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5″-(4-Chlorobenzylidene)-1′,1″-dimethyl-3′-phenylacenaphthene-1-spiro-2′-pyrrolidine-3′-spiro-3″-pyridine-2,4″-dione

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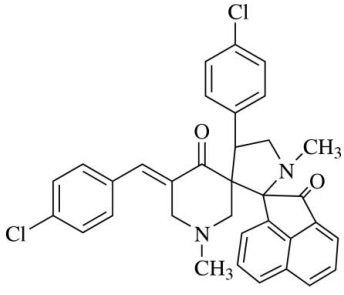
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.042; wR factor = 0.122; data-to-parameter ratio = 13.7.

In the title compound, $C_{34}H_{28}Cl_2N_2O_2$, the five-membered pyrrolidine ring adopts an envelope conformation and the six-membered piperidinone ring is in a distorted half-chair conformation. The molecular structure shows three intramolecular C—H···O interactions and the crystal packing is stabilized through intermolecular C—H···O and C—H···π interactions.

Related literature

For the biological importance of pyrrolidines, 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 puckering analysis, see: Cremer & Pople (1975). For hydrogen-bonding interactions, see: Desiraju & Steiner (1999).



Experimental

Crystal data

$C_{34}H_{28}Cl_2N_2O_2$
M_r = 567.48
Monoclinic, $P2_1/c$
a = 8.6561 (5) Å
b = 13.4732 (8) Å
c = 24.3962 (14) Å
β = 95.765 (12)°
V = 2830.8 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 293 (2) K
0.22 × 0.19 × 0.15 mm

Data collection

Nonius MACH3 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.963, T_{max} = 0.991
5802 measured reflections
4962 independent reflections
3252 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.122
S = 1.01
4962 reflections
361 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

*Cg*1 is the centroid of the C1–C6 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O1	0.93	2.40	2.783 (3)	104
C14—H14···O1	0.98	2.44	2.818 (3)	102
C22—H22*C*···O2	0.96	2.56	3.101 (3)	116
C26—H26···O1^{i}	0.93	2.38	3.307 (3)	176
C21—H21*B*···*Cg*1^{ii}	0.97	2.73	3.559 (3)	144

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

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: BT2624).

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5′′-(4-Chlorobenzylidene)-1′,1′′-dimethyl-3′-phenylacenaphthene-1-spiro-2′-pyrrolidine-3′-spiro-3′′-pyridine-2,4′′-dione

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S1. 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 (Chande *et al.*, 2005).

The envelope conformation of the five-membered ring in (I), is observed through the puckering analysis [q_2 = 0.446 (2) Å and φ_2 = 43.2 (3)°; Cremer & Pople, 1975] and the six-membered ring adopts distorted half-chair conformation [q_2 = 0.289 (3) Å, φ_2 = 117.1 (6)° and q_3 = -0.454 (2) Å] (Fig. 1). The dihedral angle between the chlorophenyl rings are 86.1 (1)° and these rings are making angles of 35.6 (1) and 51.7 (1)° with the acenaphthene group.

The molecular structure of the title compound shows three intramolecular hydrogen bonds (Desiraju & Steiner, 1999). The crystal packing is stabilized through intermolecular C—H···O and C—H···π interactions (Fig. 2; Table 1). Atom H21B interacts with the centroid of the ring C1–C6.

S2. Experimental

A mixture of 1-methyl-3,5-bis[(*E*)-4-chlorophenylmethylidene]tetrahydro-4(1*H*)-pyridinone 1 mmol), acenaphthenequinone (1 mmol) and sarcosine (1 mmol) was dissolved in methanol (10 ml) and refluxed for 1 h. After completion of the reaction as evident from TLC, the mixture was poured into water (50 ml), the precipitated solid was filtered and washed with water (100 ml) to obtain pure 1-Methyl-4-(4-chlorophenyl)pyrrolo-(spiro[2.2′′]-acenaphthene-1′′)- spiro-[3.3′]-5′-(4-chlorophenyl-methylidene)-1′-methyltetrahydro-4′-(1*H*)- pyridinone as pale yellow solid.

