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r-2,*c*-6-Bis(4-chlorophenyl)-*t*-3-isopropylpiperidin-4-one

A. Thiruvalluvar,[a]* S. Balamurugan,[a] R. J. Butcher,[b] A. Manimekalai[c] and S. Sivakumar[c]

[a]PG Research Department of Physics, Rajah Serfoji Government College (Autonomous), Thanjavur 613 005, Tamil Nadu, India, [b]Department of Chemistry, Howard University, 525 College Street NW, Washington, DC 20059, USA, and [c]Department of Chemistry, Annamalai University, Annamalai Nagar 608 002, Tamil Nadu, India
Correspondence e-mail: athiru@vsnl.net

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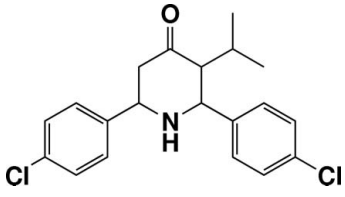
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.004 Å; R factor = 0.056; wR factor = 0.159; data-to-parameter ratio = 13.9.

The piperidine ring of the title molecule, $C_{20}H_{21}Cl_2NO$, is in a chair form. The dihedral angle between the two benzene rings is 52.4 (1)°. The chlorophenyl and isopropyl groups have equatorial orientations. Weak C—H···O and C—H···N intramolecular interactions were found in the crystal structure.

Related literature

For a related crystal structure, see: Balamurugan *et al.* (2007). For applications of piperidines, see: Jayabharathi *et al.* (2007).



Experimental

Crystal data

$C_{20}H_{21}Cl_2NO$
M_r = 362.28
Monoclinic, $P2_1/c$
a = 17.3013 (7) Å
b = 8.3742 (4) Å
c = 12.3687 (5) Å
β = 98.647 (4)°
V = 1771.66 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.37 mm^{-1}
T = 200 K
0.49 × 0.39 × 0.15 mm

Data collection

Oxford Diffraction Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
T_{min} = 0.83, T_{max} = 0.95
20264 measured reflections
3106 independent reflections
2262 reflections with $I > 2\sigma(I)$
R_{int} = 0.087

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.159
S = 1.05
3106 reflections
223 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.68 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C31—H31···O4	1.00	2.39	2.769 (4)	102
C66—H66···N1	0.95	2.49	2.832 (4)	101

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

RJB acknowledges the NSF–MRI program for funding to purchase the X-ray CCD diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2229).

References

Balamurugan, S., Thiruvalluvar, A., Manimekalai, A. & Jayabharathi, J. (2007). *Acta Cryst.* E**63**, o3504.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Jayabharathi, J., Manimekalai, A., Consalata Vani, T. & Padmavathy, M. (2007). *Eur. J. Med. Chem.* **42**, 593–605.
Oxford Diffraction (2007). *CrysAlis CCD* and *CrysAlis RED*. Versions 1.171.32. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

  doi:10.1107/S160053680705341X

supplementary materials

Acta Cryst. (2007). E**63**, o4482 [**doi:10.1107/S160053680705341X**]

r-2,*c*-6-Bis(4-chlorophenyl)-*t*-3-isopropylpiperidin-4-one

A. Thiruvalluvar, S. Balamurugan, R. J. Butcher, A. Manimekalai and S. Sivakumar

Comment

Jayabharathi *et al.* (2007) have reported synthesis, stereochemistry and antimicrobial evaluation of *t*-3-benzyl-*r*-2,*c*-6-diarylpiperidin-4-one and its derivatives. Balamurugan *et al.* (2007) have reported a crystal structure of *t*-3,*t*-5-dimethyl-*r*-2,*c*-6-diphenylpiperidin-4-one wherein the piperidine ring is in a chair form. The piperidine ring of the title molecule, $C_{20}H_{21}Cl_2NO$, is also in a chair form (Fig. 1). The dihedral angle between the two benzene rings is 52.4 (1)°. The *p*-chlorophenyl groups at the 2, 6 and the isopropyl group at the 3 position have equatorial orientations. Weak C31—H31···O4 and C66—H66···N1 intramolecular interactions were found in the crystal structure.

