Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5″-Benzylidene-1″-methyl-4′,5′-diphenyl-1*H*-indole-3-spiro-2′-pyrrolidine-3′-spiro-3″-piperidine-2,4″-dione

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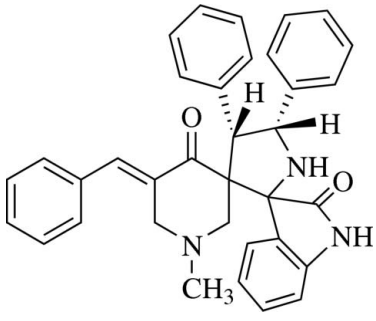
Received 7 May 2007; accepted 28 May 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.051; wR factor = 0.143; data-to-parameter ratio = 15.1.

In the title compound, $C_{35}H_{34}N_{10}O_2S_2$, the six-membered heterocyclic ring adopts a half-chair conformation, while the pyrrolidine five-membered ring exists in an envelope conformation. However the other five-membered ring in the oxindole maintains planarity. In the crystal structure, molecules form N—H···O hydrogen-bonded dimers around inversion centres. The structure is further stabilized by a C—H···π interaction.

Related literature

For general background, see: Jiang *et al.* (2006); Lundahl *et al.* (1972); Cravotto *et al.* (2001); Dimmock *et al.* (2003); Abignente & Biniecka-Picazio (1977). For puckering analysis of rings, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{35}H_{31}N_3O_2$
M_r = 525.63
Triclinic, $P\overline{1}$
a = 10.194 (4) Å
b = 10.681 (5) Å
c = 15.352 (8) Å
α = 80.76 (4)°
β = 80.31 (2)°
γ = 72.71 (5)°
V = 1562.4 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 293 (2) K
0.25 × 0.21 × 0.18 mm

Data collection

Nonius MACH3 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.968, T_{max} = 0.995
6511 measured reflections
5494 independent reflections
2506 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.143
S = 0.89
5494 reflections
363 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

*Cg*1 is the centroid of the C17–C22 phenyl ring.

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O2^{i}	0.86	2.10	2.950 (3)	170
C31—H31···*Cg*1^{ii}	0.93	2.85	3.778 (5)	175

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXTL/PC* (Bruker, 2000); program(s) used to refine structure: *SHELXTL/PC*; molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL/PC*.

SPR and BRK thank the Principal and Management of Devanga Arts College, Aruppukottai.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2320).

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supplementary materials

Acta Cryst. (2007). E**63**, o3221 [**doi:10.1107/S1600536807025822**]

5''-Benzylidene-1''-methyl-4',5'-diphenyl-1*H*-indole-3-spiro-2'-pyrrolidine-3'-spiro-3''-piperidine-2,4''-dione

B. R. Kumar, S. Pandiarajan, V. Ramakrishnan, R. R. Kumar and S. Perumal

Comment

1,3-Dipolar cycloadditions are versatile synthetic strategies employed for the construction of five-membered heterocycles (Jiang *et al.*, 2006). 1,3-dipolar cycloadditions of azomethine ylides to alkenes afford pyrrolidines, which exhibit important biological properties (Lundahl *et al.*,1972). The extensive use of isatin as a precursor for 1,3-dipolar cycloadditions stems from the fact that it generates a wide variety of azomethine ylides with primary and secondary amines, These can be trapped with suitable dipolarophiles (Cravotto *et al.*, 2001). The piperidone sub-structure is a versatile entity in synthetic organic chemistry due to its pharmacological properties. Such compounds display potent cytotoxicity towards human Molt 4/C8 and CEM T-lymphocytes as well as murine P388 and L1210 leukemic cells and spasmolytic activities (Dimmock *et al.*, 2003; Abignente & Biniecka-Picazio, 1977). We report here the synthesis and structure of a novel spiropyrrolidine, the title compound (I), Fig. 1.

The slightly distorted half-chair conformation of the piperidin-4-one ring is confirmed by the puckering analysis [q_2 = 0.312 (1) Å, φ_2 = 230 (2) °, q_3 = −0.449 (1) Å; Cremer & Pople, 1975] (Fig. 1). Also, the 5-membered ring is in an envelope conformation with the puckering values of q_2 = 0.427 (2) Å, φ_2 = 329 (2)°. The benzene ring fused to the 5-membered ring is oriented with an angle of 80.4 (1)°. The two phenyl rings (C23—C28 and C29—C34) subtend angles of 27.9 (2) and 85.8 (1)° with the methylidenephenyl ring (C17—C22) attached to the 6-membered ring.