S3. Refinement

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

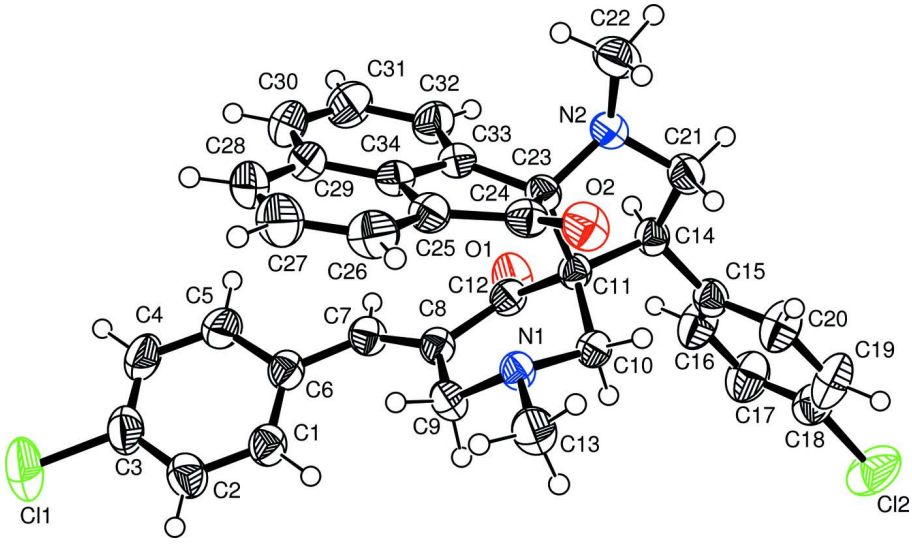

Figure 1

The molecular structure of the title compound with the numbering scheme for the atoms and 50% probability displacement ellipsoids.

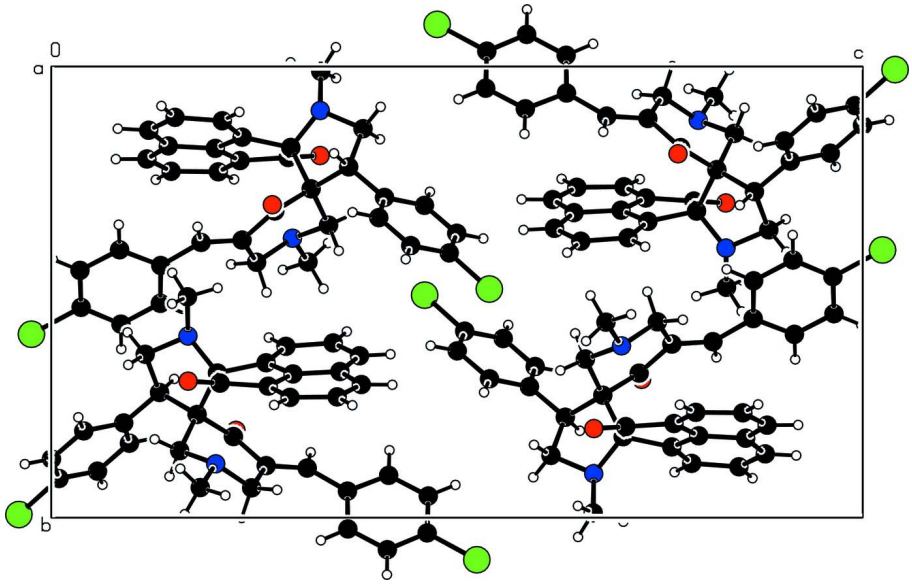

Figure 2

Packing diagram of the molecules, viewed down the *a*-axis.

5′′-(4-Chlorobenzylidene)-1′,1′′-dimethyl-3′-phenylacenaphthene-1-spiro-2′- pyrrolidine-3′-spiro-3′′-pyridine-2,4′′-dione

Crystal data

$C_{34}H_{28}Cl_2N_2O_2$
$M_r = 567.48$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.6561\ (5)$ Å
$b = 13.4732\ (8)$ Å
$c = 24.3962\ (14)$ Å
$\beta = 95.765\ (12)°$
$V = 2830.8\ (3)$ Å^3
$Z = 4$

$F(000) = 1184$
$D_x = 1.332$ Mg m^{-3}
Melting point: 188 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections

$\theta = 10.5–13.6°$
$\mu = 0.26$ mm^{-1}
$T = 293$ K
Block, yellow
0.22 × 0.19 × 0.15 mm