Experimental

A mixture of ammonium acetate (0.05 mol, 3.85 g), *p*-chlorobenzaldehyde (0.1 mol, 14 g) and isobutyl methyl ketone (0.05 mol, 8 ml) in distilled ethanol was heated first to boiling. After cooling, the viscous liquid obtained was dissolved in ether (200 ml) and shaken with 100 ml concentrated hydrochloric acid. The precipitated hydrochloride of *t*-3-isopropyl-*r*-2,*c*-6-bis(*p*-chlorophenyl)piperidin-4-one was removed by filtration and washed first with 40 ml mixture of ethanol and ether (1:1) and then with ether to remove most of the coloured impurities. The base was liberated from an alcoholic solution by adding aqueous ammonia and then diluted with water. It was recrystallized from alcohol. The yield of the isolated product was 3.5 g.

Refinement

The N-bound H atom was obtained from a difference Fourier map and refined isotropically. Other H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.95–1.00 Å, and with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

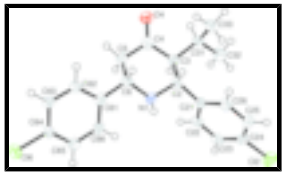


Fig. 1. The molecular structure of the title compound, with the atomic numbering and 50% probability displacement ellipsoids. H atoms are shown as small spheres of arbitrary radius.

r-2,*c*-6-Bis(4-chlorophenyl)-*t*-3-isopropylpiperidin-4-one

Crystal data

$C_{20}H_{21}Cl_2NO$	F_{000} = 760
M_r = 362.28	D_x = 1.358 Mg m^{-3}

Monoclinic, $P2_1/c$	Melting point: 374.5 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 17.3013 (7) Å	Cell parameters from 7357 reflections
b = 8.3742 (4) Å	θ = 4.5–32.6º
c = 12.3687 (5) Å	μ = 0.37 mm^{-1}
β = 98.647 (4)º	T = 200 K
V = 1771.66 (13) Å^3	Triangular plate, pale yellow
Z = 4	0.49 × 0.39 × 0.15 mm