In the crystal structure, molecules dimerize through the N—H···O hydrogen bonds around an inversion centre (Fig. 2). The crystal is further stabilized by C—H···π interactions between H31 and the C17—C22 benzene ring (centroid *Cg1*) (Table 1).

Experimental

1-Methyl-3,5-bis[(*E*)-phenylmethylidene]tetrahydro-4(1*H*)-pyridinone (1 mmol), isatin (1 mmol) and benzyl amine (1 mmol) were mixed well in a tube and kept over a water bath at 85–90^o C for 10–15 s until the mixture becomes a viscous paste to ensure thorough mixing and left aside for 10 min at ambient temperature. After completion of the reaction (TLC), the viscous paste was recrystallized from ethanol to obtain pure crystals of the title compound.

Refinement

All the H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.97 Å and N—H = 0.86 Å and U_{iso}(H) = 1.2–1.5 U_{eq} (parent atom).

There are large accessible voids of 264 Å^3 in the structure, which host disordered ethanol solvent molecules. This affected the diffraction pattern, mostly at low scattering angles, and was corrected with the SQUEEZE program (*PLATON*; Spek, 2003).

Figures

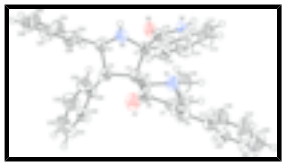


Fig. 1. The molecular structure of the title compound (I) with the numbering scheme and 50% probability displacement ellipsoids.

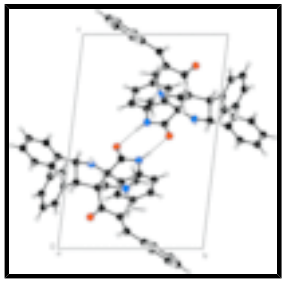


Fig. 2. Crystal packing for (I) viewed down the *a*-axis. H bonds are drawn as dashed lines.

5''-Benzylidene-1''-methyl-4',5'-diphenyl-1*H*-indole-3-spiro-2'-pyrrolidine-\ 3'-spiro-3''-piperidine-2,4''-dione

Crystal data

$C_{35}H_{31}N_3O_2$	$Z = 2$
$M_r = 525.63$	$F_{000} = 556$
Triclinic, $P\bar{1}$	$D_x = 1.118$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 10.194$ (4) Å	Cell parameters from 25 reflections
$b = 10.681$ (5) Å	$\theta = 9.1$–12.7°
$c = 15.352$ (8) Å	$\mu = 0.07$ mm^{-1}
$\alpha = 80.76$ (4)°	$T = 293$ (2) K
$\beta = 80.31$ (2)°	Block, colourless
$\gamma = 72.71$ (5)°	0.25 × 0.21 × 0.18 mm
$V = 1562.4$ (13) Å^3	

Data collection

Nonius MACH3 sealed-tube diffractometer	$R_{int} = 0.020$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.0^\circ$
Monochromator: graphite	$\theta_{min} = 2.0^\circ$
$T = 293(2)$ K	$h = -1 \rightarrow 12$
$\omega/2\theta$ scans	$k = -12 \rightarrow 12$
Absorption correction: ψ scan (North *et al.*, 1968)	$l = -18 \rightarrow 18$
$T_{min} = 0.968$, $T_{max} = 0.995$	3 standard reflections
6511 measured reflections	every 60 min
5494 independent reflections	intensity decay: none
2506 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.051$	H-atom parameters constrained
$wR(F^2) = 0.143$	$w = 1/[\sigma^2(F_o^2) + (0.0742P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.89$	$(\Delta/\sigma)_{max} < 0.001$
5494 reflections	$\Delta\rho_{max} = 0.26$ e Å^{-3}
363 parameters	$\Delta\rho_{min} = -0.22$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2$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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7418 (3)	0.2828 (2)	0.35142 (15)	0.0446 (6)
C2	0.7663 (3)	0.0554 (2)	0.40313 (16)	0.0474 (7)
H2	0.6821	0.0705	0.4462	0.057*
C3	0.7292 (3)	0.0721 (2)	0.30834 (16)	0.0446 (6)
H3	0.8171	0.0436	0.2699	0.053*
C4	0.6756 (3)	0.2245 (2)	0.28475 (16)	0.0446 (6)
C5	0.5182 (3)	0.2759 (2)	0.29432 (18)	0.0534 (7)
H5A	0.4825	0.2424	0.2509	0.064*
H5B	0.4789	0.2462	0.3533	0.064*
C6	0.5102 (3)	0.4660 (3)	0.18724 (19)	0.0614 (8)
H6A	0.4952	0.5609	0.1810	0.074*
H6B	0.4461	0.4484	0.1539	0.074*
C7	0.6546 (3)	0.4031 (2)	0.14835 (17)	0.0532 (7)
C8	0.7281 (3)	0.2684 (2)	0.18842 (17)	0.0501 (7)
C9	0.7272 (4)	0.4596 (3)	0.08092 (19)	0.0683 (9)
H9	0.8160	0.4074	0.0640	0.082*
C10	0.6297 (3)	0.3648 (2)	0.41871 (18)	0.0495 (7)
C11	0.7583 (3)	0.5007 (2)	0.34650 (17)	0.0501 (7)