Data collection

Nonius MACH3 sealed-tube diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.963$, $T_{max} = 0.991$
5802 measured reflections

4962 independent reflections
3252 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.3°$
$h = 0 \rightarrow 10$
$k = -1 \rightarrow 16$
$l = -28 \rightarrow 28$
3 standard reflections every 60 min
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.122$
$S = 1.01$
4962 reflections
361 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
$w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 1.3189P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl2	0.79695 (10)	0.00677 (7)	1.03985 (4)	0.0837 (3)
Cl1	0.16195 (12)	−0.09337 (8)	0.47557 (3)	0.0930 (3)
O1	0.59114 (19)	0.19348 (15)	0.77239 (7)	0.0545 (5)
O2	0.01134 (19)	0.30256 (13)	0.83128 (7)	0.0489 (4)
N1	0.1503 (2)	0.11928 (13)	0.79730 (7)	0.0337 (4)
N2	0.3200 (2)	0.40244 (14)	0.83147 (8)	0.0401 (5)
C11	0.3686 (2)	0.22873 (16)	0.82027 (8)	0.0319 (5)
C12	0.4534 (3)	0.17972 (17)	0.77550 (9)	0.0359 (5)
C23	0.2670 (2)	0.32052 (16)	0.79520 (9)	0.0335 (5)
C34	0.1497 (3)	0.31854 (16)	0.70216 (9)	0.0377 (5)

C14	0.4792 (3)	0.27742 (17)	0.86653 (9)	0.0355 (5)
H14	0.5639	0.3075	0.8485	0.043*
C15	0.5539 (3)	0.20976 (17)	0.91073 (9)	0.0381 (5)
C33	0.2867 (3)	0.34333 (16)	0.73527 (9)	0.0363 (5)
C10	0.2658 (3)	0.14841 (17)	0.84174 (9)	0.0347 (5)
H10A	0.3283	0.0915	0.8543	0.042*
H10B	0.2150	0.1737	0.8726	0.042*
C9	0.2157 (3)	0.06607 (18)	0.75306 (9)	0.0397 (5)
H9A	0.1378	0.0615	0.7217	0.048*
H9B	0.2418	−0.0009	0.7653	0.048*
C8	0.3586 (3)	0.11524 (16)	0.73517 (8)	0.0347 (5)
C6	0.3385 (3)	0.05882 (18)	0.63499 (9)	0.0404 (6)
C32	0.4090 (3)	0.3797 (2)	0.71080 (10)	0.0494 (6)
H32	0.4994	0.3992	0.7318	0.059*
C25	0.0276 (3)	0.29142 (17)	0.73259 (9)	0.0393 (5)
C28	−0.0111 (4)	0.2950 (2)	0.61790 (11)	0.0602 (8)
H28	−0.0268	0.2946	0.5796	0.072*
C21	0.3811 (3)	0.36211 (18)	0.88423 (9)	0.0417 (6)
H21A	0.2987	0.3386	0.9051	0.050*
H21B	0.4434	0.4106	0.9060	0.050*
C7	0.4055 (3)	0.10997 (18)	0.68459 (9)	0.0403 (5)
H7	0.4962	0.1448	0.6803	0.048*
C1	0.2430 (3)	−0.02498 (18)	0.63447 (10)	0.0433 (6)
H1	0.2161	−0.0503	0.6677	0.052*
C24	0.0865 (3)	0.29821 (17)	0.79197 (10)	0.0383 (5)
C2	0.1876 (3)	−0.0710 (2)	0.58587 (10)	0.0495 (6)
H2	0.1237	−0.1264	0.5863	0.059*
C30	0.2659 (4)	0.3587 (2)	0.62012 (11)	0.0594 (7)
H30	0.2638	0.3622	0.5820	0.071*
C20	0.4931 (3)	0.1922 (2)	0.95991 (10)	0.0581 (7)
H20	0.4002	0.2223	0.9666	0.070*
C5	0.3761 (3)	0.0938 (2)	0.58421 (10)	0.0542 (7)
H5	0.4396	0.1493	0.5832	0.065*
C4	0.3216 (4)	0.0480 (2)	0.53541 (10)	0.0632 (8)
H4	0.3479	0.0726	0.5020	0.076*
C27	−0.1289 (4)	0.2677 (2)	0.64769 (12)	0.0646 (8)
H27	−0.2232	0.2490	0.6289	0.078*
C26	−0.1144 (3)	0.2666 (2)	0.70582 (12)	0.0541 (7)
H26	−0.1977	0.2497	0.7252	0.065*
C13	0.0192 (3)	0.0667 (2)	0.81641 (10)	0.0503 (6)
H13A	−0.0529	0.0493	0.7854	0.075*
H13B	−0.0314	0.1086	0.8409	0.075*
H13C	0.0554	0.0075	0.8355	0.075*
C18	0.7050 (3)	0.0867 (2)	0.99047 (11)	0.0529 (7)
C3	0.2282 (3)	−0.0340 (2)	0.53678 (10)	0.0556 (7)
C16	0.6941 (3)	0.1650 (2)	0.90364 (11)	0.0581 (7)
H16	0.7397	0.1770	0.8713	0.070*
C17	0.7690 (3)	0.1030 (2)	0.94300 (11)	0.0635 (8)

