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5″-(4-Chlorobenzylidene)-4′-(4-chlorophenyl)-1″-methyl-5′-phenylacenaphthene-1-spiro-2′-pyrrolidine-3′-spiro-3″-piperidine-2,4″-dione

S. Athimoolam,[a]* R. Ranjith Kumar,[b] K. Vijaya,[c] R. Sayee Kannan[d] and S. Perumal[b]

[a]Department of Physics, Kalasalingam University, Anand Nagar, Krishnan Koil 626 190, India, [b]Department of Organic Chemistry, Madurai Kamaraj University, Madurai 625 021, India, [c]Department of Chemistry, P. S. N. A. College of Engineering and Technology, Dindigul 624 622, India, and [d]Department of Chemistry, Thiagarajar College, Madurai 625 009, India
Correspondence e-mail: athi81s@yahoo.co.in

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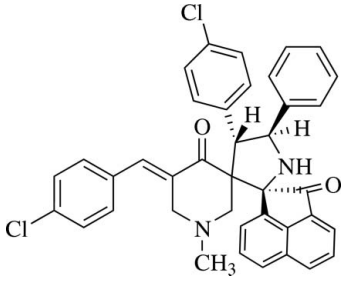
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.048; wR factor = 0.142; data-to-parameter ratio = 13.6.

In the title compound, $C_{39}H_{30}Cl_2N_2O_2$, the central six-membered piperidinone ring adopts a distorted half-chair conformation and the five-membered pyrrolidine ring is in a twist conformation. The crystal structure is stabilized by C—H···O and C—H···π interactions.

Related literature

For the pharmacological importance of similar compounds, see: Babu & Raghunathan (2007); Boruah *et al.* (2007); Chande *et al.* (2005); Horri *et al.* (1986); Karthikeyan *et al.* (2007); Watson *et al.* (2001). For ring puckering analysis, see: Cremer & Pople (1975) and for hydrogen-bonding interactions, see: Desiraju & Steiner, (1999).



Experimental

Crystal data

$C_{39}H_{30}Cl_2N_2O_2$
M_r = 629.55
Triclinic, $P\overline{1}$
a = 9.5567 (6) Å
b = 12.0442 (11) Å
c = 14.8593 (15) Å
α = 76.025 (11)°
β = 75.445 (9)°
γ = 76.220 (8)°
V = 1577.5 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 293 (2) K
0.24 × 0.15 × 0.14 mm

Data collection

Enraf–Nonius MACH3 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.947, T_{max} = 0.973
6631 measured reflections
5538 independent reflections
3434 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.142
S = 1.01
5538 reflections
406 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O1	0.98	2.41	2.789 (3)	102
C21—H21···O1	0.93	2.46	2.810 (4)	102
C27—H27···O1^{i}	0.93	2.47	3.358 (4)	160
C16—H16A···O2^{ii}	0.97	2.56	3.491 (3)	161
C32—H32···Cg^{iii}	0.93	2.83	3.498 (5)	129

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

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: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL/PC*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2401).

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supplementary materials

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5''-(4-Chlorobenzylidene)-4'-(4-chlorophenyl)-1''-methyl-5'-phenylacenaphthene-1-spiro-2'-pyrrolidine-3'-spiro-3''-piperidine-2,4''-dione

S. Athimoolam, R. R. Kumar, K. Vijaya, R. S. Kannan and S. Perumal

Comment

1,3-Dipolar cycloadditions form a subject of intensive research in organic synthesis in view of their great synthetic potential (Karthikeyan *et al.*, 2007). In particular, the cycloaddition of nonstabilized azomethine ylides with olefins represents one of the most convergent approaches for the construction of pyrrolidines (Boruah *et al.*, 2007), which are prevalent in a variety of biologically active compounds (Watson *et al.*, 2001) and find utility in the treatment of diseases such as diabetes (Horri *et al.*, 1986). Acenaphthenequinone is a versatile precursor for azomethine ylide cycloaddition as it reacts with various α-amino acids generating reactive 1,3-dipoles (Babu & Raghunathan, 2007). Synthesis of spiro compounds have drawn considerable attention of the chemists, in view of their very good antimycobacterial activity. The importance of spiro-compounds and nitrogen heterocycles led us to synthesize novel spiro heterocycles *via* 1,3-dipoar cycloaddition of azomethine ylides (Chande *et al.*, 2005).

The slightly twisted conformation of the 5-membered pyrrolidine ring is confirmed through the puckering analysis [θ_2 = 0.420 (3) Å and π_2 = 47.5 (4)°; Cremer & Pople, 1975] and the 6-membered piperidinone ring adopts a distorted half-chair conformation [θ_2 = 0.273 (3) Å, π_2 = 62.7 (6)° and θ_3 = 0.467 (3) Å] (Fig. 1). The dihedral angle between the chlorophenyl rings is observed to be 73.6 (1)° and these rings make angles of 71.5 (1) and 23.1 (2) ° with the phenyl ring (C9/C14). Further, the acenaphthene group (C28/C39) is oriented at an angle of 35.1 (1)° to the (C9/C14) phenyl ring.