Data collection

Oxford Diffraction Gemini diffractometer	3106 independent reflections
Radiation source: fine-focus sealed tube	2262 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.087
T = 200(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 4.5º
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)'	$h = -20 \rightarrow 20$
T_{min} = 0.83, T_{max} = 0.95	$k = -9 \rightarrow 9$
20264 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.056	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.159	$w = 1/[\sigma^2(F_o^2) + (0.0961P)^2 + 0.4302P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max} < 0.001$
3106 reflections	$\Delta\rho_{max}$ = 0.68 e Å^{-3}
223 parameters	$\Delta\rho_{min}$ = −0.47 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 $F^2 > 2\text{sigma}(F^2)$ 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}
Cl2	0.47429 (5)	1.43833 (11)	0.20225 (8)	0.0555 (3)
Cl6	−0.16197 (4)	0.82188 (9)	0.28263 (6)	0.0369 (3)
O4	0.21967 (14)	0.5598 (3)	−0.09979 (18)	0.0461 (8)
N1	0.17357 (14)	0.9389 (3)	0.07616 (18)	0.0282 (8)
C2	0.23615 (16)	0.9527 (3)	0.0085 (2)	0.0270 (8)
C3	0.27588 (17)	0.7871 (3)	0.0021 (2)	0.0295 (9)
C4	0.21346 (18)	0.6618 (3)	−0.0314 (2)	0.0335 (9)
C5	0.14275 (18)	0.6673 (3)	0.0264 (3)	0.0349 (9)
C6	0.10934 (17)	0.8363 (3)	0.0287 (2)	0.0282 (8)
C21	0.29219 (16)	1.0795 (3)	0.0581 (2)	0.0260 (8)
C22	0.32394 (17)	1.0747 (3)	0.1688 (2)	0.0308 (9)
C23	0.37782 (18)	1.1868 (4)	0.2133 (2)	0.0352 (9)
C24	0.40124 (18)	1.3055 (4)	0.1487 (3)	0.0358 (10)
C25	0.36874 (18)	1.3191 (4)	0.0393 (3)	0.0361 (10)
C26	0.31356 (18)	1.2058 (3)	−0.0041 (2)	0.0327 (9)
C31	0.34210 (17)	0.7759 (4)	−0.0675 (2)	0.0340 (9)
C32	0.42054 (19)	0.8408 (4)	−0.0111 (3)	0.0494 (11)
C33	0.3201 (2)	0.8477 (4)	−0.1822 (3)	0.0453 (11)
C61	0.04229 (17)	0.8390 (3)	0.0933 (2)	0.0278 (8)
C62	−0.02815 (18)	0.7682 (3)	0.0497 (2)	0.0312 (9)
C63	−0.09090 (18)	0.7606 (3)	0.1068 (2)	0.0305 (9)
C64	−0.08322 (17)	0.8306 (3)	0.2097 (2)	0.0280 (8)
C65	−0.01491 (18)	0.9056 (4)	0.2538 (2)	0.0344 (10)
C66	0.04783 (18)	0.9076 (4)	0.1971 (2)	0.0332 (9)
H1	0.1563 (17)	1.026 (3)	0.082 (2)	0.015 (7)*
H2	0.21283	0.98661	−0.06686	0.0323*
H3	0.29985	0.75999	0.07855	0.0354*
H5A	0.15739	0.62846	0.10227	0.0418*
H5B	0.10203	0.59509	−0.01127	0.0418*
H6	0.09033	0.87181	−0.04789	0.0339*
H22	0.30815	0.99297	0.21410	0.0370*
H23	0.39884	1.18196	0.28871	0.0423*
