Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6-(4-Chlorophenyl)-9,9-dimethyl-3,7-dioxo-2,3,4,6,7,8,9,10-octahydro-1*H*-pyrimido[1,2-*a*]quinoline-5-carbonitrile

Dong-Qin Chen,[a] Chun-Mei Li[b] and Shu-Jiang Tu[b]*

[a]Xuzhou Medical College, Department of Chemistry, Xuzhou 221006, People's Republic of China, and [b]School of Chemistry and Chemical Engineering, Xuzhou Normal University, Xuzhou 221116, People's Republic of China
Correspondence e-mail: laotu2001@263.net

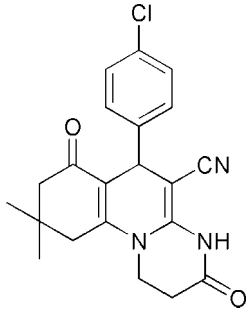
Received 27 August 2007; accepted 29 August 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.008 Å; R factor = 0.064; wR factor = 0.223; data-to-parameter ratio = 13.7.

The title compound, $C_{21}H_{20}ClN_3O_2$, was synthesized by the reaction of 4-chlorobenzaldehyde and 3-(5,5-dimethyl-3-oxocyclohex-1-enylamino)propanoic acid with malononitrile in ethylene glycol under microwave irradiation. The dihydropyridine ring has a boat conformation. The pyrimidine ring adopts a screw-boat conformation and the cyclohexene ring is in an envelope conformation. In the crystalline state, centrosymmetrically related molecules form dimeric pairs through N—H···N hydrogen bonding. Weak C—H···O hydrogen bonds are also observed.

Related literature

For general background, see: Awouters *et al.* (1986); Matsutani & Mizushima (1989); Smith *et al.* (1995); Yanagihara *et al.* (1988).



Experimental

Crystal data

$C_{21}H_{20}ClN_3O_2$
M_r = 381.85
Monoclinic, $P2_1/c$
a = 10.378 (5) Å
b = 10.131 (5) Å
c = 18.143 (9) Å
β = 97.406 (8)°
V = 1891.6 (17) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 298 (2) K
0.25 × 0.20 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.946, T_{max} = 0.969
9514 measured reflections
3340 independent reflections
1323 reflections with $I > 2\sigma(I)$
R_{int} = 0.086

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.223
S = 1.02
3340 reflections
244 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···N3^{i}	0.86	2.24	3.059 (7)	159
C12—H12B···O2^{ii}	0.97	2.29	3.005 (8)	130
C16—H16···O1^{iii}	0.93	2.57	3.480 (7)	167

Symmetry codes: (i) $-x+1, -y+2, -z+2$; (ii) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x, -y+1, -z+2$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

The authors thank the National Science Foundation of China (grant No. 20672090), the Natural Science Foundation of Jiangsu Province (grant No. BK 2006033) and the Six Kinds of Professional Elite Foundation of Jiangsu Province (grant No. 06-A-039) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2454).

References

Awouters, F., Vermeire, J., Smeyers, F., Vermote, P., Van Beek, R. & Niemegeers, C. J. E. (1986). *Drug Dev. Res.* **8**, 95–102.
Bruker (1997). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Matsutani, S. & Mizushima, Y. (1989). Eur. Patent Appl. EP 89-102635 19890216.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Smith, R. L., Barrent, R. J. & Sanders-Bush, E. (1995). *J. Pharmacol. Exp. Ther.* **275**, 1050–1057.
Yanagihara, Y., Kasai, H., Kawashima, T. & Shida, T. (1988). *Jpn. J. Pharmacol.* **48**, 91–101.

 doi:10.1107/S1600536807042389

supplementary materials

Acta Cryst. (2007). E**63**, o3931 [**doi:10.1107/S1600536807042389**]

6-(4-Chlorophenyl)-9,9-dimethyl-3,7-dioxo-2,3,4,6,7,8,9,10-octahydro-1*H*-pyrimido[1,2-*a*]quinoline-5-carbonitrile