The crystal structue of (I) features two intramolecular and two intermolecular C—H···O hydrogen bonds [Fig 2; Table 1]. It is further stabilized by an intermolecular C32—H32···π interaction, with the C1—C6 benzene ring (centroid *Cg*) (Desiraju & Steiner, 1999).

Experimental

A mixture of 1-methyl-3,5-bis[(*E*)-4-chlorophenylmethylidene]tetrahydro-4(1*H*)-pyridinone 1 mmol), acenaphthenequinone (1 mmol) and phenylglycine (1 mmol) was dissolved in methanol (10 ml) and refluxed for 1 h. After completion of the reaction evident from TLC, the mixture was poured into water (50 ml), the precipitated solid filtered and washed with water (100 ml) to obtain pure 4-(4-chlorophenyl)-5-phenylpyrrolo-(spiro[2.2"]-acenaphthene-1"-one)- spiro[3.3']-5'-(4-chlorophenylmethylid-ene)-1'-methyltetrahydro-4'- (1*H*)-pyridinone as pale yellow solid. The product was recrystallized from methanol/ethyl acetate solution (1:1).

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).

Figures

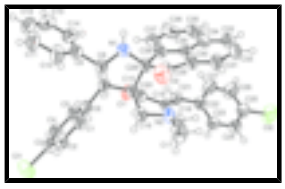

Fig. 1. The molecular structure of the title compound (I) with the numbering scheme for the atoms and 50% probability displacement ellipsoids.

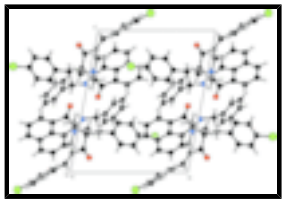

Fig. 2. Packing diagram of the molecules, viewed down the *b*-axis.

4-(4-Chlorophenyl)-5-phenylpyrrolo-(spiro[2.2'']-acenaphthene- 1''-one)-spiro[3.3']-5'-(4-chlorophenylmethyl-idene)-1'- methyltetrahydro-4'-(1*H*)-pyridinone

Crystal data

$C_{39}H_{30}Cl_2N_2O_2$	$Z = 2$
$M_r = 629.55$	$F_{000} = 656$
Triclinic, $P\overline{1}$	$D_x = 1.325$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 190 K
$a = 9.5567$ (6) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 12.0442$ (11) Å	Cell parameters from 25 reflections
$c = 14.8593$ (15) Å	$\theta = 10.3$–14.3º
$\alpha = 76.025$ (11)º	$\mu = 0.24$ mm^{-1}
$\beta = 75.445$ (9)º	$T = 293$ (2) K
$\gamma = 76.220$ (8)º	Block, pale yellow
$V = 1577.5$ (3) Å^3	0.24 × 0.15 × 0.14 mm

Data collection

Nonius MACH3 diffractometer	$R_{int} = 0.026$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.0$º
Monochromator: graphite	$\theta_{min} = 2.1$º
$T = 293$(2) K	$h = -1 \rightarrow 11$
ω–2θ scans	$k = -14 \rightarrow 14$
Absorption correction: ψ scan (North et al., 1968)	$l = -17 \rightarrow 17$
$T_{min} = 0.947$, $T_{max} = 0.973$	3 standard reflections
6631 measured reflections	every 60 min
5538 independent reflections	intensity decay: none
3434 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
$R[F^2 > 2\sigma(F^2)] = 0.048$	H-atom parameters constrained
$wR(F^2) = 0.142$	$w = 1/[\sigma^2^(F_o^2^) + (0.0646P)^2^ + 0.6033P]$ where $P = (F_o^2^ + 2F_c^2^)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = <0.001$
5538 reflections	$\Delta\rho_{max} = 0.36$ e Å^{-3}
406 parameters	$\Delta\rho_{min} = -0.29$ 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^ > 2sigma(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}
Cl1	0.39281 (11)	0.66064 (7)	0.25272 (8)	0.0971 (3)
Cl2	0.66042 (13)	−0.41648 (10)	−0.12138 (9)	0.1138 (4)
O1	0.8216 (2)	0.13701 (16)	0.10490 (13)	0.0599 (5)
O2	0.6391 (3)	−0.11023 (17)	0.46785 (14)	0.0694 (6)
N2	0.5209 (2)	−0.03202 (17)	0.29297 (15)	0.0486 (5)
N1	0.8833 (3)	0.01394 (18)	0.37587 (17)	0.0608 (6)
H1N	0.9614	−0.0209	0.3972	0.073*
C15	0.7150 (3)	0.07564 (19)	0.26833 (17)	0.0439 (6)
C7	0.7663 (3)	0.1783 (2)	0.28857 (18)	0.0460 (6)
H7	0.8590	0.1876	0.2434	0.055*
C20	0.7480 (3)	0.0734 (2)	0.16348 (18)	0.0448 (6)
C6	0.6653 (3)	0.2963 (2)	0.27850 (18)	0.0453 (6)
C22	0.7278 (3)	−0.1436 (2)	0.01626 (18)	0.0491 (6)
C24	0.5669 (3)	−0.2443 (2)	−0.0226 (2)	0.0587 (7)
H24	0.4724	−0.2546	−0.0197	0.070*
C19	0.6821 (3)	−0.0111 (2)	0.13483 (17)	0.0440 (6)
C21	0.7584 (3)	−0.0567 (2)	0.05952 (19)	0.0502 (6)
H21	0.8448	−0.0290	0.0297	0.060*
C18	0.5404 (3)	−0.0415 (2)	0.19504 (18)	0.0524 (7)

