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8-(4-Chlorophenyl)-11,11-dimethyl-5,9-dioxo-6,8,9,10,11,12-hexahydro-5*H*-quinolino[1,2-*a*]quinazoline-7-carbonitrile *N*,*N*-dimethylformamide solvate

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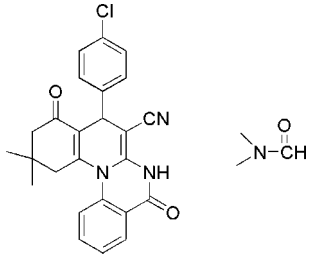
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.074; wR factor = 0.216; data-to-parameter ratio = 13.3.

The title compound, $C_{25}H_{20}ClN_3O_2\cdot C_3H_7NO$, was synthesized by the reaction of 4-chlorobenzaldehyde, 2-(5,5-dimethyl-3-oxocyclohex-1-enylamino)benzoic acid and malononitrile in ethylene glycol under microwave irradiation. The dihydropyridine and pyrimidine rings both adopt boat conformations. The cyclohexene ring adopts an envelope conformation. In the crystal structure, the main molecules exist as N—H···O hydrogen-bonded dimers. The crystal packing is further stabilized by C—H···O and C—H···π interactions. The *N*,*N*-dimethylformamide molecules lie within a channel formed by the quinolino[1,2-*a*]quinazoline molecules.

Related literature

For related literature, see: Colpaert (2003); Doria *et al.* (1983); Hermecz *et al.* (1996); Hermecz & Meszaros (1988); Knoll *et al.* (1987); Kozlovskaya *et al.* (1995).



Experimental

Crystal data

$C_{25}H_{20}ClN_3O_2\cdot C_3H_7NO$
M_r = 502.99
Monoclinic, $C2/c$
a = 36.340 (15) Å
b = 11.780 (5) Å
c = 11.870 (5) Å
β = 102.714 (5)°
V = 4957 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 298 (2) K
0.23 × 0.14 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.957, T_{max} = 0.981
12316 measured reflections
4326 independent reflections
2288 reflections with $I > 2\sigma(I)$
R_{int} = 0.063

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.216
S = 1.02
4326 reflections
325 parameters
60 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2^{i}	0.86	2.04	2.849 (5)	157
C20—H20···O1^{ii}	0.93	2.38	3.269 (6)	160
C13—H13···$Cg1^{iii}$	0.93	2.84	3.530 (5)	132
C26—H26···$Cg2^{iv}$	0.93	2.96	3.720 (10)	140

Symmetry codes: (i) $-x, y, -z+\frac{1}{2}$; (ii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+\frac{1}{2}, y+\frac{3}{2}, -z+\frac{1}{2}$; (iv) $x, y-1, z$. $Cg1$ and $Cg2$ are the cenroids of atoms C11–C16 and C18–C23, respectively.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); 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, 1999); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2492).

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supplementary materials

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8-(4-Chlorophenyl)-11,11-dimethyl-5,9-dioxo-6,8,9,10,11,12-hexahydro-5*H*-quinolino[1,2-*a*]quinazoline-7-carbonitrile *N*,*N*-dimethylformamide solvate

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Comment

The pyrido[1,2-*a*]pyrimidine core has been a successful motif for the development of biologically interesting molecules. Compounds containing the pyrido[1,2-*a*]pyrimidine ring system have been used as analgesics (Hermecz & Meszaros, 1988), antiallergics (Doria *et al.*, 1983), antiasthmatics, antipsychotics (Colpaert, 2003), gastrointestinal protective (Knoll *et al.*, 1987), neurotropic and stress-protecting agents (Kozlovskaya *et al.*, 1995). Moreover, some examples are key intermediates for the synthesis of rutaecarpine alkaloids and several are neutral hydrogen chloride acceptors in organic synthesis (Hermecz *et al.*, 1996). The discovery of quinolino[1,2-*a*]quinazoline including imidazo[1,2-*a*]pyridine moiety as new potential pharmacological molecules may be of great significance. We report herein the crystal structure of the title compound.