D.-Q. Chen, C.-M. Li and S.-J. Tu

Comment

Pyrido[1,2-*a*]pyrimidine core has been successfully used a motif for the development of biologically interesting molecules, including pirenperone, a tranquilizer (Smith *et al.*, 1995), barmastine, an antiallergic agent (Awouters *et al.*, 1986), an antiulcerative agent (Matsutani *et al.*, 1989), and pemirolast, an antiasthmatic agent (Yanagihara *et al.*, 1988). Pyrimido[1,2-*a*]quinoline belongs to a class of compounds which are special not only because of their interesting chemical and physical properties, but also due to their immense utility in the pharmaceutical industries. We report here the crystal structure of the title compound, a pyrimido[1,2-*a*]quinoline derivative.

In the title molecule, the dihydropyridine ring is planar, with a maximum deviation of 0.039 (4) Å for atom C4 (Fig. 1). The pyrimidine ring adopts a screw-boat conformation, with atoms C11 and C10 deviating from the C12/N1/C1/N2 plane by 0.876 (10) and 0.451 (10) Å (Fig. 1). The cyclohexene ring adopts an envelope conformation, with atom C7 deviating by 0.620 (8) Å from the C4/C5/C6/C8/C9 plane. The C1—C4/C9/N1 plane forms dihedral angles 0.5 (4)° and 8.8 (3)°, respectively, with the C12/N1/C1/N2 and C4—C6/C8/C9 planes. The dihedral angle between the dihydropyridine and chlorophenyl rings is 88.6 (2)°.

In the crystalline state, centrosymmetrically related molecules form dimeric pairs through N—H···O hydrogen bonding. In addition, weak C—H···O hydrogen bonds are observed (Table 1).

Experimental

The title compound was prepared by the reaction of 4-chlorobenzaldehyde (0.141 g, 1 mmol), 3-(5,5-dimethyl-3-oxocyclohex-1-enylamino)propanoic acid (0.211 g, 1 mmol) with malononitrile (0.066 g, 1 mmol) in ethylene glycol (2.0 ml) at 393 K under microwave irradiation (maximum power 200 W, initial power 100 W) for 6 min (yield: 0.324 g, 85%; m.p. 539–541 K). Single crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution (95%).

Refinement

H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C). Owing to the large number of weak high-angle reflections, the ratio of observed to unique reflections is low (40%).

Figures

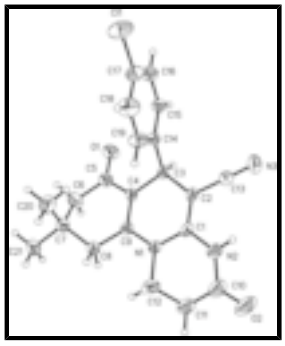


Fig. 1. The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

6-(4-Chlorophenyl)-9,9-dimethyl-3,7-dioxo-2,3,4,6,7,8,9,10-octahydro- 1*H*-pyrimido[1,2-*a*]quinoline-5-carbonitrile

Crystal data

$C_{21}H_{20}ClN_3O_2$	F_{000} = 800
M_r = 381.85	D_x = 1.341 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 539-541 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 10.378 (5) Å	Cell parameters from 981 reflections
b = 10.131 (5) Å	θ = 2.8–25.1º
c = 18.143 (9) Å	μ = 0.22 mm^{-1}
β = 97.406 (8)º	T = 298 (2) K
V = 1891.6 (17) Å^3	Block, colourless
Z = 4	0.25 × 0.20 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer	3340 independent reflections
Radiation source: fine-focus sealed tube	1323 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.086
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −12→12
T_{min} = 0.946, T_{max} = 0.969	k = −12→11
9514 measured reflections	l = −11→21

