	0.4618	0.051*
H3B	0.8485	0.2612	0.4935	0.051*
C4	0.8552 (2)	0.24660 (11)	0.32605 (19)	0.0370 (6)
C5	0.7557 (2)	0.20414 (11)	0.2654 (2)	0.0377 (6)

C6	0.81725 (19)	0.08258 (11)	0.27869 (19)	0.0364 (6)
C7	0.6081 (2)	0.04033 (11)	0.2593 (2)	0.0463 (7)
H7A	0.6121	0.0177	0.3307	0.056*
H7B	0.5409	0.0212	0.2091	0.056*
C8	0.58871 (19)	0.11328 (11)	0.2732 (2)	0.0391 (6)
H8	0.5710	0.1315	0.1979	0.047*
C9	0.9106 (2)	0.15392 (13)	0.5956 (2)	0.0562 (7)
H9A	0.8554	0.1784	0.6401	0.084*
H9B	0.9972	0.1681	0.6124	0.084*
H9C	0.9041	0.1081	0.6124	0.084*
C10	0.7805 (2)	-0.02622 (13)	0.1881 (2)	0.0615 (8)
H10A	0.8615	-0.0219	0.1573	0.092*
H10B	0.7216	-0.0485	0.1354	0.092*
H10C	0.7911	-0.0510	0.2562	0.092*
C11	0.9050 (2)	0.29475 (11)	0.2683 (2)	0.0423 (6)
H11	0.8740	0.2975	0.1935	0.051*
C12	1.0017 (2)	0.34419 (11)	0.3053 (2)	0.0408 (6)
C13	1.0215 (2)	0.36746 (13)	0.4136 (2)	0.0546 (7)
H13	0.9709	0.3519	0.4678	0.066*
C14	1.1149 (3)	0.41337 (14)	0.4432 (3)	0.0657 (8)
H14	1.1273	0.4279	0.5168	0.079*
C15	1.1892 (3)	0.43743 (14)	0.3646 (3)	0.0658 (8)
H15	1.2537	0.4674	0.3849	0.079*
C16	1.1685 (3)	0.41725 (14)	0.2555 (3)	0.0667 (9)
H16	1.2169	0.4347	0.2012	0.080*
C17	1.0760 (2)	0.37125 (12)	0.2266 (2)	0.0540 (7)
H17	1.0627	0.3579	0.1524	0.065*
C18	0.8882 (2)	0.04577 (11)	0.3795 (2)	0.0407 (6)
C19	1.0428 (2)	0.07885 (11)	0.2700 (2)	0.0385 (6)
C20	1.1587 (2)	0.08588 (12)	0.2266 (2)	0.0485 (7)
H20	1.2336	0.0717	0.2662	0.058*
C21	1.1613 (2)	0.11466 (12)	0.1225 (2)	0.0524 (7)
H21	1.2388	0.1201	0.0915	0.063*
C22	1.0499 (2)	0.13532 (12)	0.0645 (2)	0.0478 (7)
C23	0.9324 (2)	0.12955 (11)	0.1090 (2)	0.0435 (6)
H23	0.8577	0.1440	0.0693	0.052*
C24	0.9298 (2)	0.10180 (11)	0.2136 (2)	0.0368 (6)
C25	0.4792 (2)	0.13400 (12)	0.3399 (2)	0.0394 (6)
C26	0.4383 (2)	0.09737 (14)	0.4274 (2)	0.0544 (7)
H26	0.4793	0.0582	0.4477	0.065*
C27	0.3363 (2)	0.11858 (17)	0.4853 (2)	0.0652 (8)
H27	0.3094	0.0935	0.5436	0.078*
C28	0.2756 (3)	0.17634 (18)	0.4566 (3)	0.0722 (9)
H28	0.2083	0.1907	0.4959	0.087*
C29	0.3145 (3)	0.21279 (15)	0.3698 (3)	0.0701 (9)
H29	0.2731	0.2518	0.3497	0.084*
C30	0.4153 (2)	0.19161 (13)	0.3119 (2)	0.0553 (7)
H30	0.4406	0.2167	0.2529	0.066*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0823 (5)	0.0892 (6)	0.0655 (6)	0.0142 (4)	0.0285 (4)	0.0289 (4)
O1	0.0515 (10)	0.0547 (11)	0.0467 (12)	-0.0077 (8)	-0.0102 (9)	0.0147 (9)
O2	0.0486 (10)	0.0543 (11)	0.0466 (12)	-0.0018 (9)	-0.0003 (9)	0.0159 (9)
N1	0.0364 (11)	0.0465 (13)	0.0341 (13)	-0.0047 (9)	-0.0047 (9)	0.0055 (10)