C12	0.8105 (4)	0.6082 (3)	0.3224 (2)	0.0679 (8)
H12	0.7677	0.6873	0.3463	0.081*
C13	0.9275 (4)	0.5945 (3)	0.2620 (2)	0.0828 (10)
H13	0.9637	0.6663	0.2439	0.099*
C14	0.9937 (4)	0.4764 (4)	0.2270 (2)	0.0793 (9)
H14	1.0732	0.4696	0.1861	0.095*
C15	0.9413 (3)	0.3688 (3)	0.25317 (19)	0.0648 (8)
H15	0.9869	0.2886	0.2314	0.078*
C16	0.8218 (3)	0.3812 (2)	0.31119 (16)	0.0467 (7)
C17	0.6895 (4)	0.5905 (3)	0.02937 (19)	0.0708 (9)
C18	0.7963 (5)	0.6480 (4)	−0.0052 (2)	0.1059 (13)
H18	0.8864	0.6036	0.0057	0.127*
C19	0.7696 (6)	0.7709 (4)	−0.0559 (3)	0.1232 (17)
H19	0.8415	0.8088	−0.0781	0.148*
C20	0.6374 (7)	0.8359 (4)	−0.0729 (3)	0.1199 (17)
H20	0.6196	0.9175	−0.1075	0.144*
C21	0.5315 (5)	0.7812 (4)	−0.0390 (3)	0.1031 (13)
H21	0.4417	0.8266	−0.0501	0.124*
C22	0.5567 (4)	0.6586 (3)	0.0116 (2)	0.0843 (11)
H22	0.4838	0.6220	0.0336	0.101*
C23	0.8664 (3)	−0.0761 (2)	0.42894 (17)	0.0485 (7)
C24	0.8285 (3)	−0.1674 (3)	0.4941 (2)	0.0679 (8)
H24	0.7399	−0.1471	0.5254	0.081*
C25	0.9218 (4)	−0.2906 (3)	0.5138 (2)	0.0793 (10)
H25	0.8946	−0.3513	0.5583	0.095*
C26	1.0515 (4)	−0.3227 (3)	0.4690 (2)	0.0732 (9)
H26	1.1123	−0.4057	0.4815	0.088*
C27	1.0917 (3)	−0.2317 (3)	0.4053 (2)	0.0748 (9)
H27	1.1811	−0.2519	0.3751	0.090*
C28	0.9998 (3)	−0.1098 (3)	0.3856 (2)	0.0642 (8)
H28	1.0286	−0.0489	0.3420	0.077*
C29	0.6387 (3)	−0.0084 (2)	0.29153 (17)	0.0471 (7)
C30	0.6614 (3)	−0.0580 (3)	0.21036 (19)	0.0621 (8)
H30	0.7315	−0.0409	0.1671	0.075*
C31	0.5803 (4)	−0.1329 (3)	0.1932 (2)	0.0823 (10)
H31	0.5965	−0.1660	0.1388	0.099*
C32	0.4765 (4)	−0.1580 (3)	0.2564 (3)	0.0885 (11)
H32	0.4216	−0.2075	0.2447	0.106*
C33	0.4539 (4)	−0.1109 (3)	0.3358 (2)	0.0861 (10)
H33	0.3834	−0.1283	0.3787	0.103*
C34	0.5342 (3)	−0.0373 (3)	0.3538 (2)	0.0681 (8)
H34	0.5176	−0.0064	0.4090	0.082*
C35	0.3367 (3)	0.4792 (3)	0.3149 (2)	0.0911 (11)
H35A	0.3223	0.4527	0.3778	0.137*
H35B	0.2763	0.4508	0.2857	0.137*
H35C	0.3169	0.5736	0.3042	0.137*
N1	0.4800 (2)	0.41942 (19)	0.28030 (15)	0.0553 (6)
N2	0.6481 (2)	0.48676 (19)	0.41089 (14)	0.0534 (6)
H2A	0.5978	0.5482	0.4420	0.064*