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.064$	H-atom parameters constrained
$wR(F^2) = 0.223$	$w = 1/[\sigma^2(F_o^2) + (0.035P)^2 + 2.4335P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} < 0.001$
3340 reflections	$\Delta\rho_{max} = 0.24$ e Å^{-3}
244 parameters	$\Delta\rho_{min} = -0.28$ 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}
Cl1	0.3493 (2)	0.23717 (18)	1.02619 (13)	0.1007 (8)
N1	0.2832 (4)	0.8830 (4)	0.7610 (2)	0.0424 (11)
N2	0.4295 (4)	1.0056 (5)	0.8419 (3)	0.0555 (13)
H2	0.4812	1.0035	0.8829	0.067*
N3	0.3718 (5)	0.9263 (5)	1.0240 (3)	0.0665 (15)
O1	−0.0473 (4)	0.6160 (4)	0.8355 (2)	0.0685 (13)
O2	0.5347 (6)	1.1839 (5)	0.8066 (3)	0.124 (2)
C1	0.3300 (5)	0.9138 (5)	0.8329 (3)	0.0413 (13)
C2	0.2840 (5)	0.8574 (5)	0.8912 (3)	0.0404 (13)
C3	0.1777 (5)	0.7546 (5)	0.8833 (3)	0.0389 (13)
H3	0.1056	0.7882	0.9079	0.047*
C4	0.1294 (5)	0.7358 (5)	0.8030 (3)	0.0377 (13)
C5	0.0121 (6)	0.6585 (6)	0.7855 (3)	0.0499 (15)
C6	−0.0426 (6)	0.6372 (7)	0.7057 (3)	0.0667 (19)
H6A	−0.0897	0.5543	0.7015	0.080*
H6B	−0.1040	0.7072	0.6904	0.080*
C7	0.0588 (6)	0.6346 (6)	0.6547 (3)	0.0593 (17)
C8	0.1424 (5)	0.7582 (5)	0.6664 (3)	0.0468 (14)
H8A	0.2194	0.7464	0.6419	0.056*
H8B	0.0942	0.8323	0.6429	0.056*
C9	0.1836 (5)	0.7907 (5)	0.7464 (3)	0.0390 (13)
C10	0.4522 (7)	1.0985 (7)	0.7915 (4)	0.073 (2)
C11	0.3695 (6)	1.0920 (6)	0.7190 (4)	0.0691 (19)
H11A	0.4141	1.1322	0.6809	0.083*