N2	0.0362 (11)	0.0430 (13)	0.0475 (14)	-0.0005 (10)	-0.0004 (10)	-0.0057 (10)
N3	0.0350 (11)	0.0538 (14)	0.0420 (14)	0.0067 (10)	-0.0051 (10)	0.0087 (10)
C1	0.0314 (12)	0.0416 (15)	0.0326 (15)	-0.0011 (11)	-0.0019 (10)	0.0045 (12)
C2	0.0385 (13)	0.0408 (15)	0.0369 (16)	-0.0052 (11)	0.0010 (11)	0.0029 (12)
C3	0.0380 (13)	0.0440 (16)	0.0445 (17)	-0.0070 (11)	-0.0019 (11)	0.0012 (13)
C4	0.0345 (13)	0.0433 (15)	0.0328 (15)	-0.0015 (11)	0.0004 (11)	0.0029 (12)
C5	0.0338 (13)	0.0426 (15)	0.0365 (17)	0.0044 (11)	0.0027 (12)	0.0039 (13)
C6	0.0344 (12)	0.0389 (14)	0.0349 (15)	-0.0007 (11)	-0.0032 (11)	0.0030 (11)
C7	0.0381 (14)	0.0516 (17)	0.0479 (17)	-0.0072 (12)	-0.0034 (12)	-0.0004 (13)
C8	0.0333 (12)	0.0450 (16)	0.0378 (15)	-0.0002 (11)	-0.0033 (11)	0.0037 (12)
C9	0.0597 (16)	0.0632 (19)	0.0434 (19)	-0.0088 (14)	-0.0096 (13)	0.0069 (14)
C10	0.0593 (17)	0.0547 (19)	0.070 (2)	-0.0006 (14)	0.0051 (15)	-0.0124 (15)
C11	0.0415 (14)	0.0472 (16)	0.0379 (16)	0.0002 (13)	0.0014 (12)	-0.0011 (13)
C12	0.0403 (13)	0.0411 (15)	0.0405 (17)	-0.0034 (12)	0.0010 (12)	0.0031 (13)
C13	0.0698 (18)	0.0511 (17)	0.0429 (19)	-0.0159 (15)	0.0052 (14)	0.0042 (14)
C14	0.085 (2)	0.060 (2)	0.049 (2)	-0.0203 (17)	-0.0112 (16)	0.0024 (16)
C15	0.0552 (18)	0.060 (2)	0.080 (3)	-0.0204 (14)	-0.0069 (17)	0.0028 (18)
C16	0.0612 (18)	0.066 (2)	0.076 (3)	-0.0189 (16)	0.0223 (17)	-0.0040 (18)
C17	0.0590 (16)	0.0555 (17)	0.0490 (19)	-0.0108 (15)	0.0136 (14)	-0.0047 (15)
C18	0.0404 (15)	0.0385 (15)	0.0424 (17)	0.0005 (12)	-0.0014 (12)	0.0041 (13)
C19	0.0393 (14)	0.0391 (15)	0.0363 (16)	0.0001 (11)	-0.0004 (12)	-0.0020 (12)
C20	0.0363 (14)	0.0548 (17)	0.0537 (19)	0.0049 (12)	0.0003 (13)	-0.0009 (14)
C21	0.0422 (15)	0.0568 (18)	0.060 (2)	-0.0043 (13)	0.0140 (14)	-0.0018 (15)
C22	0.0522 (16)	0.0494 (17)	0.0430 (18)	0.0022 (13)	0.0120 (14)	0.0046 (13)
C23	0.0407 (14)	0.0439 (16)	0.0450 (17)	0.0038 (12)	-0.0009 (12)	0.0048 (13)
C24	0.0375 (13)	0.0371 (14)	0.0354 (16)	0.0005 (11)	0.0001 (11)	-0.0005 (12)
C25	0.0291 (12)	0.0450 (16)	0.0430 (17)	-0.0059 (12)	-0.0040 (11)	-0.0003 (13)
C26	0.0436 (15)	0.0665 (19)	0.0527 (19)	-0.0041 (14)	0.0013 (14)	0.0088 (15)
C27	0.0514 (17)	0.095 (3)	0.050 (2)	-0.0111 (17)	0.0109 (15)	-0.0051 (17)
C28	0.0448 (17)	0.096 (3)	0.077 (3)	-0.0018 (18)	0.0135 (16)	-0.030 (2)
C29	0.0516 (18)	0.063 (2)	0.097 (3)	0.0026 (15)	0.0102 (18)	-0.0110 (19)
C30	0.0409 (15)	0.0537 (18)	0.071 (2)	-0.0044 (14)	0.0042 (14)	-0.0004 (15)