H18A	0.4593	0.0098	0.1690	0.063*
H18B	0.5373	−0.1206	0.1928	0.063*
C39	0.8791 (3)	−0.2214 (2)	0.2780 (2)	0.0535 (7)
C4	0.6137 (4)	0.4930 (2)	0.1905 (2)	0.0618 (8)
H4	0.6370	0.5491	0.1368	0.074*
C38	0.9203 (3)	−0.1123 (2)	0.26017 (19)	0.0515 (7)
C16	0.5501 (3)	0.0771 (2)	0.30363 (19)	0.0480 (6)
H16A	0.5211	0.0845	0.3696	0.058*
H16B	0.4946	0.1427	0.2669	0.058*
C28	0.8110 (3)	−0.0362 (2)	0.32360 (18)	0.0489 (6)
C1	0.5444 (3)	0.3265 (2)	0.3482 (2)	0.0564 (7)
H1	0.5195	0.2708	0.4018	0.068*
C9	0.8973 (3)	0.2003 (2)	0.4117 (2)	0.0551 (7)
C23	0.5880 (3)	−0.1606 (2)	0.01949 (19)	0.0527 (7)
H23	0.5065	−0.1146	0.0507	0.063*
C8	0.8048 (3)	0.1311 (2)	0.38675 (19)	0.0517 (7)
H8	0.7143	0.1268	0.4353	0.062*
C3	0.4972 (3)	0.5199 (2)	0.2609 (2)	0.0597 (8)
C2	0.4596 (3)	0.4379 (2)	0.3397 (2)	0.0622 (8)
H2	0.3783	0.4567	0.3869	0.075*
C5	0.6977 (3)	0.3811 (2)	0.1992 (2)	0.0541 (7)
H5	0.7772	0.3629	0.1507	0.065*
C34	0.9571 (4)	−0.3103 (3)	0.2282 (2)	0.0674 (9)
C29	0.7140 (3)	−0.1243 (2)	0.3912 (2)	0.0537 (7)
C37	1.0424 (3)	−0.0926 (3)	0.1927 (2)	0.0654 (8)
H37	1.0748	−0.0226	0.1814	0.078*
C30	0.7584 (4)	−0.2334 (2)	0.3522 (2)	0.0566 (7)
C31	0.7090 (4)	−0.3377 (2)	0.3807 (2)	0.0706 (9)
H31	0.6281	−0.3476	0.4300	0.085*
C12	1.0636 (5)	0.3326 (3)	0.4550 (3)	0.0874 (12)
H12	1.1187	0.3780	0.4689	0.105*
C10	0.8448 (4)	0.2578 (3)	0.4865 (2)	0.0706 (9)
H10	0.7510	0.2530	0.5229	0.085*
C25	0.6862 (4)	−0.3114 (3)	−0.0683 (2)	0.0665 (8)
C27	0.8453 (3)	−0.2117 (3)	−0.0329 (2)	0.0739 (9)
H27	0.9401	−0.2006	−0.0378	0.089*
C17	0.3784 (4)	−0.0545 (3)	0.3471 (2)	0.0766 (10)
H17A	0.3686	−0.0478	0.4116	0.115*
H17B	0.3698	−0.1318	0.3454	0.115*
H17C	0.3023	0.0011	0.3203	0.115*
C14	1.0365 (4)	0.2101 (3)	0.3594 (3)	0.0808 (10)
H14	1.0753	0.1713	0.3089	0.097*
C13	1.1201 (4)	0.2763 (4)	0.3803 (3)	0.0940 (12)
H13	1.2135	0.2825	0.3440	0.113*
C11	0.9284 (5)	0.3229 (3)	0.5089 (3)	0.0831 (11)
H11	0.8918	0.3596	0.5607	0.100*
C32	0.7860 (5)	−0.4286 (3)	0.3325 (3)	0.0830 (11)
H32	0.7550	−0.4998	0.3512	0.100*
C26	0.8260 (4)	−0.2958 (3)	−0.0751 (3)	0.0860 (11)