H11B	0.2894	1.1402	0.7215	0.083*
C12	0.3395 (6)	0.9501 (6)	0.6998 (3)	0.0619 (18)
H12A	0.2786	0.9456	0.6546	0.074*
H12B	0.4186	0.9052	0.6909	0.074*
C13	0.3332 (6)	0.8955 (5)	0.9641 (4)	0.0499 (15)
C14	0.2223 (5)	0.6256 (5)	0.9202 (3)	0.0382 (13)
C15	0.1652 (5)	0.5741 (5)	0.9779 (3)	0.0491 (15)
H15	0.0986	0.6211	0.9956	0.059*
C16	0.2031 (6)	0.4553 (6)	1.0105 (3)	0.0565 (16)
H16	0.1608	0.4214	1.0485	0.068*
C17	0.3035 (6)	0.3881 (6)	0.9865 (4)	0.0597 (17)
C18	0.3645 (7)	0.4362 (7)	0.9297 (4)	0.078 (2)
H18	0.4328	0.3898	0.9134	0.094*
C19	0.3238 (6)	0.5540 (6)	0.8970 (4)	0.0638 (18)
H19	0.3652	0.5866	0.8583	0.077*
C20	0.1454 (7)	0.5117 (6)	0.6716 (4)	0.085 (2)
H20A	0.2109	0.5099	0.6388	0.128*
H20B	0.0930	0.4336	0.6643	0.128*
H20C	0.1861	0.5152	0.7221	0.128*
C21	−0.0004 (7)	0.6253 (7)	0.5739 (3)	0.080 (2)
H21A	0.0676	0.6237	0.5428	0.120*
H21B	−0.0553	0.7005	0.5614	0.120*
H21C	−0.0510	0.5460	0.5665	0.120*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1119 (16)	0.0658 (12)	0.1244 (19)	0.0265 (11)	0.0145 (14)	0.0319 (12)
N1	0.043 (3)	0.041 (3)	0.045 (3)	−0.012 (2)	0.015 (2)	−0.003 (2)
N2	0.048 (3)	0.054 (3)	0.060 (3)	−0.016 (2)	−0.010 (3)	0.014 (3)
N3	0.066 (4)	0.073 (4)	0.058 (4)	−0.009 (3)	−0.003 (3)	−0.016 (3)
O1	0.060 (3)	0.094 (3)	0.054 (3)	−0.032 (2)	0.017 (2)	0.004 (2)
O2	0.122 (5)	0.110 (4)	0.127 (5)	−0.079 (4)	−0.029 (4)	0.038 (4)
C1	0.039 (3)	0.032 (3)	0.052 (4)	−0.007 (2)	0.003 (3)	0.000 (3)
C2	0.040 (3)	0.036 (3)	0.043 (3)	−0.007 (2)	−0.003 (3)	−0.006 (3)
C3	0.038 (3)	0.038 (3)	0.042 (3)	−0.010 (2)	0.012 (3)	−0.006 (3)
C4	0.037 (3)	0.042 (3)	0.035 (3)	−0.009 (2)	0.004 (3)	−0.008 (3)
C5	0.048 (4)	0.059 (4)	0.043 (4)	−0.010 (3)	0.008 (3)	0.000 (3)
C6	0.060 (4)	0.090 (5)	0.049 (4)	−0.029 (4)	0.005 (4)	−0.002 (4)
C7	0.074 (4)	0.061 (4)	0.043 (4)	−0.027 (3)	0.006 (3)	−0.009 (3)
C8	0.050 (3)	0.050 (3)	0.042 (4)	−0.009 (3)	0.011 (3)	−0.005 (3)
C9	0.037 (3)	0.038 (3)	0.044 (3)	−0.005 (2)	0.010 (3)	−0.003 (3)
C10	0.063 (5)	0.066 (5)	0.086 (5)	−0.020 (4)	−0.003 (4)	0.008 (4)
C11	0.061 (4)	0.069 (5)	0.078 (5)	−0.018 (3)	0.009 (4)	0.012 (4)
C12	0.063 (4)	0.064 (4)	0.061 (4)	−0.014 (3)	0.017 (4)	0.012 (3)
C13	0.053 (4)	0.045 (4)	0.051 (4)	−0.003 (3)	0.006 (3)	0.001 (3)
C14	0.040 (3)	0.038 (3)	0.038 (3)	−0.004 (2)	0.011 (3)	−0.004 (3)
C15	0.060 (4)	0.046 (3)	0.045 (3)	0.003 (3)	0.019 (3)	0.007 (3)

C16	0.057 (4)	0.061 (4)	0.054 (4)	−0.003 (3)	0.018 (3)	0.006 (3)
C17	0.062 (4)	0.049 (4)	0.068 (4)	0.010 (3)	0.005 (4)	0.010 (3)
C18	0.072 (5)	0.079 (5)	0.090 (5)	0.020 (4)	0.034 (4)	0.010 (4)
C19	0.061 (4)	0.064 (4)	0.071 (5)	0.007 (3)	0.028 (4)	0.019 (4)
C20	0.113 (6)	0.062 (5)	0.086 (6)	−0.013 (4)	0.034 (5)	−0.013 (4)
C21	0.108 (6)	0.087 (5)	0.045 (4)	−0.044 (4)	0.006 (4)	−0.015 (4)

Geometric parameters (Å, °)