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

C11—C22	1.738 (3)	C10—H10C	0.9600
O1—C5	1.224 (3)	C11—C12	1.473 (3)
O2—C18	1.232 (3)	C11—H11	0.9300
N1—C2	1.453 (3)	C12—C13	1.382 (3)
N1—C3	1.455 (3)	C12—C17	1.390 (3)
N1—C9	1.463 (3)	C13—C14	1.381 (4)
N2—C7	1.454 (3)	C13—H13	0.9300
N2—C10	1.460 (3)	C14—C15	1.366 (4)

N2—C6	1.470 (3)	C14—H14	0.9300
N3—C18	1.347 (3)	C15—C16	1.371 (4)
N3—C19	1.400 (3)	C15—H15	0.9300
N3—H3n	0.8600	C16—C17	1.374 (3)
C1—C5	1.531 (3)	C16—H16	0.9300
C1—C2	1.529 (3)	C17—H17	0.9300
C1—C8	1.564 (3)	C19—C20	1.371 (3)
C1—C6	1.598 (3)	C19—C24	1.396 (3)
C2—H2A	0.9700	C20—C21	1.382 (3)
C2—H2B	0.9700	C20—H20	0.9300
C3—C4	1.502 (3)	C21—C22	1.375 (3)
C3—H3A	0.9700	C21—H21	0.9300
C3—H3B	0.9700	C22—C23	1.391 (3)
C4—C11	1.334 (3)	C23—C24	1.380 (3)
C4—C5	1.498 (3)	C23—H23	0.9300
C6—C24	1.522 (3)	C25—C30	1.381 (3)
C6—C18	1.559 (3)	C25—C26	1.386 (3)
C7—C8	1.514 (3)	C26—C27	1.394 (3)
C7—H7A	0.9700	C26—H26	0.9300
C7—H7B	0.9700	C27—C28	1.370 (4)
C8—C25	1.516 (3)	C27—H27	0.9300
C8—H8	0.9800	C28—C29	1.370 (4)
C9—H9A	0.9600	C28—H28	0.9300
C9—H9B	0.9600	C29—C30	1.384 (4)
C9—H9C	0.9600	C29—H29	0.9300
C10—H10A	0.9600	C30—H30	0.9300
C10—H10B	0.9600		
C2—N1—C3	109.14 (18)	H10B—C10—H10C	109.5
C2—N1—C9	113.61 (17)	C4—C11—C12	129.7 (2)
C3—N1—C9	110.35 (18)	C4—C11—H11	115.2
C7—N2—C10	116.12 (18)	C12—C11—H11	115.2
C7—N2—C6	107.14 (17)	C13—C12—C17	117.0 (2)
C10—N2—C6	116.30 (18)	C13—C12—C11	124.3 (2)
C18—N3—C19	111.96 (19)	C17—C12—C11	118.7 (2)
C18—N3—H3n	124.0	C14—C13—C12	121.5 (2)
C19—N3—H3n	124.0	C14—C13—H13	119.3
C5—C1—C2	106.50 (19)	C12—C13—H13	119.3
C5—C1—C8	111.60 (18)	C15—C14—C13	120.1 (3)
C2—C1—C8	113.73 (17)	C15—C14—H14	120.0
C5—C1—C6	110.15 (16)	C13—C14—H14	120.0
C2—C1—C6	111.83 (18)	C14—C15—C16	119.8 (3)
C8—C1—C6	103.10 (17)	C14—C15—H15	120.1
N1—C2—C1	108.31 (17)	C16—C15—H15	120.1
N1—C2—H2A	110.0	C15—C16—C17	119.9 (3)
C1—C2—H2A	110.0	C15—C16—H16	120.1
N1—C2—H2B	110.0	C17—C16—H16	120.1
C1—C2—H2B	110.0	C16—C17—C12	121.7 (3)
H2A—C2—H2B	108.4	C16—C17—H17	119.2