N3	0.8292 (2)	0.16302 (18)	0.39558 (14)	0.0521 (6)
H3A	0.9061	0.1571	0.4142	0.063*
O1	0.8263 (2)	0.19800 (18)	0.14694 (12)	0.0719 (6)
O2	0.5457 (2)	0.32358 (17)	0.47439 (12)	0.0623 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0525 (16)	0.0346 (14)	0.0450 (15)	−0.0081 (12)	−0.0037 (13)	−0.0104 (11)
C2	0.0566 (17)	0.0362 (14)	0.0483 (15)	−0.0070 (13)	−0.0072 (13)	−0.0125 (11)
C3	0.0507 (16)	0.0352 (13)	0.0445 (14)	−0.0039 (12)	−0.0028 (12)	−0.0139 (11)
C4	0.0500 (17)	0.0328 (13)	0.0490 (15)	−0.0051 (12)	−0.0046 (13)	−0.0132 (11)
C5	0.0594 (19)	0.0430 (15)	0.0569 (16)	−0.0081 (14)	−0.0092 (14)	−0.0131 (12)
C6	0.074 (2)	0.0427 (16)	0.067 (2)	−0.0064 (15)	−0.0240 (17)	−0.0074 (14)
C7	0.072 (2)	0.0441 (16)	0.0438 (16)	−0.0110 (15)	−0.0142 (15)	−0.0074 (13)
C8	0.0634 (19)	0.0365 (14)	0.0490 (16)	−0.0078 (14)	−0.0070 (14)	−0.0124 (12)
C9	0.097 (2)	0.0515 (18)	0.0545 (18)	−0.0143 (17)	−0.0146 (18)	−0.0060 (14)
C10	0.0620 (18)	0.0371 (15)	0.0455 (15)	−0.0046 (13)	−0.0096 (14)	−0.0084 (12)
C11	0.0649 (19)	0.0403 (15)	0.0470 (16)	−0.0123 (14)	−0.0164 (15)	−0.0055 (12)
C12	0.087 (2)	0.0459 (17)	0.077 (2)	−0.0211 (17)	−0.0241 (19)	−0.0044 (15)
C13	0.094 (3)	0.075 (2)	0.094 (3)	−0.050 (2)	−0.018 (2)	0.003 (2)
C14	0.076 (2)	0.088 (3)	0.083 (2)	−0.040 (2)	−0.0053 (19)	−0.010 (2)
C15	0.065 (2)	0.066 (2)	0.0660 (19)	−0.0185 (17)	−0.0067 (17)	−0.0156 (15)
C16	0.0540 (17)	0.0400 (15)	0.0464 (15)	−0.0114 (13)	−0.0081 (14)	−0.0076 (12)
C17	0.111 (3)	0.0475 (18)	0.0534 (18)	−0.020 (2)	−0.013 (2)	−0.0044 (14)
C18	0.130 (3)	0.067 (2)	0.098 (3)	−0.021 (2)	0.018 (3)	0.009 (2)
C19	0.168 (5)	0.074 (3)	0.109 (3)	−0.037 (3)	0.009 (3)	0.018 (2)
C20	0.194 (5)	0.064 (3)	0.090 (3)	−0.026 (3)	−0.026 (3)	0.016 (2)
C21	0.161 (4)	0.071 (3)	0.081 (3)	−0.025 (3)	−0.056 (3)	0.007 (2)
C22	0.128 (3)	0.063 (2)	0.069 (2)	−0.026 (2)	−0.044 (2)	0.0035 (17)
C23	0.0549 (18)	0.0354 (14)	0.0537 (16)	−0.0028 (13)	−0.0144 (14)	−0.0119 (13)
C24	0.076 (2)	0.0466 (17)	0.077 (2)	−0.0122 (16)	−0.0164 (17)	0.0030 (16)
C25	0.101 (3)	0.052 (2)	0.087 (2)	−0.022 (2)	−0.034 (2)	0.0125 (17)
C26	0.077 (2)	0.0435 (18)	0.100 (3)	0.0014 (17)	−0.039 (2)	−0.0192 (18)
C27	0.074 (2)	0.054 (2)	0.090 (2)	0.0014 (18)	−0.0168 (19)	−0.0219 (18)
C28	0.068 (2)	0.0442 (17)	0.0727 (19)	−0.0004 (15)	−0.0106 (17)	−0.0113 (14)
C29	0.0611 (18)	0.0312 (13)	0.0478 (15)	−0.0086 (13)	−0.0090 (14)	−0.0070 (11)
C30	0.085 (2)	0.0437 (16)	0.0569 (17)	−0.0112 (15)	−0.0152 (16)	−0.0089 (13)
C31	0.120 (3)	0.062 (2)	0.075 (2)	−0.023 (2)	−0.033 (2)	−0.0225 (17)
C32	0.115 (3)	0.063 (2)	0.109 (3)	−0.043 (2)	−0.044 (3)	−0.004 (2)
C33	0.108 (3)	0.085 (2)	0.085 (3)	−0.058 (2)	−0.016 (2)	−0.004 (2)
C34	0.085 (2)	0.0649 (19)	0.0622 (19)	−0.0303 (18)	−0.0074 (18)	−0.0134 (15)
C35	0.062 (2)	0.075 (2)	0.115 (3)	0.0094 (18)	0.001 (2)	−0.019 (2)
N1	0.0521 (15)	0.0398 (13)	0.0667 (16)	0.0008 (11)	−0.0059 (12)	−0.0130 (11)
N2	0.0724 (16)	0.0319 (12)	0.0531 (13)	−0.0044 (11)	−0.0087 (12)	−0.0156 (10)
N3	0.0548 (14)	0.0348 (12)	0.0692 (15)	−0.0068 (10)	−0.0222 (12)	−0.0086 (10)
O1	0.0881 (15)	0.0495 (11)	0.0595 (12)	−0.0009 (11)	0.0131 (12)	−0.0109 (10)
O2	0.0826 (15)	0.0434 (11)	0.0538 (12)	−0.0135 (10)	0.0120 (11)	−0.0147 (9)