H17	0.8623	0.0728	0.9368	0.076*
C19	0.5681 (4)	0.1303 (3)	0.99969 (11)	0.0674 (9)
H19	0.5248	0.1187	1.0325	0.081*
C29	0.1345 (3)	0.32391 (18)	0.64422 (10)	0.0474 (6)
C22	0.2206 (3)	0.48950 (19)	0.83214 (12)	0.0555 (7)
H22A	0.1872	0.5091	0.7950	0.083*
H22B	0.2775	0.5429	0.8508	0.083*
H22C	0.1316	0.4740	0.8511	0.083*
C31	0.3962 (4)	0.3872 (2)	0.65256 (11)	0.0622 (8)
H31	0.4796	0.4126	0.6358	0.075*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl2	0.0812 (6)	0.0873 (6)	0.0762 (5)	−0.0048 (5)	−0.0242 (4)	0.0362 (5)
Cl1	0.1263 (8)	0.1056 (7)	0.0430 (4)	−0.0021 (6)	−0.0113 (4)	−0.0216 (4)
O1	0.0353 (10)	0.0801 (13)	0.0498 (10)	−0.0078 (9)	0.0125 (8)	−0.0128 (9)
O2	0.0444 (9)	0.0534 (11)	0.0518 (10)	0.0026 (8)	0.0182 (8)	−0.0063 (8)
N1	0.0334 (10)	0.0378 (10)	0.0308 (9)	−0.0055 (8)	0.0082 (8)	−0.0027 (8)
N2	0.0466 (11)	0.0340 (10)	0.0388 (10)	0.0000 (9)	−0.0001 (9)	−0.0031 (8)
C11	0.0319 (11)	0.0348 (12)	0.0294 (11)	−0.0015 (9)	0.0054 (9)	−0.0003 (9)
C12	0.0343 (13)	0.0387 (13)	0.0356 (12)	−0.0006 (10)	0.0079 (10)	0.0025 (10)
C23	0.0321 (11)	0.0345 (12)	0.0342 (11)	−0.0031 (10)	0.0043 (9)	−0.0030 (9)
C34	0.0417 (13)	0.0314 (12)	0.0396 (12)	0.0022 (10)	0.0025 (10)	0.0010 (10)
C14	0.0334 (11)	0.0405 (13)	0.0325 (11)	−0.0049 (10)	0.0032 (9)	−0.0018 (10)
C15	0.0361 (13)	0.0419 (13)	0.0355 (12)	−0.0056 (11)	0.0003 (10)	−0.0029 (10)
C33	0.0395 (13)	0.0322 (12)	0.0374 (12)	−0.0018 (10)	0.0044 (10)	0.0021 (9)
C10	0.0369 (12)	0.0361 (12)	0.0317 (11)	−0.0014 (10)	0.0069 (9)	0.0006 (9)
C9	0.0399 (12)	0.0415 (13)	0.0381 (12)	−0.0048 (11)	0.0057 (10)	−0.0084 (10)
C8	0.0353 (12)	0.0364 (12)	0.0330 (11)	0.0057 (10)	0.0069 (9)	−0.0020 (10)
C6	0.0455 (13)	0.0423 (13)	0.0344 (12)	0.0088 (12)	0.0087 (10)	−0.0021 (10)
C32	0.0511 (15)	0.0525 (16)	0.0451 (14)	−0.0128 (13)	0.0083 (12)	0.0064 (12)
C25	0.0360 (12)	0.0378 (13)	0.0430 (13)	0.0032 (11)	−0.0012 (10)	0.0001 (10)
C28	0.079 (2)	0.0536 (17)	0.0435 (15)	−0.0013 (15)	−0.0166 (14)	−0.0021 (13)
C21	0.0456 (14)	0.0420 (13)	0.0370 (12)	−0.0065 (11)	0.0028 (10)	−0.0056 (10)
C7	0.0404 (13)	0.0424 (13)	0.0394 (13)	0.0009 (11)	0.0106 (10)	−0.0025 (11)
C1	0.0490 (14)	0.0457 (14)	0.0364 (12)	0.0062 (12)	0.0097 (11)	−0.0025 (11)
C24	0.0366 (12)	0.0349 (13)	0.0444 (13)	0.0022 (10)	0.0089 (11)	−0.0009 (10)
C2	0.0529 (15)	0.0507 (15)	0.0447 (14)	0.0044 (13)	0.0046 (12)	−0.0083 (12)
C30	0.084 (2)	0.0600 (17)	0.0349 (14)	−0.0036 (16)	0.0117 (14)	0.0073 (13)
C20	0.0528 (16)	0.078 (2)	0.0446 (15)	0.0064 (15)	0.0097 (12)	0.0115 (14)
C5	0.0750 (19)	0.0486 (15)	0.0409 (14)	−0.0019 (14)	0.0147 (13)	0.0030 (12)
C4	0.097 (2)	0.0628 (19)	0.0303 (13)	0.0078 (18)	0.0092 (14)	0.0061 (13)
C27	0.0573 (18)	0.0663 (19)	0.0642 (18)	−0.0066 (15)	−0.0237 (15)	−0.0011 (15)
C26	0.0410 (14)	0.0542 (17)	0.0654 (18)	−0.0037 (13)	−0.0036 (12)	0.0035 (14)
C13	0.0458 (14)	0.0588 (16)	0.0479 (14)	−0.0162 (13)	0.0131 (12)	−0.0055 (13)
C18	0.0520 (16)	0.0557 (17)	0.0472 (15)	−0.0054 (13)	−0.0130 (12)	0.0098 (13)
C3	0.0690 (18)	0.0595 (18)	0.0367 (14)	0.0136 (15)	−0.0026 (12)	−0.0080 (12)