Cl1—C17	1.731 (6)	C8—C9	1.496 (7)
N1—C1	1.368 (7)	C8—H8A	0.97
N1—C9	1.394 (6)	C8—H8B	0.97
N1—C12	1.485 (6)	C10—C11	1.476 (9)
N2—C10	1.354 (7)	C11—C12	1.503 (8)
N2—C1	1.384 (6)	C11—H11A	0.97
N2—H2	0.86	C11—H11B	0.97
N3—C13	1.152 (7)	C12—H12A	0.97
O1—C5	1.239 (6)	C12—H12B	0.97
O2—C10	1.224 (7)	C14—C15	1.371 (7)
C1—C2	1.343 (7)	C14—C19	1.388 (7)
C2—C13	1.408 (8)	C15—C16	1.376 (7)
C2—C3	1.511 (6)	C15—H15	0.93
C3—C4	1.492 (7)	C16—C17	1.363 (7)
C3—C14	1.513 (7)	C16—H16	0.93
C3—H3	0.98	C17—C18	1.366 (8)
C4—C9	1.351 (6)	C18—C19	1.376 (8)
C4—C5	1.448 (7)	C18—H18	0.93
C5—C6	1.500 (8)	C19—H19	0.93
C6—C7	1.489 (8)	C20—H20A	0.96
C6—H6A	0.97	C20—H20B	0.96
C6—H6B	0.97	C20—H20C	0.96
C7—C21	1.517 (8)	C21—H21A	0.96
C7—C8	1.523 (7)	C21—H21B	0.96
C7—C20	1.543 (9)	C21—H21C	0.96
C1—N1—C9	119.8 (4)	O2—C10—C11	122.7 (6)
C1—N1—C12	118.9 (4)	N2—C10—C11	116.1 (6)
C9—N1—C12	121.3 (5)	C10—C11—C12	109.1 (6)
C10—N2—C1	125.4 (5)	C10—C11—H11A	109.9
C10—N2—H2	117.3	C12—C11—H11A	109.9
C1—N2—H2	117.3	C10—C11—H11B	109.9
C2—C1—N1	122.4 (5)	C12—C11—H11B	109.9
C2—C1—N2	121.9 (5)	H11A—C11—H11B	108.3
N1—C1—N2	115.7 (5)	N1—C12—C11	110.7 (5)
C1—C2—C13	120.0 (5)	N1—C12—H12A	109.5
C1—C2—C3	123.2 (5)	C11—C12—H12A	109.5
C13—C2—C3	116.8 (5)	N1—C12—H12B	109.5
C4—C3—C2	109.3 (4)	C11—C12—H12B	109.5
C4—C3—C14	111.5 (4)	H12A—C12—H12B	108.1
C2—C3—C14	112.0 (4)	N3—C13—C2	179.1 (7)