H25	0.38369	1.40327	−0.00471	0.0433*
H26	0.28992	1.21520	−0.07828	0.0393*
H31	0.35046	0.65928	−0.07880	0.0408*
H31B	0.43102	0.80007	0.06399	0.0741*
H32A	0.46223	0.80630	−0.05135	0.0741*
H32C	0.41862	0.95777	−0.00966	0.0741*
H33A	0.31687	0.96417	−0.17656	0.0682*
H33B	0.26943	0.80529	−0.21596	0.0682*
H33C	0.36012	0.81951	−0.22723	0.0682*
H62	−0.03336	0.72355	−0.02164	0.0375*
H63	−0.13807	0.70893	0.07638	0.0366*
H65	−0.01101	0.95583	0.32328	0.0412*
H66	0.09544	0.95627	0.22897	0.0399*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl2	0.0381 (5)	0.0589 (6)	0.0684 (6)	−0.0143 (4)	0.0042 (4)	−0.0239 (4)
Cl6	0.0310 (4)	0.0479 (5)	0.0328 (4)	−0.0013 (3)	0.0084 (3)	0.0031 (3)
O4	0.0437 (14)	0.0436 (13)	0.0524 (13)	−0.0041 (11)	0.0118 (11)	−0.0164 (11)
N1	0.0255 (13)	0.0287 (14)	0.0303 (12)	−0.0016 (12)	0.0041 (10)	−0.0022 (10)
C2	0.0235 (14)	0.0358 (16)	0.0211 (12)	0.0003 (12)	0.0019 (11)	0.0041 (11)
C3	0.0286 (16)	0.0381 (16)	0.0215 (13)	0.0016 (13)	0.0030 (11)	0.0015 (11)
C4	0.0314 (17)	0.0336 (16)	0.0342 (15)	0.0032 (14)	0.0004 (13)	0.0043 (13)
C5	0.0301 (16)	0.0331 (16)	0.0412 (16)	−0.0053 (14)	0.0049 (13)	0.0011 (13)
C6	0.0261 (15)	0.0338 (16)	0.0238 (13)	−0.0027 (13)	0.0005 (11)	0.0003 (11)
C21	0.0207 (14)	0.0294 (15)	0.0281 (14)	0.0008 (12)	0.0039 (11)	−0.0003 (11)
C22	0.0313 (16)	0.0363 (16)	0.0251 (14)	0.0032 (14)	0.0049 (12)	0.0009 (12)
C23	0.0308 (16)	0.0447 (18)	0.0282 (14)	0.0069 (15)	−0.0019 (12)	−0.0066 (13)
C24	0.0271 (16)	0.0362 (17)	0.0441 (17)	−0.0032 (14)	0.0051 (13)	−0.0142 (14)
C25	0.0334 (17)	0.0314 (16)	0.0436 (17)	−0.0056 (14)	0.0060 (14)	0.0036 (13)
C26	0.0290 (16)	0.0361 (16)	0.0318 (14)	−0.0014 (13)	0.0004 (12)	0.0055 (12)
C31	0.0302 (17)	0.0389 (17)	0.0339 (15)	0.0008 (14)	0.0078 (13)	−0.0065 (13)
C32	0.0269 (17)	0.063 (2)	0.059 (2)	−0.0004 (17)	0.0086 (15)	−0.0101 (18)
C33	0.050 (2)	0.055 (2)	0.0339 (16)	−0.0043 (18)	0.0159 (15)	−0.0026 (15)
C61	0.0260 (15)	0.0298 (15)	0.0267 (13)	−0.0015 (13)	0.0007 (12)	0.0040 (11)
C62	0.0316 (17)	0.0344 (16)	0.0268 (14)	−0.0032 (14)	0.0018 (12)	−0.0041 (12)
C63	0.0255 (16)	0.0321 (16)	0.0319 (14)	−0.0034 (13)	−0.0021 (12)	−0.0031 (12)
C64	0.0271 (15)	0.0314 (15)	0.0263 (13)	−0.0007 (13)	0.0070 (12)	0.0076 (12)
C65	0.0379 (18)	0.0456 (18)	0.0190 (13)	−0.0073 (15)	0.0023 (12)	−0.0002 (12)
C66	0.0298 (16)	0.0410 (17)	0.0278 (14)	−0.0096 (14)	0.0009 (12)	−0.0012 (12)