C16	0.0562 (17)	0.075 (2)	0.0441 (15)	0.0151 (15)	0.0114 (13)	0.0113 (14)
C17	0.0539 (16)	0.080 (2)	0.0558 (17)	0.0180 (15)	0.0009 (13)	0.0116 (15)
C19	0.0661 (19)	0.096 (2)	0.0403 (15)	−0.0029 (18)	0.0058 (13)	0.0192 (16)
C29	0.0653 (17)	0.0386 (14)	0.0369 (13)	0.0038 (12)	−0.0014 (12)	0.0014 (11)
C22	0.0649 (17)	0.0398 (14)	0.0602 (17)	0.0069 (13)	−0.0015 (14)	−0.0080 (12)
C31	0.075 (2)	0.0643 (19)	0.0504 (16)	−0.0144 (16)	0.0210 (15)	0.0113 (14)

Geometric parameters (Å, º)

Cl2—C18	1.747 (3)	C25—C26	1.374 (3)
Cl1—C3	1.740 (3)	C25—C24	1.489 (3)
O1—C12	1.217 (3)	C28—C27	1.361 (4)
O2—C24	1.213 (3)	C28—C29	1.411 (4)
N1—C10	1.453 (3)	C28—H28	0.9300
N1—C13	1.454 (3)	C21—H21A	0.9700
N1—C9	1.457 (3)	C21—H21B	0.9700
N2—C21	1.447 (3)	C7—H7	0.9300
N2—C22	1.456 (3)	C1—C2	1.381 (3)
N2—C23	1.459 (3)	C1—H1	0.9300
C11—C12	1.526 (3)	C2—C3	1.376 (4)
C11—C10	1.527 (3)	C2—H2	0.9300
C11—C14	1.550 (3)	C30—C31	1.366 (4)
C11—C23	1.603 (3)	C30—C29	1.412 (4)
C12—C8	1.495 (3)	C30—H30	0.9300
C23—C33	1.520 (3)	C20—C19	1.390 (4)
C23—C24	1.586 (3)	C20—H20	0.9300
C34—C25	1.399 (3)	C5—C4	1.381 (4)
C34—C33	1.407 (3)	C5—H5	0.9300
C34—C29	1.408 (3)	C4—C3	1.371 (4)
C14—C15	1.507 (3)	C4—H4	0.9300
C14—C21	1.511 (3)	C27—C26	1.411 (4)
C14—H14	0.9800	C27—H27	0.9300
C15—C20	1.378 (3)	C26—H26	0.9300
C15—C16	1.381 (3)	C13—H13A	0.9600
C33—C32	1.358 (3)	C13—H13B	0.9600
C10—H10A	0.9700	C13—H13C	0.9600
C10—H10B	0.9700	C18—C17	1.351 (4)
C9—C8	1.506 (3)	C18—C19	1.361 (4)
C9—H9A	0.9700	C16—C17	1.384 (4)
C9—H9B	0.9700	C16—H16	0.9300
C8—C7	1.339 (3)	C17—H17	0.9300
C6—C5	1.394 (3)	C19—H19	0.9300
C6—C1	1.399 (3)	C22—H22A	0.9600
C6—C7	1.461 (3)	C22—H22B	0.9600
C32—C31	1.418 (4)	C22—H22C	0.9600
C32—H32	0.9300	C31—H31	0.9300
C10—N1—C13	113.15 (17)	N2—C21—H21B	111.5

C10—N1—C9	113.37 (17)	C14—C21—H21B	111.5
C13—N1—C9	111.81 (18)	H21A—C21—H21B	109.3