H26	0.9067	−0.3412	−0.1075	0.103*
C33	0.9037 (5)	−0.4166 (3)	0.2597 (3)	0.0861 (11)
H33	0.9503	−0.4793	0.2300	0.103*
C36	1.1187 (4)	−0.1793 (3)	0.1403 (3)	0.0833 (10)
H36	1.1999	−0.1644	0.0926	0.100*
C35	1.0774 (4)	−0.2846 (3)	0.1571 (3)	0.0845 (11)
H35	1.1303	−0.3397	0.1207	0.101*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1059 (7)	0.0439 (4)	0.1351 (9)	0.0062 (4)	−0.0465 (6)	−0.0028 (5)
Cl2	0.1153 (8)	0.1074 (8)	0.1551 (10)	−0.0216 (6)	−0.0370 (7)	−0.0831 (8)
O1	0.0768 (14)	0.0599 (12)	0.0485 (11)	−0.0347 (11)	−0.0094 (10)	−0.0030 (9)
O2	0.1019 (17)	0.0576 (12)	0.0456 (12)	−0.0180 (11)	−0.0145 (12)	−0.0022 (9)
N2	0.0609 (14)	0.0387 (11)	0.0464 (13)	−0.0177 (10)	−0.0054 (11)	−0.0065 (9)
N1	0.0783 (17)	0.0404 (12)	0.0738 (16)	−0.0015 (11)	−0.0435 (14)	−0.0105 (11)
C15	0.0549 (16)	0.0339 (12)	0.0444 (14)	−0.0091 (11)	−0.0159 (12)	−0.0038 (10)
C7	0.0567 (16)	0.0375 (13)	0.0473 (15)	−0.0121 (12)	−0.0170 (13)	−0.0050 (11)
C20	0.0491 (15)	0.0385 (13)	0.0457 (15)	−0.0075 (12)	−0.0141 (12)	−0.0022 (11)
C6	0.0564 (16)	0.0357 (13)	0.0503 (15)	−0.0131 (12)	−0.0207 (13)	−0.0061 (11)
C22	0.0522 (16)	0.0493 (15)	0.0480 (15)	−0.0113 (13)	−0.0116 (13)	−0.0105 (12)
C24	0.0622 (18)	0.0606 (17)	0.0612 (18)	−0.0147 (15)	−0.0217 (15)	−0.0144 (14)
C19	0.0516 (15)	0.0407 (13)	0.0431 (14)	−0.0131 (11)	−0.0159 (12)	−0.0034 (11)
C21	0.0554 (17)	0.0494 (15)	0.0505 (16)	−0.0187 (13)	−0.0132 (13)	−0.0069 (12)
C18	0.0614 (17)	0.0486 (15)	0.0519 (16)	−0.0198 (13)	−0.0114 (13)	−0.0098 (12)
C39	0.0692 (19)	0.0420 (14)	0.0536 (17)	0.0012 (13)	−0.0305 (15)	−0.0099 (12)
C4	0.081 (2)	0.0408 (15)	0.0654 (19)	−0.0191 (15)	−0.0290 (18)	0.0086 (13)
C38	0.0612 (18)	0.0423 (14)	0.0514 (16)	−0.0013 (13)	−0.0232 (14)	−0.0059 (12)
C16	0.0607 (17)	0.0362 (13)	0.0479 (15)	−0.0107 (12)	−0.0128 (13)	−0.0066 (11)
C28	0.0670 (18)	0.0355 (13)	0.0474 (15)	−0.0110 (12)	−0.0210 (13)	−0.0029 (11)
C1	0.0693 (19)	0.0379 (14)	0.0622 (18)	−0.0132 (13)	−0.0177 (16)	−0.0027 (13)
C9	0.072 (2)	0.0417 (14)	0.0577 (17)	−0.0094 (13)	−0.0313 (16)	−0.0041 (13)
C23	0.0532 (17)	0.0526 (15)	0.0561 (16)	−0.0063 (13)	−0.0169 (13)	−0.0155 (13)
C8	0.0682 (18)	0.0390 (13)	0.0520 (16)	−0.0111 (13)	−0.0221 (14)	−0.0055 (12)
C3	0.066 (2)	0.0378 (14)	0.082 (2)	−0.0095 (13)	−0.0353 (18)	−0.0039 (14)
C2	0.0610 (18)	0.0472 (16)	0.080 (2)	−0.0084 (14)	−0.0171 (16)	−0.0132 (15)
C5	0.0671 (18)	0.0440 (15)	0.0539 (16)	−0.0175 (13)	−0.0186 (14)	−0.0011 (12)
C34	0.079 (2)	0.0525 (18)	0.079 (2)	0.0094 (16)	−0.0411 (19)	−0.0228 (16)
C29	0.077 (2)	0.0399 (14)	0.0447 (16)	−0.0096 (13)	−0.0219 (15)	−0.0011 (12)
C37	0.0594 (19)	0.0588 (18)	0.073 (2)	−0.0011 (15)	−0.0200 (17)	−0.0062 (16)
C30	0.082 (2)	0.0362 (14)	0.0572 (17)	−0.0094 (13)	−0.0330 (16)	−0.0020 (12)
C31	0.100 (2)	0.0431 (16)	0.077 (2)	−0.0200 (16)	−0.0420 (19)	0.0045 (15)
C12	0.106 (3)	0.070 (2)	0.111 (3)	−0.028 (2)	−0.066 (3)	−0.008 (2)
C10	0.105 (3)	0.0562 (17)	0.0602 (19)	−0.0328 (17)	−0.0207 (18)	−0.0078 (15)
C25	0.074 (2)	0.0631 (19)	0.074 (2)	−0.0123 (16)	−0.0220 (17)	−0.0280 (16)
C27	0.0538 (19)	0.093 (2)	0.088 (2)	−0.0225 (17)	−0.0032 (17)	−0.045 (2)
C17	0.087 (2)	0.071 (2)	0.074 (2)	−0.0421 (18)	0.0139 (19)	−0.0241 (17)