Geometric parameters (Å, °)

Cl2—C24	1.738 (3)	C63—C64	1.389 (3)
Cl6—C64	1.745 (3)	C64—C65	1.377 (4)
O4—C4	1.218 (3)	C65—C66	1.378 (4)
N1—C2	1.469 (4)	C2—H2	1.0000
N1—C6	1.457 (4)	C3—H3	1.0000
N1—H1	0.80 (3)	C5—H5A	0.9900
C2—C21	1.505 (4)	C5—H5B	0.9900
C2—C3	1.555 (4)	C6—H6	1.0000
C3—C4	1.518 (4)	C22—H22	0.9500
C3—C31	1.536 (4)	C23—H23	0.9500
C4—C5	1.508 (4)	C25—H25	0.9500
C5—C6	1.531 (4)	C26—H26	0.9500
C6—C61	1.505 (4)	C31—H31	1.0000
C21—C26	1.390 (4)	C32—H31B	0.9800
C21—C22	1.397 (3)	C32—H32A	0.9800
C22—C23	1.378 (4)	C32—H32C	0.9800
C23—C24	1.374 (5)	C33—H33A	0.9800
C24—C25	1.390 (5)	C33—H33B	0.9800

C25—C26	1.395 (4)	C33—H33C	0.9800
C31—C32	1.529 (4)	C62—H62	0.9500
C31—C33	1.535 (4)	C63—H63	0.9500
C61—C66	1.397 (4)	C65—H65	0.9500
C61—C62	1.389 (4)	C66—H66	0.9500
C62—C63	1.383 (4)		
Cl2···C23^{i}	3.341 (3)	H2···H26	2.3500
Cl6···C22^{ii}	3.606 (3)	H2···H33A	2.4200
Cl2···H32A^{iii}	3.0800	H2···Cl6^{vi}	3.1200
Cl2···H33C^{iv}	3.0500	H2···C63^{vi}	2.9800
Cl2···H23^{i}	2.9900	H2···C64^{vi}	3.0500
Cl6···H1^{ii}	2.98 (2)	H3···C22	2.8700
Cl6···H5A^{v}	2.9300	H3···H22	2.5600
Cl6···H2^{vi}	3.1200	H3···H31B	2.3300
Cl6···H26^{vi}	3.1100	H3···C33^{vii}	3.0600
Cl6···H62^{vii}	3.0600	H3···H33C^{vii}	2.5600
O4···C33	3.225 (4)	H5A···Cl6^{ii}	2.9300
O4···H31	2.3900	H5B···C62	2.8700
O4···H33B	2.7200	H5B···H62	2.5600
O4···H63^{viii}	2.7000	H6···H2	2.3700
O4···H66^{ix}	2.7800	H6···H62	2.5400
N1···H22	2.7100	H22···N1	2.7100
N1···H66	2.4900	H22···H3	2.5600
C21···C32	3.199 (4)	H23···Cl2^{x}	2.9900
C22···Cl6^{v}	3.606 (3)	H25···H31^{xi}	2.3700
C22···C32	3.564 (4)	H26···H2	2.3500
C23···Cl2^{x}	3.341 (3)	H26···H33A	2.5100
C26···C32	3.581 (4)	H26···Cl6^{vi}	3.1100
C32···C21	3.199 (4)	H31···O4	2.3900
C32···C22	3.564 (4)	H31···H25^{xii}	2.3700
C32···C26	3.581 (4)	H31B···H3	2.3300
C33···O4	3.225 (4)	H32A···H33C	2.5900
C2···H33A	2.8600	H32A···Cl2^{iii}	3.0800
C4···H33B	2.8700	H32A···C24^{iii}	2.9600
C5···H62	3.0500	H32A···C25^{iii}	3.0900
C21···H32C	2.6600	H32C···C21	2.6600
C22···H3	2.8700	H32C···C22	3.1000
C22···H32C	3.1000	H32C···C26	2.7700
C22···H1	2.97 (3)	H32C···H33A	2.5000
C24···H32A^{iii}	2.9600	H33A···C2	2.8600
C25···H32A^{iii}	3.0900	H33A···C26	2.9500
C26···H32C	2.7700	H33A···H2	2.4200
C26···H33A	2.9500	H33A···H26	2.5100
C33···H2	2.7600	H33A···H32C	2.5000