C21—N2—C22	117.14 (19)	C8—C7—C6	131.0 (2)
C21—N2—C23	108.65 (18)	C8—C7—H7	114.5
C22—N2—C23	117.80 (18)	C6—C7—H7	114.5
C12—C11—C10	106.12 (17)	C2—C1—C6	121.6 (2)
C12—C11—C14	113.47 (18)	C2—C1—H1	119.2
C10—C11—C14	112.87 (17)	C6—C1—H1	119.2
C12—C11—C23	110.21 (16)	O2—C24—C25	127.9 (2)
C10—C11—C23	111.17 (17)	O2—C24—C23	123.8 (2)
C14—C11—C23	103.08 (17)	C25—C24—C23	107.40 (18)
O1—C12—C8	121.6 (2)	C3—C2—C1	119.2 (3)
O1—C12—C11	121.6 (2)	C3—C2—H2	120.4
C8—C12—C11	116.82 (18)	C1—C2—H2	120.4
N2—C23—C33	111.81 (18)	C31—C30—C29	120.3 (2)
N2—C23—C24	114.92 (18)	C31—C30—H30	119.8
C33—C23—C24	101.28 (17)	C29—C30—H30	119.8
N2—C23—C11	103.02 (16)	C15—C20—C19	121.3 (3)
C33—C23—C11	114.38 (17)	C15—C20—H20	119.4
C24—C23—C11	111.90 (17)	C19—C20—H20	119.4
C25—C34—C33	113.3 (2)	C4—C5—C6	121.7 (3)
C25—C34—C29	123.1 (2)	C4—C5—H5	119.2
C33—C34—C29	123.5 (2)	C6—C5—H5	119.2
C15—C14—C21	117.74 (18)	C3—C4—C5	119.3 (2)
C15—C14—C11	117.01 (19)	C3—C4—H4	120.3
C21—C14—C11	101.79 (18)	C5—C4—H4	120.3
C15—C14—H14	106.5	C28—C27—C26	122.9 (3)
C21—C14—H14	106.5	C28—C27—H27	118.5
C11—C14—H14	106.5	C26—C27—H27	118.5
C20—C15—C16	116.8 (2)	C25—C26—C27	117.4 (3)
C20—C15—C14	123.6 (2)	C25—C26—H26	121.3
C16—C15—C14	119.6 (2)	C27—C26—H26	121.3
C32—C33—C34	118.9 (2)	N1—C13—H13A	109.5
C32—C33—C23	131.5 (2)	N1—C13—H13B	109.5
C34—C33—C23	109.56 (19)	H13A—C13—H13B	109.5
N1—C10—C11	108.45 (17)	N1—C13—H13C	109.5
N1—C10—H10A	110.0	H13A—C13—H13C	109.5
C11—C10—H10A	110.0	H13B—C13—H13C	109.5
N1—C10—H10B	110.0	C17—C18—C19	120.7 (2)
C11—C10—H10B	110.0	C17—C18—Cl2	119.5 (2)
H10A—C10—H10B	108.4	C19—C18—Cl2	119.7 (2)
N1—C9—C8	112.94 (18)	C4—C3—C2	121.1 (2)
N1—C9—H9A	109.0	C4—C3—Cl1	119.6 (2)
C8—C9—H9A	109.0	C2—C3—Cl1	119.3 (2)
N1—C9—H9B	109.0	C15—C16—C17	122.2 (2)
C8—C9—H9B	109.0	C15—C16—H16	118.9
H9A—C9—H9B	107.8	C17—C16—H16	118.9
C7—C8—C12	116.1 (2)	C18—C17—C16	119.2 (3)