Geometric parameters (Å, °)

C1—N3	1.462 (3)	C17—C18	1.393 (5)
C1—C16	1.511 (3)	C18—C19	1.390 (5)
C1—C10	1.561 (3)	C18—H18	0.9300
C1—C4	1.606 (3)	C19—C20	1.367 (6)
C2—N3	1.455 (3)	C19—H19	0.9300
C2—C23	1.509 (3)	C20—C21	1.365 (6)
C2—C3	1.534 (3)	C20—H20	0.9300
C2—H2	0.9800	C21—C22	1.387 (4)
C3—C29	1.509 (4)	C21—H21	0.9300
C3—C4	1.558 (3)	C22—H22	0.9300
C3—H3	0.9800	C23—C24	1.371 (4)
C4—C5	1.523 (3)	C23—C28	1.379 (4)
C4—C8	1.534 (3)	C24—C25	1.397 (4)
C5—N1	1.454 (3)	C24—H24	0.9300
C5—H5A	0.9700	C25—C26	1.356 (4)
C5—H5B	0.9700	C25—H25	0.9300
C6—N1	1.448 (3)	C26—C27	1.366 (4)
C6—C7	1.490 (4)	C26—H26	0.9300
C6—H6A	0.9700	C27—C28	1.382 (4)
C6—H6B	0.9700	C27—H27	0.9300
C7—C9	1.338 (4)	C28—H28	0.9300
C7—C8	1.496 (4)	C29—C34	1.379 (4)
C8—O1	1.210 (3)	C29—C30	1.389 (3)
C9—C17	1.468 (4)	C30—C31	1.387 (4)
C9—H9	0.9300	C30—H30	0.9300
C10—O2	1.230 (3)	C31—C32	1.369 (5)
C10—N2	1.354 (3)	C31—H31	0.9300
C11—C12	1.378 (4)	C32—C33	1.351 (5)
C11—N2	1.393 (3)	C32—H32	0.9300
C11—C16	1.397 (3)	C33—C34	1.375 (4)
C12—C13	1.368 (4)	C33—H33	0.9300
C12—H12	0.9300	C34—H34	0.9300
C13—C14	1.387 (4)	C35—N1	1.455 (3)
C13—H13	0.9300	C35—H35A	0.9600
C14—C15	1.383 (4)	C35—H35B	0.9600
C14—H14	0.9300	C35—H35C	0.9600
C15—C16	1.368 (4)	N2—H2A	0.8600
C15—H15	0.9300	N3—H3A	0.8600
C17—C22	1.383 (4)		
N3—C1—C16	112.0 (2)	C18—C17—C9	117.2 (3)
N3—C1—C10	112.3 (2)	C19—C18—C17	120.8 (4)
C16—C1—C10	100.90 (19)	C19—C18—H18	119.6
N3—C1—C4	102.18 (17)	C17—C18—H18	119.6
C16—C1—C4	117.40 (19)	C20—C19—C18	119.8 (5)
C10—C1—C4	112.6 (2)	C20—C19—H19	120.1
N3—C2—C23	111.4 (2)	C18—C19—H19	120.1