C14	0.067 (2)	0.086 (2)	0.106 (3)	−0.0006 (18)	−0.037 (2)	−0.042 (2)
C13	0.068 (2)	0.103 (3)	0.131 (3)	−0.017 (2)	−0.043 (2)	−0.034 (3)
C11	0.132 (3)	0.063 (2)	0.076 (2)	−0.035 (2)	−0.038 (2)	−0.0168 (17)
C32	0.123 (3)	0.0386 (17)	0.107 (3)	−0.0197 (19)	−0.067 (3)	−0.0030 (18)
C26	0.065 (2)	0.099 (3)	0.110 (3)	−0.0075 (19)	−0.009 (2)	−0.066 (2)
C33	0.112 (3)	0.0479 (19)	0.116 (3)	0.0064 (19)	−0.058 (3)	−0.033 (2)
C36	0.065 (2)	0.087 (3)	0.082 (2)	0.0141 (19)	−0.0155 (18)	−0.015 (2)
C35	0.082 (3)	0.077 (2)	0.091 (3)	0.027 (2)	−0.028 (2)	−0.039 (2)

Geometric parameters (Å, °)

Cl1—C3	1.743 (3)	C28—C29	1.575 (4)
Cl2—C25	1.735 (3)	C1—C2	1.388 (4)
O1—C20	1.211 (3)	C1—H1	0.9300
O2—C29	1.209 (3)	C9—C10	1.376 (4)
N2—C17	1.447 (3)	C9—C14	1.378 (4)
N2—C18	1.449 (3)	C9—C8	1.510 (4)
N2—C16	1.459 (3)	C23—H23	0.9300
N1—C8	1.458 (3)	C8—H8	0.9800
N1—C28	1.463 (3)	C3—C2	1.369 (4)
N1—H1N	0.8600	C2—H2	0.9300
C15—C20	1.516 (3)	C5—H5	0.9300
C15—C16	1.527 (4)	C34—C35	1.391 (5)
C15—C7	1.550 (3)	C34—C33	1.421 (5)
C15—C28	1.608 (3)	C29—C30	1.491 (4)
C7—C6	1.513 (3)	C37—C36	1.405 (5)
C7—C8	1.535 (3)	C37—H37	0.9300
C7—H7	0.9800	C30—C31	1.380 (4)
C20—C19	1.505 (3)	C31—C32	1.409 (5)
C6—C5	1.384 (4)	C31—H31	0.9300
C6—C1	1.386 (4)	C12—C11	1.354 (5)
C22—C27	1.381 (4)	C12—C13	1.367 (5)
C22—C23	1.386 (4)	C12—H12	0.9300
C22—C21	1.470 (4)	C10—C11	1.386 (5)
C24—C25	1.360 (4)	C10—H10	0.9300
C24—C23	1.384 (4)	C25—C26	1.369 (4)
C24—H24	0.9300	C27—C26	1.381 (4)
C19—C21	1.335 (4)	C27—H27	0.9300
C19—C18	1.495 (4)	C17—H17A	0.9600
C21—H21	0.9300	C17—H17B	0.9600
C18—H18A	0.9700	C17—H17C	0.9600
C18—H18B	0.9700	C14—C13	1.385 (5)
C39—C30	1.389 (4)	C14—H14	0.9300
C39—C34	1.406 (4)	C13—H13	0.9300
C39—C38	1.407 (4)	C11—H11	0.9300
C4—C3	1.359 (4)	C32—C33	1.358 (5)
C4—C5	1.389 (4)	C32—H32	0.9300
C4—H4	0.9300	C26—H26	0.9300
C38—C37	1.360 (4)	C33—H33	0.9300