C33···H3^{ix}	3.0600	H33B···O4	2.7200
C62···H5B	2.8700	H33B···C4	2.8700
C62···H65^{ii}	3.0700	H33C···H32A	2.5900
C62···H1^{vi}	3.08 (3)	H33C···Cl2^{xiii}	3.0500
C63···H1^{vi}	3.02 (3)	H33C···H3^{ix}	2.5600
C63···H2^{vi}	2.9800	H62···C5	3.0500
C64···H2^{vi}	3.0500	H62···H5B	2.5600
C65···H62^{vii}	3.0400	H62···H6	2.5400
C66···H1	2.71 (3)	H62···Cl6^{ix}	3.0600
H1···C22	2.97 (3)	H62···C65^{ix}	3.0400
H1···C66	2.71 (3)	H62···H65^{ix}	2.5100
H1···H66	2.3100	H63···O4^{viii}	2.7000
H1···Cl6^{v}	2.98 (2)	H65···C62^{v}	3.0700
H1···C62^{vi}	3.08 (3)	H65···H62^{vii}	2.5100
H1···C63^{vi}	3.02 (3)	H66···N1	2.4900
H2···C33	2.7600	H66···H1	2.3100
H2···H6	2.3700	H66···O4^{vii}	2.7800
C2—N1—C6	113.6 (2)	C2—C3—H3	106.00
C6—N1—H1	108 (2)	C4—C3—H3	106.00
C2—N1—H1	107 (2)	C31—C3—H3	106.00
N1—C2—C21	107.9 (2)	C4—C5—H5A	109.00
N1—C2—C3	109.2 (2)	C4—C5—H5B	109.00
C3—C2—C21	112.8 (2)	C6—C5—H5A	109.00
C2—C3—C31	117.1 (2)	C6—C5—H5B	109.00
C2—C3—C4	109.2 (2)	H5A—C5—H5B	108.00
C4—C3—C31	111.4 (2)	N1—C6—H6	109.00
C3—C4—C5	116.4 (2)	C5—C6—H6	109.00
O4—C4—C3	122.7 (3)	C61—C6—H6	109.00
O4—C4—C5	120.9 (3)	C21—C22—H22	120.00
C4—C5—C6	111.9 (2)	C23—C22—H22	119.00
N1—C6—C5	106.6 (2)	C22—C23—H23	120.00
N1—C6—C61	112.3 (2)	C24—C23—H23	120.00
C5—C6—C61	110.3 (2)	C24—C25—H25	121.00
C2—C21—C22	120.9 (2)	C26—C25—H25	121.00
C22—C21—C26	117.8 (2)	C21—C26—H26	119.00
C2—C21—C26	121.3 (2)	C25—C26—H26	119.00
C21—C22—C23	121.0 (2)	C3—C31—H31	106.00
C22—C23—C24	120.1 (3)	C32—C31—H31	106.00
C23—C24—C25	120.9 (3)	C33—C31—H31	106.00
Cl2—C24—C23	120.2 (3)	C31—C32—H31B	109.00
Cl2—C24—C25	118.9 (3)	C31—C32—H32A	110.00
C24—C25—C26	118.2 (3)	C31—C32—H32C	109.00
C21—C26—C25	121.9 (3)	H31B—C32—H32A	109.00
C3—C31—C33	113.1 (2)	H31B—C32—H32C	109.00
C32—C31—C33	111.2 (3)	H32A—C32—H32C	109.00
C3—C31—C32	114.1 (2)	C31—C33—H33A	109.00