N1—C3—C4	112.41 (19)	C12—C17—H17	119.2
N1—C3—H3A	109.1	O2—C18—N3	125.4 (2)
C4—C3—H3A	109.1	O2—C18—C6	125.4 (2)
N1—C3—H3B	109.1	N3—C18—C6	108.9 (2)
C4—C3—H3B	109.1	C20—C19—C24	121.8 (2)
H3A—C3—H3B	107.9	C20—C19—N3	128.7 (2)
C11—C4—C5	117.5 (2)	C24—C19—N3	109.40 (19)
C11—C4—C3	123.4 (2)	C19—C20—C21	118.2 (2)
C5—C4—C3	119.0 (2)	C19—C20—H20	120.9
O1—C5—C4	120.7 (2)	C21—C20—H20	120.9
O1—C5—C1	121.0 (2)	C22—C21—C20	120.4 (2)
C4—C5—C1	118.3 (2)	C22—C21—H21	119.8
N2—C6—C24	110.25 (18)	C20—C21—H21	119.8
N2—C6—C18	111.47 (18)	C21—C22—C23	121.6 (2)
C24—C6—C18	100.44 (17)	C21—C22—C11	118.77 (19)
N2—C6—C1	103.40 (16)	C23—C22—C11	119.6 (2)
C24—C6—C1	119.26 (18)	C24—C23—C22	118.1 (2)
C18—C6—C1	112.25 (18)	C24—C23—H23	121.0
N2—C7—C8	101.46 (17)	C22—C23—H23	121.0
N2—C7—H7A	111.5	C23—C24—C19	119.8 (2)
C8—C7—H7A	111.5	C23—C24—C6	130.4 (2)
N2—C7—H7B	111.5	C19—C24—C6	109.3 (2)
C8—C7—H7B	111.5	C30—C25—C26	117.9 (2)
H7A—C7—H7B	109.3	C30—C25—C8	118.9 (2)
C7—C8—C25	116.62 (19)	C26—C25—C8	123.2 (2)
C7—C8—C1	103.47 (17)	C25—C26—C27	120.7 (3)
C25—C8—C1	115.89 (19)	C25—C26—H26	119.7
C7—C8—H8	106.7	C27—C26—H26	119.7
C25—C8—H8	106.7	C28—C27—C26	120.2 (3)
C1—C8—H8	106.7	C28—C27—H27	119.9
N1—C9—H9A	109.5	C26—C27—H27	119.9
N1—C9—H9B	109.5	C29—C28—C27	119.7 (3)
H9A—C9—H9B	109.5	C29—C28—H28	120.1
N1—C9—H9C	109.5	C27—C28—H28	120.1
H9A—C9—H9C	109.5	C28—C29—C30	120.1 (3)
H9B—C9—H9C	109.5	C28—C29—H29	119.9
N2—C10—H10A	109.5	C30—C29—H29	119.9
N2—C10—H10B	109.5	C25—C30—C29	121.4 (3)
H10A—C10—H10B	109.5	C25—C30—H30	119.3
N2—C10—H10C	109.5	C29—C30—H30	119.3
H10A—C10—H10C	109.5		
C3—N1—C2—C1	76.1 (2)	C11—C12—C13—C14	179.2 (2)
C9—N1—C2—C1	-160.35 (19)	C12—C13—C14—C15	0.8 (4)
C5—C1—C2—N1	-62.7 (2)	C13—C14—C15—C16	1.8 (4)
C8—C1—C2—N1	173.98 (18)	C14—C15—C16—C17	-2.2 (4)
C6—C1—C2—N1	57.7 (2)	C15—C16—C17—C12	0.0 (4)
C2—N1—C3—C4	-53.4 (2)	C13—C12—C17—C16	2.4 (4)
C9—N1—C3—C4	-178.89 (18)	C11—C12—C17—C16	-179.5 (2)