C38—C28	1.512 (4)	C36—C35	1.364 (5)
C16—H16A	0.9700	C36—H36	0.9300
C16—H16B	0.9700	C35—H35	0.9300
C17—N2—C18	111.3 (2)	N1—C8—C9	112.6 (2)
C17—N2—C16	112.4 (2)	N1—C8—C7	100.4 (2)
C18—N2—C16	113.4 (2)	C9—C8—C7	114.1 (2)
C8—N1—C28	110.7 (2)	N1—C8—H8	109.8
C8—N1—H1N	124.7	C9—C8—H8	109.8
C28—N1—H1N	124.7	C7—C8—H8	109.8
C20—C15—C16	106.4 (2)	C4—C3—C2	121.1 (3)
C20—C15—C7	112.3 (2)	C4—C3—Cl1	120.4 (2)
C16—C15—C7	113.7 (2)	C2—C3—Cl1	118.6 (3)
C20—C15—C28	110.50 (19)	C3—C2—C1	119.0 (3)
C16—C15—C28	111.5 (2)	C3—C2—H2	120.5
C7—C15—C28	102.45 (18)	C1—C2—H2	120.5
C6—C7—C8	114.7 (2)	C6—C5—C4	121.2 (3)
C6—C7—C15	117.6 (2)	C6—C5—H5	119.4
C8—C7—C15	103.61 (19)	C4—C5—H5	119.4
C6—C7—H7	106.7	C35—C34—C39	116.6 (3)
C8—C7—H7	106.7	C35—C34—C33	128.6 (3)
C15—C7—H7	106.7	C39—C34—C33	114.8 (3)
O1—C20—C19	121.1 (2)	O2—C29—C30	127.8 (3)
O1—C20—C15	122.4 (2)	O2—C29—C28	124.3 (2)
C19—C20—C15	116.5 (2)	C30—C29—C28	106.6 (2)
C5—C6—C1	117.5 (2)	C38—C37—C36	119.2 (3)
C5—C6—C7	119.5 (2)	C38—C37—H37	120.4
C1—C6—C7	122.8 (2)	C36—C37—H37	120.4
C27—C22—C23	117.2 (2)	C31—C30—C39	119.8 (3)
C27—C22—C21	118.3 (3)	C31—C30—C29	132.1 (3)
C23—C22—C21	124.5 (2)	C39—C30—C29	107.9 (2)
C25—C24—C23	119.2 (3)	C30—C31—C32	117.4 (3)
C25—C24—H24	120.4	C30—C31—H31	121.3
C23—C24—H24	120.4	C32—C31—H31	121.3
C21—C19—C18	125.2 (2)	C11—C12—C13	120.7 (3)
C21—C19—C20	117.0 (2)	C11—C12—H12	119.7
C18—C19—C20	117.8 (2)	C13—C12—H12	119.7
C19—C21—C22	130.3 (3)	C9—C10—C11	121.5 (3)
C19—C21—H21	114.9	C9—C10—H10	119.2
C22—C21—H21	114.9	C11—C10—H10	119.2
N2—C18—C19	113.2 (2)	C24—C25—C26	121.2 (3)
N2—C18—H18A	108.9	C24—C25—Cl2	119.4 (2)
C19—C18—H18A	108.9	C26—C25—Cl2	119.3 (2)
N2—C18—H18B	108.9	C22—C27—C26	121.9 (3)
C19—C18—H18B	108.9	C22—C27—H27	119.0
H18A—C18—H18B	107.8	C26—C27—H27	119.0
C30—C39—C34	124.0 (3)	N2—C17—H17A	109.5
C30—C39—C38	113.1 (2)	N2—C17—H17B	109.5
C34—C39—C38	122.9 (3)	H17A—C17—H17B	109.5
C3—C4—C5	119.6 (3)	N2—C17—H17C	109.5