N3—C2—C3	99.96 (19)	C21—C20—C19	120.1 (4)
C23—C2—C3	113.86 (19)	C21—C20—H20	120.0
N3—C2—H2	110.4	C19—C20—H20	120.0
C23—C2—H2	110.4	C20—C21—C22	120.7 (4)
C3—C2—H2	110.4	C20—C21—H21	119.7
C29—C3—C2	117.1 (2)	C22—C21—H21	119.7
C29—C3—C4	116.4 (2)	C17—C22—C21	120.3 (4)
C2—C3—C4	103.98 (18)	C17—C22—H22	119.8
C29—C3—H3	106.2	C21—C22—H22	119.8
C2—C3—H3	106.2	C24—C23—C28	117.5 (3)
C4—C3—H3	106.2	C24—C23—C2	121.9 (3)
C5—C4—C8	106.4 (2)	C28—C23—C2	120.5 (2)
C5—C4—C3	113.4 (2)	C23—C24—C25	120.6 (3)
C8—C4—C3	111.58 (19)	C23—C24—H24	119.7
C5—C4—C1	112.41 (18)	C25—C24—H24	119.7
C8—C4—C1	109.30 (19)	C26—C25—C24	120.9 (3)
C3—C4—C1	103.79 (19)	C26—C25—H25	119.6
N1—C5—C4	108.6 (2)	C24—C25—H25	119.6
N1—C5—H5A	110.0	C25—C26—C27	119.2 (3)
C4—C5—H5A	110.0	C25—C26—H26	120.4
N1—C5—H5B	110.0	C27—C26—H26	120.4
C4—C5—H5B	110.0	C26—C27—C28	120.1 (3)
H5A—C5—H5B	108.4	C26—C27—H27	119.9
N1—C6—C7	113.2 (2)	C28—C27—H27	119.9
N1—C6—H6A	108.9	C23—C28—C27	121.7 (3)
C7—C6—H6A	108.9	C23—C28—H28	119.2
N1—C6—H6B	108.9	C27—C28—H28	119.2
C7—C6—H6B	108.9	C34—C29—C30	117.5 (3)
H6A—C6—H6B	107.8	C34—C29—C3	123.1 (2)
C9—C7—C6	124.9 (3)	C30—C29—C3	119.4 (2)
C9—C7—C8	116.3 (3)	C31—C30—C29	120.6 (3)
C6—C7—C8	118.7 (2)	C31—C30—H30	119.7
O1—C8—C7	121.3 (2)	C29—C30—H30	119.7
O1—C8—C4	121.5 (2)	C32—C31—C30	120.0 (3)
C7—C8—C4	117.3 (2)	C32—C31—H31	120.0
C7—C9—C17	130.5 (3)	C30—C31—H31	120.0
C7—C9—H9	114.7	C33—C32—C31	119.9 (3)
C17—C9—H9	114.7	C33—C32—H32	120.0
O2—C10—N2	125.2 (2)	C31—C32—H32	120.0
O2—C10—C1	126.3 (2)	C32—C33—C34	120.5 (3)
N2—C10—C1	108.3 (2)	C32—C33—H33	119.7
C12—C11—N2	128.4 (3)	C34—C33—H33	119.7
C12—C11—C16	121.8 (3)	C33—C34—C29	121.4 (3)
N2—C11—C16	109.7 (2)	C33—C34—H34	119.3
C13—C12—C11	117.6 (3)	C29—C34—H34	119.3
C13—C12—H12	121.2	N1—C35—H35A	109.5
C11—C12—H12	121.2	N1—C35—H35B	109.5
C12—C13—C14	121.7 (3)	H35A—C35—H35B	109.5
C12—C13—H13	119.1	N1—C35—H35C	109.5