C4—C3—H3	107.9	C15—C14—C19	116.9 (5)
C2—C3—H3	107.9	C15—C14—C3	121.9 (5)
C14—C3—H3	107.9	C19—C14—C3	121.2 (5)
C9—C4—C5	118.5 (5)	C14—C15—C16	122.2 (5)
C9—C4—C3	124.7 (4)	C14—C15—H15	118.9
C5—C4—C3	116.7 (5)	C16—C15—H15	118.9
O1—C5—C4	120.8 (5)	C17—C16—C15	119.2 (6)
O1—C5—C6	119.7 (5)	C17—C16—H16	120.4
C4—C5—C6	119.4 (5)	C15—C16—H16	120.4
C7—C6—C5	113.1 (5)	C16—C17—C18	120.7 (6)
C7—C6—H6A	109.0	C16—C17—Cl1	119.2 (5)
C5—C6—H6A	109.0	C18—C17—Cl1	120.0 (5)
C7—C6—H6B	109.0	C17—C18—C19	119.2 (6)
C5—C6—H6B	109.0	C17—C18—H18	120.4
H6A—C6—H6B	107.8	C19—C18—H18	120.4
C6—C7—C21	111.8 (6)	C18—C19—C14	121.7 (6)
C6—C7—C8	109.5 (5)	C18—C19—H19	119.1
C21—C7—C8	109.8 (5)	C14—C19—H19	119.1
C6—C7—C20	109.3 (5)	C7—C20—H20A	109.5
C21—C7—C20	107.1 (5)	C7—C20—H20B	109.5
C8—C7—C20	109.2 (5)	H20A—C20—H20B	109.5
C9—C8—C7	113.7 (4)	C7—C20—H20C	109.5
C9—C8—H8A	108.8	H20A—C20—H20C	109.5
C7—C8—H8A	108.8	H20B—C20—H20C	109.5
C9—C8—H8B	108.8	C7—C21—H21A	109.5
C7—C8—H8B	108.8	C7—C21—H21B	109.5
H8A—C8—H8B	107.7	H21A—C21—H21B	109.5
C4—C9—N1	120.2 (5)	C7—C21—H21C	109.5
C4—C9—C8	123.4 (5)	H21A—C21—H21C	109.5
N1—C9—C8	116.4 (4)	H21B—C21—H21C	109.5
O2—C10—N2	121.2 (7)		
C9—N1—C1—C2	0.5 (8)	C3—C4—C9—N1	−7.2 (8)
C12—N1—C1—C2	−180.0 (5)	C5—C4—C9—C8	−10.5 (8)
C9—N1—C1—N2	179.5 (4)	C3—C4—C9—C8	172.9 (5)
C12—N1—C1—N2	−1.0 (7)	C1—N1—C9—C4	2.8 (7)
C10—N2—C1—C2	−156.1 (6)	C12—N1—C9—C4	−176.7 (5)
C10—N2—C1—N1	25.0 (8)	C1—N1—C9—C8	−177.3 (5)
N1—C1—C2—C13	−178.2 (5)	C12—N1—C9—C8	3.2 (7)
N2—C1—C2—C13	2.9 (8)	C7—C8—C9—C4	−14.4 (8)
N1—C1—C2—C3	0.4 (8)	C7—C8—C9—N1	165.7 (5)
N2—C1—C2—C3	−178.5 (5)	C1—N2—C10—O2	172.9 (6)
C1—C2—C3—C4	−3.9 (7)	C1—N2—C10—C11	−5.4 (9)
C13—C2—C3—C4	174.8 (5)	O2—C10—C11—C12	147.4 (7)
C1—C2—C3—C14	120.3 (5)	N2—C10—C11—C12	−34.3 (8)
C13—C2—C3—C14	−61.1 (6)	C1—N1—C12—C11	−37.7 (7)
C2—C3—C4—C9	7.3 (7)	C9—N1—C12—C11	141.8 (5)
C14—C3—C4—C9	−117.1 (5)	C10—C11—C12—N1	53.8 (7)
C2—C3—C4—C5	−169.4 (4)	C4—C3—C14—C15	−118.8 (5)
C14—C3—C4—C5	66.2 (6)	C2—C3—C14—C15	118.4 (5)