C3—C4—H4	120.2	H17A—C17—H17C	109.5
C5—C4—H4	120.2	H17B—C17—H17C	109.5
C37—C38—C39	118.5 (3)	C9—C14—C13	121.4 (3)
C37—C38—C28	132.1 (2)	C9—C14—H14	119.3
C39—C38—C28	109.4 (2)	C13—C14—H14	119.3
N2—C16—C15	107.9 (2)	C12—C13—C14	119.3 (4)
N2—C16—H16A	110.1	C12—C13—H13	120.4
C15—C16—H16A	110.1	C14—C13—H13	120.4
N2—C16—H16B	110.1	C12—C11—C10	119.6 (3)
C15—C16—H16B	110.1	C12—C11—H11	120.2
H16A—C16—H16B	108.4	C10—C11—H11	120.2
N1—C28—C38	112.1 (2)	C33—C32—C31	122.7 (3)
N1—C28—C29	112.1 (2)	C33—C32—H32	118.6
C38—C28—C29	102.2 (2)	C31—C32—H32	118.6
N1—C28—C15	103.73 (18)	C25—C26—C27	118.9 (3)
C38—C28—C15	114.5 (2)	C25—C26—H26	120.6
C29—C28—C15	112.6 (2)	C27—C26—H26	120.6
C6—C1—C2	121.5 (3)	C32—C33—C34	121.3 (3)
C6—C1—H1	119.2	C32—C33—H33	119.3
C2—C1—H1	119.2	C34—C33—H33	119.3
C10—C9—C14	117.5 (3)	C35—C36—C37	122.1 (4)
C10—C9—C8	121.3 (3)	C35—C36—H36	119.0
C14—C9—C8	121.3 (3)	C37—C36—H36	119.0
C24—C23—C22	121.6 (3)	C36—C35—C34	120.7 (3)
C24—C23—H23	119.2	C36—C35—H35	119.7
C22—C23—H23	119.2	C34—C35—H35	119.7
C20—C15—C7—C6	82.7 (3)	C10—C9—C8—N1	−130.0 (3)
C16—C15—C7—C6	−38.3 (3)	C14—C9—C8—N1	51.3 (4)
C28—C15—C7—C6	−158.8 (2)	C10—C9—C8—C7	116.4 (3)
C20—C15—C7—C8	−149.5 (2)	C14—C9—C8—C7	−62.3 (4)
C16—C15—C7—C8	89.5 (2)	C6—C7—C8—N1	172.6 (2)
C28—C15—C7—C8	−30.9 (2)	C15—C7—C8—N1	43.1 (3)
C16—C15—C20—O1	133.2 (2)	C6—C7—C8—C9	−66.7 (3)
C7—C15—C20—O1	8.1 (3)	C15—C7—C8—C9	163.7 (2)
C28—C15—C20—O1	−105.6 (3)	C5—C4—C3—C2	−1.1 (4)
C16—C15—C20—C19	−45.8 (3)	C5—C4—C3—Cl1	178.5 (2)
C7—C15—C20—C19	−170.8 (2)	C4—C3—C2—C1	1.5 (4)
C28—C15—C20—C19	75.4 (3)	Cl1—C3—C2—C1	−178.2 (2)
C8—C7—C6—C5	134.4 (3)	C6—C1—C2—C3	−0.5 (4)
C15—C7—C6—C5	−103.3 (3)	C1—C6—C5—C4	1.1 (4)
C8—C7—C6—C1	−41.9 (3)	C7—C6—C5—C4	−175.4 (2)
C15—C7—C6—C1	80.4 (3)	C3—C4—C5—C6	−0.2 (4)
O1—C20—C19—C21	32.7 (4)	C30—C39—C34—C35	−178.6 (3)
C15—C20—C19—C21	−148.3 (2)	C38—C39—C34—C35	−1.7 (4)
O1—C20—C19—C18	−149.9 (2)	C30—C39—C34—C33	−0.3 (4)
C15—C20—C19—C18	29.0 (3)	C38—C39—C34—C33	176.6 (3)
C18—C19—C21—C22	0.3 (4)	N1—C28—C29—O2	39.2 (4)
C20—C19—C21—C22	177.4 (2)	C38—C28—C29—O2	159.4 (3)
C27—C22—C21—C19	−152.9 (3)	C15—C28—C29—O2	−77.3 (3)