C7—C8—C9	125.3 (2)	C18—C17—H17	120.4
C12—C8—C9	118.53 (18)	C16—C17—H17	120.4
C5—C6—C1	117.1 (2)	C18—C19—C20	119.8 (3)
C5—C6—C7	117.9 (2)	C18—C19—H19	120.1
C1—C6—C7	125.0 (2)	C20—C19—H19	120.1
C33—C32—C31	118.7 (2)	C34—C29—C28	115.6 (2)
C33—C32—H32	120.6	C34—C29—C30	115.9 (2)
C31—C32—H32	120.6	C28—C29—C30	128.5 (2)
C26—C25—C34	119.9 (2)	N2—C22—H22A	109.5
C26—C25—C24	132.8 (2)	N2—C22—H22B	109.5
C34—C25—C24	107.30 (19)	H22A—C22—H22B	109.5
C27—C28—C29	121.0 (3)	N2—C22—H22C	109.5
C27—C28—H28	119.5	H22A—C22—H22C	109.5
C29—C28—H28	119.5	H22B—C22—H22C	109.5
N2—C21—C14	101.25 (18)	C30—C31—C32	122.5 (3)
N2—C21—H21A	111.5	C30—C31—H31	118.7
C14—C21—H21A	111.5	C32—C31—H31	118.7

C10—C11—C12—O1	−136.4 (2)	C33—C34—C25—C26	179.1 (2)
C14—C11—C12—O1	−11.9 (3)	C29—C34—C25—C26	1.3 (4)
C23—C11—C12—O1	103.1 (2)	C33—C34—C25—C24	0.0 (3)
C10—C11—C12—C8	44.5 (2)	C29—C34—C25—C24	−177.8 (2)
C14—C11—C12—C8	168.98 (18)	C22—N2—C21—C14	−178.3 (2)
C23—C11—C12—C8	−76.0 (2)	C23—N2—C21—C14	45.1 (2)
C21—N2—C23—C33	−148.01 (19)	C15—C14—C21—N2	−174.89 (19)
C22—N2—C23—C33	75.8 (3)	C11—C14—C21—N2	−45.6 (2)
C21—N2—C23—C24	97.3 (2)	C12—C8—C7—C6	−177.0 (2)
C22—N2—C23—C24	−39.0 (3)	C9—C8—C7—C6	−0.2 (4)
C21—N2—C23—C11	−24.7 (2)	C5—C6—C7—C8	156.9 (3)
C22—N2—C23—C11	−160.95 (19)	C1—C6—C7—C8	−25.3 (4)
C12—C11—C23—N2	−126.14 (18)	C5—C6—C1—C2	−0.3 (4)
C10—C11—C23—N2	116.48 (18)	C7—C6—C1—C2	−178.1 (2)
C14—C11—C23—N2	−4.7 (2)	C26—C25—C24—O2	−16.8 (4)
C12—C11—C23—C33	−4.6 (2)	C34—C25—C24—O2	162.2 (2)
C10—C11—C23—C33	−121.96 (19)	C26—C25—C24—C23	174.4 (3)
C14—C11—C23—C33	116.85 (19)	C34—C25—C24—C23	−6.7 (2)
C12—C11—C23—C24	109.9 (2)	N2—C23—C24—O2	−38.6 (3)
C10—C11—C23—C24	−7.5 (2)	C33—C23—C24—O2	−159.3 (2)
C14—C11—C23—C24	−128.71 (18)	C11—C23—C24—O2	78.5 (3)
C12—C11—C14—C15	−80.6 (2)	N2—C23—C24—C25	130.85 (19)
C10—C11—C14—C15	40.2 (3)	C33—C23—C24—C25	10.2 (2)
C23—C11—C14—C15	160.19 (18)	C11—C23—C24—C25	−112.1 (2)
C12—C11—C14—C21	149.59 (19)	C6—C1—C2—C3	0.4 (4)
C10—C11—C14—C21	−89.6 (2)	C16—C15—C20—C19	−1.6 (4)
C23—C11—C14—C21	30.4 (2)	C14—C15—C20—C19	−179.0 (3)
C21—C14—C15—C20	28.1 (3)	C1—C6—C5—C4	0.1 (4)
C11—C14—C15—C20	−93.8 (3)	C7—C6—C5—C4	178.1 (3)
C21—C14—C15—C16	−149.2 (2)	C6—C5—C4—C3	−0.1 (4)