The asymmetric unit of the title compound (Fig. 1) contains one $C_{25}H_{20}ClN_3O_2$ molecule and one C_3H_7NO molecule. In the main molecule, the dihydropyridine ring adopts a boat conformation, with the atoms C3 and N1 deviating from the C1/C2/C9/C4 plane by 0.356 (6) and 0.272 (5) Å, respectively. The pyrimidine ring adopts a flattened boat conformation, with the atoms C10 and N1 deviating from the C1/N2/C12/C11 plane by 0.084 (6) and 0.223 (5) Å, respectively. The cyclohexene ring adopts an envelop conformation, with the atom C7 deviating from the C4/C5/C6/C8/C9 plane by 0.709 (6) Å. The dihedral angle between the C1/C2/C9/C4 and C18—C23 planes is 85.2 (2)°, and that between the C1/N2/C12/C11 and C11—C16 planes is 9.5 (1)°.

In the crystal structure, the main molecules exist as N—H···O hydrogen-bonded dimers. The crystal packing is further stabilized by C—H···O and C—H···π interactions (Table 1). The *N*,*N*-dimethylformamide molecules lie within a channel formed by the $C_{25}H_{20}ClN_3O_2$ molecules (Fig.2).

Experimental

The title compound was prepared by the reaction of 4-chlorobenzaldehyde (0.141 g, 1 mmol), 2-(5,5-dimethyl-3-oxocyclohex-1-enylamino)benzoic acid (0.259 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; 0.370 g, 86%, m.p. 513–515 K). Single crystals suitable for X-ray analysis were obtained from an ethanol-DMF solution by slow evaporation.

Refinement

H atoms were positioned geometrically (N—H = 0.86 Å and C—H = 0.93–0.98 Å) and treated as riding, with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C,*N*) for others. Non-H atoms of the solvent molecule were restrained to have the same U^{ij} components within 0.01 Å^2.

Figures

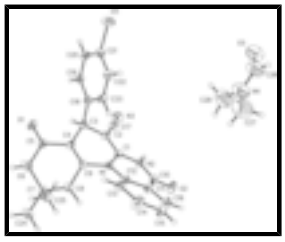


Fig. 1. The molecular structure of the title compound, showing 30% probability displacement ellipsoids.

Fig. 2. The crystal packing of the title compound. Hydrogen bonds are shown as dashed lines.

8-(4-Chlorophenyl)-11,11-dimethyl-5,9-dioxo-6,8,9,10,11,12-hexahydro- 5*H*-quinolino[1,2-*a*]quinazoline-7-carbonitrile *N*,*N*-dimethylformamide solvate

Crystal data

$C_{25}H_{20}ClN_3O_2 \cdot C_3H_7NO$	$F_{000} = 2112$
$M_r = 502.99$	$D_x = 1.348$ Mg m^{-3}
Monoclinic, *C*2/*c*	Melting point: 513-515 K
Hall symbol: -C 2yc	Mo *K*α radiation λ = 0.71073 Å
a = 36.340 (15) Å	Cell parameters from 2136 reflections
b = 11.780 (5) Å	θ = 2.4–25.3º
c = 11.870 (5) Å	μ = 0.19 mm^{-1}
β = 102.714 (5)º	*T* = 298 (2) K
V = 4957 (4) Å^3	Block, colourless
Z = 8	0.23 × 0.14 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer	4326 independent reflections
Radiation source: fine-focus sealed tube	2288 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.063$
T = 298(2) K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 1.8$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −42→43
$T_{min} = 0.957$, $T_{max} = 0.981$	*k* = −13→11
12316 measured reflections	*l* = −14→13