C14—C13—H13	119.1	H35A—C35—H35C	109.5
C15—C14—C13	119.9 (3)	H35B—C35—H35C	109.5
C15—C14—H14	120.1	C6—N1—C5	110.8 (2)
C13—C14—H14	120.1	C6—N1—C35	111.5 (2)
C16—C15—C14	119.5 (3)	C5—N1—C35	112.8 (2)
C16—C15—H15	120.2	C10—N2—C11	111.9 (2)
C14—C15—H15	120.2	C10—N2—H2A	124.1
C15—C16—C11	119.4 (2)	C11—N2—H2A	124.1
C15—C16—C1	131.4 (2)	C2—N3—C1	109.5 (2)
C11—C16—C1	109.1 (2)	C2—N3—H3A	125.2
C22—C17—C18	118.3 (3)	C1—N3—H3A	125.2
C22—C17—C9	124.6 (3)		
N3—C2—C3—C29	−170.15 (19)	N3—C1—C16—C15	53.8 (3)
C23—C2—C3—C29	71.0 (3)	C10—C1—C16—C15	173.4 (3)
N3—C2—C3—C4	−40.2 (2)	C4—C1—C16—C15	−63.9 (4)
C23—C2—C3—C4	−159.1 (2)	N3—C1—C16—C11	−122.9 (2)
C29—C3—C4—C5	29.9 (3)	C10—C1—C16—C11	−3.3 (3)
C2—C3—C4—C5	−100.5 (2)	C4—C1—C16—C11	119.4 (2)
C29—C3—C4—C8	−90.3 (3)	C7—C9—C17—C22	−31.2 (5)
C2—C3—C4—C8	139.3 (2)	C7—C9—C17—C18	150.1 (3)
C29—C3—C4—C1	152.1 (2)	C22—C17—C18—C19	0.3 (5)
C2—C3—C4—C1	21.8 (2)	C9—C17—C18—C19	179.1 (3)
N3—C1—C4—C5	127.7 (2)	C17—C18—C19—C20	−0.6 (6)
C16—C1—C4—C5	−109.5 (2)	C18—C19—C20—C21	1.0 (7)
C10—C1—C4—C5	7.0 (3)	C19—C20—C21—C22	−1.0 (6)
N3—C1—C4—C8	−114.4 (2)	C18—C17—C22—C21	−0.4 (5)
C16—C1—C4—C8	8.5 (3)	C9—C17—C22—C21	−179.0 (3)
C10—C1—C4—C8	125.0 (2)	C20—C21—C22—C17	0.7 (5)
N3—C1—C4—C3	4.8 (2)	N3—C2—C23—C24	134.2 (3)
C16—C1—C4—C3	127.6 (2)	C3—C2—C23—C24	−113.7 (3)
C10—C1—C4—C3	−115.9 (2)	N3—C2—C23—C28	−47.4 (3)
C8—C4—C5—N1	−63.1 (2)	C3—C2—C23—C28	64.8 (3)
C3—C4—C5—N1	173.8 (2)	C28—C23—C24—C25	−1.3 (4)
C1—C4—C5—N1	56.5 (3)	C2—C23—C24—C25	177.2 (3)
N1—C6—C7—C9	−151.3 (3)	C23—C24—C25—C26	−0.2 (5)
N1—C6—C7—C8	26.5 (3)	C24—C25—C26—C27	1.6 (5)
C9—C7—C8—O1	−22.9 (4)	C25—C26—C27—C28	−1.5 (5)
C6—C7—C8—O1	159.1 (3)	C24—C23—C28—C27	1.3 (4)
C9—C7—C8—C4	155.9 (2)	C2—C23—C28—C27	−177.2 (3)
C6—C7—C8—C4	−22.1 (3)	C26—C27—C28—C23	0.0 (5)
C5—C4—C8—O1	−142.5 (3)	C2—C3—C29—C34	35.1 (3)
C3—C4—C8—O1	−18.3 (3)	C4—C3—C29—C34	−88.8 (3)
C1—C4—C8—O1	95.9 (3)	C2—C3—C29—C30	−144.0 (2)
C5—C4—C8—C7	38.8 (3)	C4—C3—C29—C30	92.1 (3)
C3—C4—C8—C7	162.9 (2)	C34—C29—C30—C31	0.5 (4)
C1—C4—C8—C7	−82.9 (3)	C3—C29—C30—C31	179.7 (3)
C6—C7—C9—C17	0.3 (5)	C29—C30—C31—C32	0.3 (5)
C8—C7—C9—C17	−177.6 (3)	C30—C31—C32—C33	−0.6 (5)
N3—C1—C10—O2	−53.5 (3)	C31—C32—C33—C34	0.2 (5)