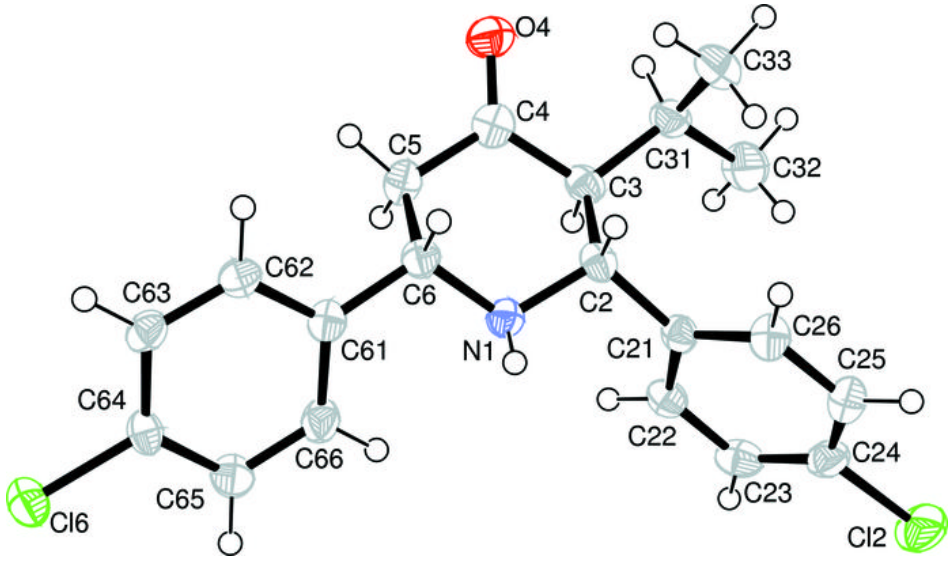
C62—C61—C66	117.9 (3)	C31—C33—H33B	109.00
C6—C61—C62	119.3 (2)	C31—C33—H33C	109.00
C6—C61—C66	122.9 (3)	H33A—C33—H33B	110.00
C61—C62—C63	122.1 (2)	H33A—C33—H33C	109.00
C62—C63—C64	118.3 (3)	H33B—C33—H33C	110.00
Cl6—C64—C65	120.3 (2)	C61—C62—H62	119.00
Cl6—C64—C63	118.8 (2)	C63—C62—H62	119.00
C63—C64—C65	120.9 (3)	C62—C63—H63	121.00
C64—C65—C66	119.9 (2)	C64—C63—H63	121.00
C61—C66—C65	120.8 (3)	C64—C65—H65	120.00
N1—C2—H2	109.00	C66—C65—H65	120.00
C3—C2—H2	109.00	C61—C66—H66	120.00
C21—C2—H2	109.00	C65—C66—H66	120.00
C6—N1—C2—C3	−65.3 (3)	N1—C6—C61—C62	169.8 (2)
C6—N1—C2—C21	171.8 (2)	N1—C6—C61—C66	−11.4 (4)
C2—N1—C6—C5	65.5 (3)	C5—C6—C61—C62	−71.5 (3)
C2—N1—C6—C61	−173.6 (2)	C5—C6—C61—C66	107.4 (3)
N1—C2—C3—C4	51.0 (3)	C2—C21—C22—C23	177.2 (3)
N1—C2—C3—C31	178.7 (2)	C26—C21—C22—C23	−3.4 (4)
C21—C2—C3—C4	171.0 (2)	C2—C21—C26—C25	−176.3 (3)
C21—C2—C3—C31	−61.3 (3)	C22—C21—C26—C25	4.2 (4)
N1—C2—C21—C22	51.9 (3)	C21—C22—C23—C24	−0.1 (5)
N1—C2—C21—C26	−127.5 (3)	C22—C23—C24—Cl2	−175.4 (2)
C3—C2—C21—C22	−68.8 (3)	C22—C23—C24—C25	2.9 (5)
C3—C2—C21—C26	111.8 (3)	Cl2—C24—C25—C26	176.2 (2)
C2—C3—C4—O4	135.7 (3)	C23—C24—C25—C26	−2.1 (5)
C2—C3—C4—C5	−45.4 (3)	C24—C25—C26—C21	−1.6 (5)
C31—C3—C4—O4	4.8 (4)	C6—C61—C62—C63	177.2 (2)
C31—C3—C4—C5	−176.3 (2)	C66—C61—C62—C63	−1.7 (4)
C2—C3—C31—C32	78.8 (3)	C6—C61—C66—C65	−179.2 (3)
C2—C3—C31—C33	−49.6 (3)	C62—C61—C66—C65	−0.4 (4)
C4—C3—C31—C32	−154.5 (3)	C61—C62—C63—C64	1.9 (4)
C4—C3—C31—C33	77.1 (3)	C62—C63—C64—Cl6	180.0 (2)
O4—C4—C5—C6	−133.0 (3)	C62—C63—C64—C65	0.0 (4)
C3—C4—C5—C6	48.2 (3)	Cl6—C64—C65—C66	178.0 (2)
C4—C5—C6—N1	−54.1 (3)	C63—C64—C65—C66	−2.0 (4)
C4—C5—C6—C61	−176.3 (2)	C64—C65—C66—C61	2.2 (5)

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) −*x*, *y*−1/2, −*z*+1/2; (iii) −*x*+1, −*y*+2, −*z*; (iv) *x*, −*y*+5/2, *z*+1/2; (v) −*x*, *y*+1/2, −*z*+1/2; (vi) −*x*, −*y*+2, −*z*; (vii) *x*, −*y*+3/2, *z*+1/2; (viii) −*x*, −*y*+1, −*z*; (ix) *x*, −*y*+3/2, *z*−1/2; (x) −*x*+1, *y*−1/2, −*z*+1/2; (xi) *x*, *y*+1, *z*; (xii) *x*, *y*−1, *z*; (xiii) *x*, −*y*+5/2, *z*−1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C31—H31···O4	1.00	2.39	2.769 (4)	102
C66—H66···N1	0.95	2.49	2.832 (4)	101

Fig. 1


O4
C33
C4
C5
C31
C32
C3
C62
C63
C6
C2
C26
C61
N1
C21
C25
C64
C22
C66
C24
C65
C23
Cl6
Cl2