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.074$	H-atom parameters constrained
$wR(F^2) = 0.216$	$w = 1/[\sigma^2(F_o^2) + (0.1126P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
4326 reflections	$\Delta\rho_{max} = 0.34$ e Å^{-3}
325 parameters	$\Delta\rho_{min} = -0.42$ e Å^{-3}
60 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
Cl1	0.22931 (4)	0.20405 (11)	0.35844 (12)	0.0610 (4)
N1	0.10967 (9)	0.6994 (3)	0.4721 (3)	0.0318 (8)
N2	0.05505 (9)	0.6470 (3)	0.3342 (3)	0.0377 (9)
H2	0.0443	0.6346	0.2631	0.045*
N3	0.09005 (14)	0.6117 (4)	0.0656 (4)	0.0663 (13)
N4	0.0483 (2)	0.0499 (6)	0.5512 (7)	0.120 (2)
O1	0.22370 (9)	0.7976 (3)	0.3680 (3)	0.0526 (9)
O2	−0.00184 (9)	0.6354 (3)	0.3812 (3)	0.0583 (10)
O3	0.04761 (18)	−0.0973 (5)	0.4284 (6)	0.1298 (19)
C1	0.09373 (11)	0.6650 (3)	0.3591 (4)	0.0307 (10)
C2	0.11552 (11)	0.6560 (4)	0.2798 (4)	0.0330 (10)
C3	0.15792 (11)	0.6684 (3)	0.3142 (4)	0.0334 (10)
H3	0.1660	0.7058	0.2499	0.040*
C4	0.16683 (11)	0.7482 (3)	0.4158 (4)	0.0318 (10)
C5	0.20159 (12)	0.8182 (4)	0.4284 (4)	0.0374 (11)
C6	0.20743 (12)	0.9120 (4)	0.5151 (4)	0.0434 (12)
H6A	0.2204	0.9740	0.4865	0.052*
H6B	0.2236	0.8849	0.5861	0.052*
C7	0.17098 (12)	0.9572 (4)	0.5417 (4)	0.0406 (11)
C8	0.15012 (13)	0.8553 (4)	0.5792 (4)	0.0401 (11)
H8A	0.1648	0.8252	0.6512	0.048*

H8B	0.1260	0.8804	0.5926	0.048*
C9	0.14360 (11)	0.7626 (3)	0.4891 (3)	0.0306 (10)
C10	0.03270 (12)	0.6470 (4)	0.4115 (4)	0.0412 (11)
C11	0.05214 (11)	0.6549 (4)	0.5329 (4)	0.0357 (10)
C12	0.09081 (11)	0.6765 (3)	0.5618 (4)	0.0326 (10)
C13	0.10988 (13)	0.6722 (4)	0.6773 (4)	0.0393 (11)
H13	0.1359	0.6828	0.6979	0.047*
C14	0.08950 (13)	0.6519 (4)	0.7605 (4)	0.0478 (12)
H14	0.1020	0.6514	0.8379	0.057*
C15	0.05093 (14)	0.6324 (4)	0.7322 (5)	0.0560 (14)
H15	0.0377	0.6198	0.7896	0.067*
C16	0.03289 (14)	0.6318 (4)	0.6192 (4)	0.0503 (13)
H16	0.0072	0.6157	0.5993	0.060*
C17	0.09971 (13)	0.6297 (4)	0.1621 (4)	0.0415 (11)
C18	0.17739 (12)	0.5544 (3)	0.3316 (4)	0.0335 (10)
C19	0.19864 (12)	0.5168 (4)	0.2540 (4)	0.0401 (11)
H19	0.2019	0.5651	0.1951	0.048*
C20	0.21478 (12)	0.4114 (4)	0.2618 (4)	0.0439 (12)
H20	0.2287	0.3882	0.2089	0.053*
C21	0.21002 (12)	0.3403 (4)	0.3495 (4)	0.0410 (11)
C22	0.18959 (13)	0.3743 (4)	0.4295 (4)	0.0449 (12)
H22	0.1867	0.3259	0.4889	0.054*
C23	0.17380 (13)	0.4804 (4)	0.4195 (4)	0.0418 (12)
H23	0.1602	0.5037	0.4734	0.050*
C24	0.17994 (15)	1.0424 (4)	0.6416 (5)	0.0585 (14)
H24A	0.1568	1.0703	0.6578	0.088*
H24B	0.1941	1.1046	0.6206	0.088*
H24C	0.1945	1.0055	0.7090	0.088*
C25	0.14643 (14)	1.0169 (4)	0.4364 (5)	0.0555 (14)
H25A	0.1236	1.0437	0.4557	0.083*
H25B	0.1403	0.9642	0.3733	0.083*
H25C	0.1600	1.0799	0.4144	0.083*
C26	0.0489 (3)	−0.0566 (8)	0.5279 (10)	0.125 (3)
H26	0.0503	−0.1075	0.5886	0.150*
C27	0.0500 (3)	0.0870 (9)	0.6655 (9)	0.163 (3)
H27A	0.0484	0.1683	0.6667	0.244*
H27B	0.0733	0.0628	0.7143	0.244*
H27C	0.0293	0.0550	0.6930	0.244*
C28	0.0440 (3)	0.1374 (7)	0.4606 (9)	0.144 (3)
H28A	0.0435	0.2111	0.4948	0.217*
H28B	0.0208	0.1251	0.4048	0.217*
H28C	0.0648	0.1331	0.4232	0.217*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0635 (9)	0.0489 (8)	0.0656 (9)	0.0194 (6)	0.0034 (7)	−0.0071 (7)
N1	0.0321 (19)	0.038 (2)	0.027 (2)	−0.0028 (16)	0.0105 (16)	−0.0018 (16)