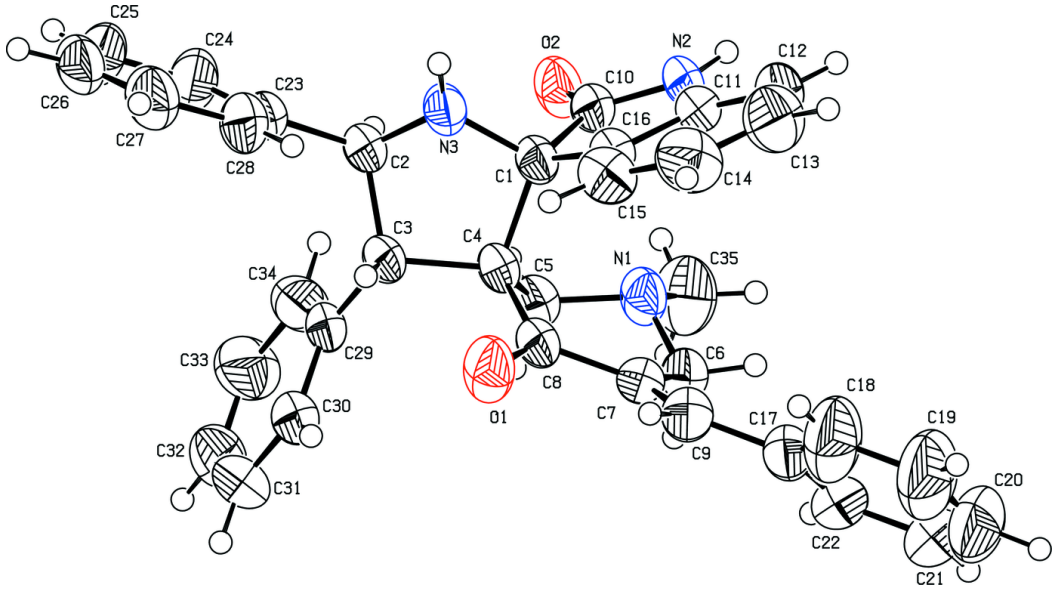
C16—C1—C10—O2	−172.9 (2)	C32—C33—C34—C29	0.6 (5)
C4—C1—C10—O2	61.1 (3)	C30—C29—C34—C33	−0.9 (4)
N3—C1—C10—N2	121.2 (2)	C3—C29—C34—C33	179.9 (3)
C16—C1—C10—N2	1.8 (3)	C7—C6—N1—C5	−51.3 (3)
C4—C1—C10—N2	−124.2 (2)	C7—C6—N1—C35	−177.8 (2)
N2—C11—C12—C13	174.7 (3)	C4—C5—N1—C6	72.6 (3)
C16—C11—C12—C13	0.1 (4)	C4—C5—N1—C35	−161.7 (2)
C11—C12—C13—C14	−1.2 (5)	O2—C10—N2—C11	175.0 (2)
C12—C13—C14—C15	0.1 (5)	C1—C10—N2—C11	0.3 (3)
C13—C14—C15—C16	2.1 (5)	C12—C11—N2—C10	−177.6 (3)
C14—C15—C16—C11	−3.1 (4)	C16—C11—N2—C10	−2.5 (3)
C14—C15—C16—C1	−179.5 (3)	C23—C2—N3—C1	167.17 (19)
C12—C11—C16—C15	2.0 (4)	C3—C2—N3—C1	46.5 (2)
N2—C11—C16—C15	−173.4 (2)	C16—C1—N3—C2	−158.78 (19)
C12—C11—C16—C1	179.2 (2)	C10—C1—N3—C2	88.6 (2)
N2—C11—C16—C1	3.7 (3)	C4—C1—N3—C2	−32.3 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O2^{i}	0.86	2.10	2.950 (3)	170
C31—H31···Cg1^{ii}	0.93	2.85	3.778 (5)	175

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

Fig. 1


C25
C24
C23
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N2
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C1
C15
C3
C4
C5
N1
C35
C34
C29
C33
C8
C6
C18
O1
C7
C17
C19
C30
C9
C32
C31
C20
C22
C21

Fig. 2