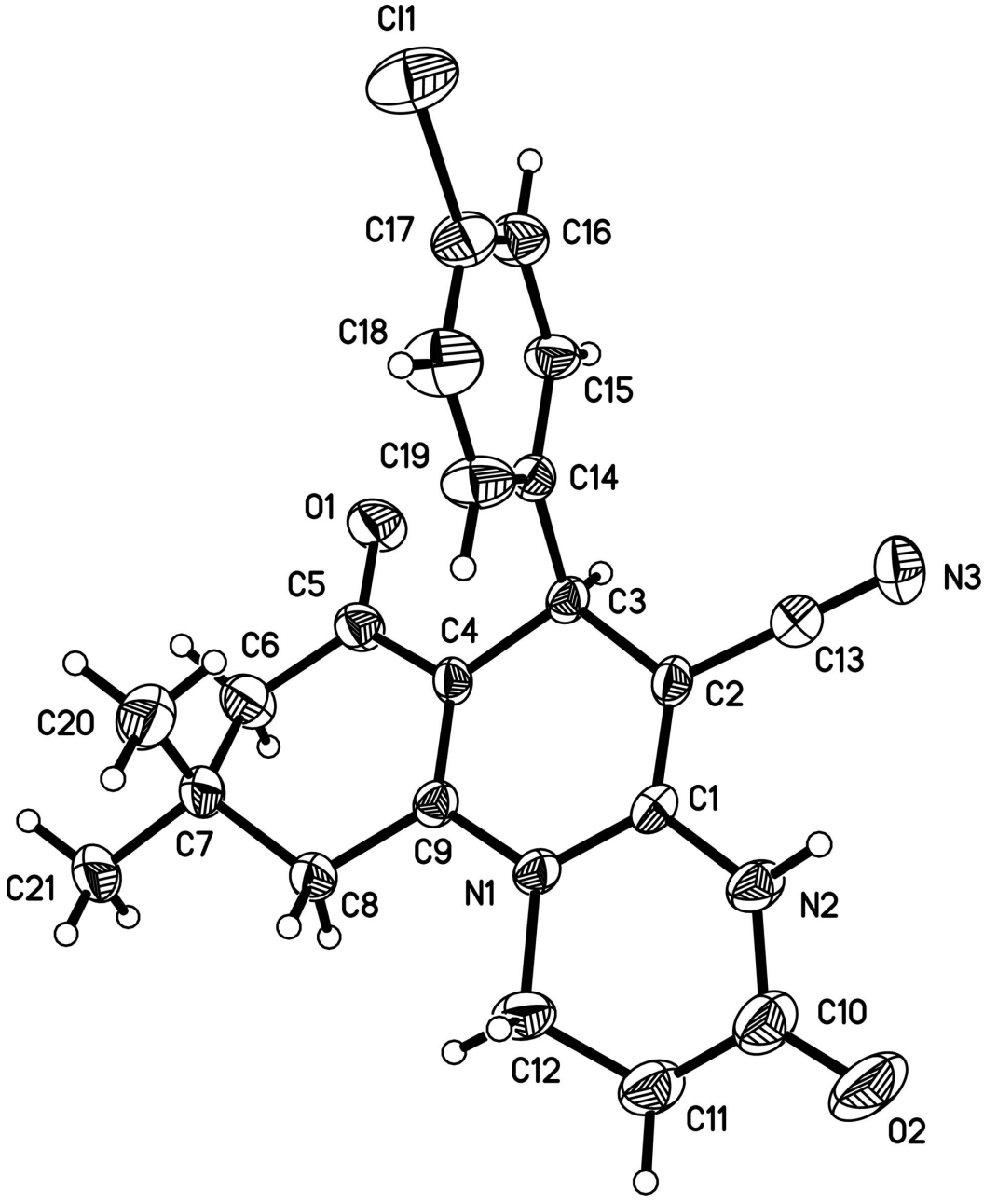
C9—C4—C5—O1	−173.6 (5)	C4—C3—C14—C19	61.4 (6)
C3—C4—C5—O1	3.3 (8)	C2—C3—C14—C19	−61.5 (7)
C9—C4—C5—C6	2.4 (8)	C19—C14—C15—C16	−1.8 (8)
C3—C4—C5—C6	179.3 (5)	C3—C14—C15—C16	178.4 (5)
O1—C5—C6—C7	−153.3 (6)	C14—C15—C16—C17	2.1 (9)
C4—C5—C6—C7	30.7 (8)	C15—C16—C17—C18	−1.3 (10)
C5—C6—C7—C21	−174.9 (5)	C15—C16—C17—Cl1	−178.8 (5)
C5—C6—C7—C8	−53.0 (7)	C16—C17—C18—C19	0.2 (11)
C5—C6—C7—C20	66.7 (7)	Cl1—C17—C18—C19	177.6 (5)
C6—C7—C8—C9	45.4 (7)	C17—C18—C19—C14	0.1 (11)
C21—C7—C8—C9	168.5 (5)	C15—C14—C19—C18	0.6 (9)
C20—C7—C8—C9	−74.3 (6)	C3—C14—C19—C18	−179.5 (6)
C5—C4—C9—N1	169.4 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···N3^{i}	0.86	2.24	3.059 (7)	159
C12—H12B···O2^{ii}	0.97	2.29	3.005 (8)	130
C16—H16···O1^{iii}	0.93	2.57	3.480 (7)	167

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+2; (ii) −*x*+1, *y*−1/2, −*z*+3/2; (iii) −*x*, −*y*+1, −*z*+2.

Fig. 1


Cl1
C17
C16
C18
C15
C19
C14
O1
C5
C3
N3
C4
C6
C13
C2
C20
C1
C7
C9
C21
C8
N1
N2
C10
C12
C11
O2