C23—C22—C21—C19	28.5 (4)	N1—C28—C29—C30	−129.1 (2)
C17—N2—C18—C19	176.1 (2)	C38—C28—C29—C30	−8.8 (3)
C16—N2—C18—C19	48.2 (3)	C15—C28—C29—C30	114.4 (2)
C21—C19—C18—N2	149.3 (2)	C39—C38—C37—C36	3.0 (4)
C20—C19—C18—N2	−27.8 (3)	C28—C38—C37—C36	−176.1 (3)
C30—C39—C38—C37	176.3 (2)	C34—C39—C30—C31	0.1 (4)
C34—C39—C38—C37	−1.0 (4)	C38—C39—C30—C31	−177.1 (3)
C30—C39—C38—C28	−4.5 (3)	C34—C39—C30—C29	175.6 (2)
C34—C39—C38—C28	178.3 (2)	C38—C39—C30—C29	−1.6 (3)
C17—N2—C16—C15	164.1 (2)	O2—C29—C30—C31	13.8 (5)
C18—N2—C16—C15	−68.6 (3)	C28—C29—C30—C31	−178.5 (3)
C20—C15—C16—N2	64.1 (2)	O2—C29—C30—C39	−161.0 (3)
C7—C15—C16—N2	−171.7 (2)	C28—C29—C30—C39	6.7 (3)
C28—C15—C16—N2	−56.5 (3)	C39—C30—C31—C32	0.4 (4)
C8—N1—C28—C38	144.7 (2)	C29—C30—C31—C32	−173.8 (3)
C8—N1—C28—C29	−101.1 (2)	C14—C9—C10—C11	−0.2 (5)
C8—N1—C28—C15	20.6 (3)	C8—C9—C10—C11	−179.0 (3)
C37—C38—C28—N1	−52.6 (4)	C23—C24—C25—C26	−1.4 (5)
C39—C38—C28—N1	128.3 (2)	C23—C24—C25—Cl2	−179.9 (2)
C37—C38—C28—C29	−172.8 (3)	C23—C22—C27—C26	−1.8 (5)
C39—C38—C28—C29	8.1 (3)	C21—C22—C27—C26	179.6 (3)
C37—C38—C28—C15	65.2 (4)	C10—C9—C14—C13	−0.7 (5)
C39—C38—C28—C15	−113.9 (2)	C8—C9—C14—C13	178.0 (3)
C20—C15—C28—N1	127.4 (2)	C11—C12—C13—C14	0.8 (6)
C16—C15—C28—N1	−114.5 (2)	C9—C14—C13—C12	0.5 (6)
C7—C15—C28—N1	7.5 (3)	C13—C12—C11—C10	−1.8 (6)
C20—C15—C28—C38	4.9 (3)	C9—C10—C11—C12	1.5 (5)
C16—C15—C28—C38	123.0 (2)	C30—C31—C32—C33	−0.6 (5)
C7—C15—C28—C38	−115.0 (2)	C24—C25—C26—C27	1.2 (6)
C20—C15—C28—C29	−111.2 (2)	Cl2—C25—C26—C27	179.8 (3)
C16—C15—C28—C29	6.9 (3)	C22—C27—C26—C25	0.4 (6)
C7—C15—C28—C29	128.9 (2)	C31—C32—C33—C34	0.4 (5)
C5—C6—C1—C2	−0.8 (4)	C35—C34—C33—C32	178.2 (3)
C7—C6—C1—C2	175.6 (2)	C39—C34—C33—C32	0.1 (5)
C25—C24—C23—C22	−0.1 (4)	C38—C37—C36—C35	−2.4 (5)
C27—C22—C23—C24	1.6 (4)	C37—C36—C35—C34	−0.4 (5)
C21—C22—C23—C24	−179.8 (3)	C39—C34—C35—C36	2.3 (5)
C28—N1—C8—C9	−162.0 (2)	C33—C34—C35—C36	−175.7 (3)
C28—N1—C8—C7	−40.3 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O1	0.98	2.41	2.789 (3)	102
C21—H21···O1	0.93	2.46	2.810 (4)	102
C27—H27···O1^{i}	0.93	2.47	3.358 (4)	160
C16—H16A···O2^{ii}	0.97	2.56	3.491 (3)	161
C32—H32···Cg^{iii}	0.93	2.83	3.498 (5)	129

Symmetry codes: (i) −*x*+2, −*y*, −*z*; (ii) −*x*+1, −*y*, −*z*+1; (iii) *x*, *y*−1, *z*.

Fig. 1

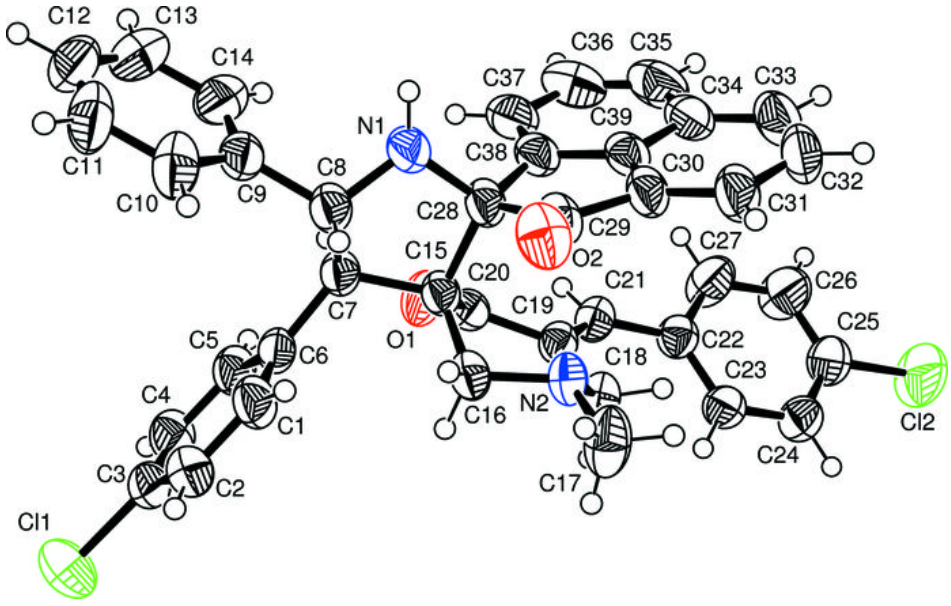

Fig. 2