N1—C3—C4—C11	-155.0 (2)	C19—N3—C18—O2	173.4 (2)
N1—C3—C4—C5	24.1 (3)	C19—N3—C18—C6	-0.1 (3)
C11—C4—C5—O1	-17.4 (3)	N2—C6—C18—O2	-56.8 (3)
C3—C4—C5—O1	163.4 (2)	C24—C6—C18—O2	-173.6 (2)
C11—C4—C5—C1	163.4 (2)	C1—C6—C18—O2	58.7 (3)
C3—C4—C5—C1	-15.7 (3)	N2—C6—C18—N3	116.7 (2)
C2—C1—C5—O1	-145.8 (2)	C24—C6—C18—N3	0.0 (2)
C8—C1—C5—O1	-21.2 (3)	C1—C6—C18—N3	-127.80 (19)
C6—C1—C5—O1	92.7 (2)	C18—N3—C19—C20	-175.8 (2)
C2—C1—C5—C4	33.3 (2)	C18—N3—C19—C24	0.3 (3)
C8—C1—C5—C4	157.97 (19)	C24—C19—C20—C21	-2.2 (4)
C6—C1—C5—C4	-88.1 (2)	N3—C19—C20—C21	173.5 (2)
C7—N2—C6—C24	-160.87 (19)	C19—C20—C21—C22	-0.2 (4)
C10—N2—C6—C24	67.3 (3)	C20—C21—C22—C23	1.6 (4)
C7—N2—C6—C18	88.5 (2)	C20—C21—C22—C11	-177.5 (2)
C10—N2—C6—C18	-43.3 (3)	C21—C22—C23—C24	-0.5 (4)
C7—N2—C6—C1	-32.3 (2)	C11—C22—C23—C24	178.52 (19)
C10—N2—C6—C1	-164.09 (19)	C22—C23—C24—C19	-1.8 (3)
C5—C1—C6—N2	-114.14 (19)	C22—C23—C24—C6	-173.0 (2)
C2—C1—C6—N2	127.64 (18)	C20—C19—C24—C23	3.2 (3)
C8—C1—C6—N2	5.1 (2)	N3—C19—C24—C23	-173.2 (2)
C5—C1—C6—C24	8.6 (3)	C20—C19—C24—C6	176.1 (2)
C2—C1—C6—C24	-109.6 (2)	N3—C19—C24—C6	-0.3 (3)
C8—C1—C6—C24	127.8 (2)	N2—C6—C24—C23	54.4 (3)
C5—C1—C6—C18	125.6 (2)	C18—C6—C24—C23	172.1 (2)
C2—C1—C6—C18	7.4 (2)	C1—C6—C24—C23	-64.9 (3)
C8—C1—C6—C18	-115.19 (19)	N2—C6—C24—C19	-117.5 (2)
C10—N2—C7—C8	179.1 (2)	C18—C6—C24—C19	0.2 (2)
C6—N2—C7—C8	47.2 (2)	C1—C6—C24—C19	123.2 (2)
N2—C7—C8—C25	-170.10 (19)	C7—C8—C25—C30	-147.6 (2)
N2—C7—C8—C1	-41.6 (2)	C1—C8—C25—C30	90.2 (3)
C5—C1—C8—C7	140.24 (19)	C7—C8—C25—C26	31.5 (3)
C2—C1—C8—C7	-99.2 (2)	C1—C8—C25—C26	-90.7 (3)
C6—C1—C8—C7	22.0 (2)	C30—C25—C26—C27	-0.4 (4)
C5—C1—C8—C25	-90.8 (2)	C8—C25—C26—C27	-179.5 (2)
C2—C1—C8—C25	29.7 (3)	C25—C26—C27—C28	-0.4 (4)
C6—C1—C8—C25	150.97 (19)	C26—C27—C28—C29	0.8 (4)
C5—C4—C11—C12	179.0 (2)	C27—C28—C29—C30	-0.5 (4)
C3—C4—C11—C12	-1.9 (4)	C26—C25—C30—C29	0.7 (4)
C4—C11—C12—C13	-28.2 (4)	C8—C25—C30—C29	179.9 (2)
C4—C11—C12—C17	153.9 (2)	C28—C29—C30—C25	-0.3 (4)
C17—C12—C13—C14	-2.9 (4)		

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of the C25—C30 ring.

<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>
N3—H3 <i>n</i> ···O2 ⁱ	0.86	2.01	2.854 (3)	165

C14—H14 \cdots N2 ⁱⁱ	0.93	2.58	3.480 (4)	163
C20—H20 \cdots Cg1 ⁱⁱⁱ	0.93	2.70	3.268 (3)	121

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