C11—C14—C15—C16	89.0 (3)	C29—C28—C27—C26	0.1 (5)
C25—C34—C33—C32	−174.0 (2)	C34—C25—C26—C27	1.4 (4)
C29—C34—C33—C32	3.8 (4)	C24—C25—C26—C27	−179.8 (3)
C25—C34—C33—C23	7.1 (3)	C28—C27—C26—C25	−2.1 (4)
C29—C34—C33—C23	−175.1 (2)	C5—C4—C3—C2	0.3 (4)
N2—C23—C33—C32	48.1 (3)	C5—C4—C3—Cl1	−178.8 (2)
C24—C23—C33—C32	171.0 (3)	C1—C2—C3—C4	−0.4 (4)
C11—C23—C33—C32	−68.5 (3)	C1—C2—C3—Cl1	178.66 (19)
N2—C23—C33—C34	−133.19 (19)	C20—C15—C16—C17	1.9 (4)
C24—C23—C33—C34	−10.3 (2)	C14—C15—C16—C17	179.4 (3)
C11—C23—C33—C34	110.2 (2)	C19—C18—C17—C16	0.3 (5)
C13—N1—C10—C11	−162.47 (19)	Cl2—C18—C17—C16	178.8 (2)
C9—N1—C10—C11	68.8 (2)	C15—C16—C17—C18	−1.3 (5)
C12—C11—C10—N1	−64.3 (2)	C17—C18—C19—C20	0.0 (5)
C14—C11—C10—N1	170.79 (17)	Cl2—C18—C19—C20	−178.6 (2)
C23—C11—C10—N1	55.5 (2)	C15—C20—C19—C18	0.7 (5)
C10—N1—C9—C8	−46.6 (3)	C25—C34—C29—C28	−3.1 (4)
C13—N1—C9—C8	−175.97 (19)	C33—C34—C29—C28	179.3 (2)
O1—C12—C8—C7	−28.8 (3)	C25—C34—C29—C30	176.0 (2)
C11—C12—C8—C7	150.3 (2)	C33—C34—C29—C30	−1.5 (4)
O1—C12—C8—C9	154.2 (2)	C27—C28—C29—C34	2.4 (4)
C11—C12—C8—C9	−26.7 (3)	C27—C28—C29—C30	−176.6 (3)
N1—C9—C8—C7	−151.4 (2)	C31—C30—C29—C34	−1.7 (4)
N1—C9—C8—C12	25.3 (3)	C31—C30—C29—C28	177.4 (3)
C34—C33—C32—C31	−2.7 (4)	C29—C30—C31—C32	2.7 (5)
C23—C33—C32—C31	175.9 (2)	C33—C32—C31—C30	−0.5 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O1	0.93	2.40	2.783 (3)	104
C14—H14···O1	0.98	2.44	2.818 (3)	102
C22—H22*C*···O2	0.96	2.56	3.101 (3)	116
C26—H26···O1^{i}	0.93	2.38	3.307 (3)	176
C21—H21*B*···*Cg*1^{ii}	0.97	2.73	3.559 (3)	144

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