N2	0.030 (2)	0.050 (2)	0.032 (2)	−0.0033 (16)	0.0035 (17)	−0.0042 (17)
N3	0.076 (3)	0.087 (3)	0.035 (3)	0.000 (3)	0.009 (2)	−0.003 (2)
N4	0.135 (5)	0.089 (4)	0.131 (5)	0.005 (4)	0.017 (4)	−0.021 (4)
O1	0.0463 (19)	0.067 (2)	0.053 (2)	−0.0070 (16)	0.0300 (18)	−0.0006 (18)
O2	0.0293 (19)	0.090 (3)	0.053 (2)	−0.0075 (17)	0.0044 (16)	0.0061 (19)
O3	0.158 (5)	0.098 (4)	0.134 (5)	0.008 (3)	0.033 (4)	−0.011 (3)
C1	0.027 (2)	0.032 (2)	0.030 (2)	0.0022 (17)	0.0016 (19)	0.0009 (18)
C2	0.033 (2)	0.040 (2)	0.026 (2)	0.0052 (19)	0.0060 (19)	0.0004 (18)
C3	0.032 (2)	0.044 (3)	0.026 (2)	0.0015 (19)	0.0108 (19)	0.0036 (19)
C4	0.030 (2)	0.039 (2)	0.029 (2)	0.0024 (18)	0.0115 (19)	0.0019 (19)
C5	0.034 (2)	0.038 (3)	0.042 (3)	0.0014 (19)	0.011 (2)	0.007 (2)
C6	0.032 (3)	0.043 (3)	0.055 (3)	−0.006 (2)	0.011 (2)	0.001 (2)
C7	0.040 (3)	0.043 (3)	0.041 (3)	−0.005 (2)	0.013 (2)	−0.002 (2)
C8	0.043 (3)	0.046 (3)	0.034 (3)	−0.002 (2)	0.015 (2)	−0.008 (2)
C9	0.032 (2)	0.034 (2)	0.025 (2)	−0.0053 (18)	0.0035 (19)	0.0029 (18)
C10	0.030 (3)	0.047 (3)	0.047 (3)	−0.005 (2)	0.008 (2)	0.002 (2)
C11	0.027 (2)	0.041 (3)	0.040 (3)	−0.0006 (19)	0.009 (2)	0.002 (2)
C12	0.033 (2)	0.032 (2)	0.035 (3)	0.0010 (18)	0.012 (2)	0.0017 (19)
C13	0.039 (3)	0.045 (3)	0.034 (3)	−0.002 (2)	0.010 (2)	0.002 (2)
C14	0.048 (3)	0.069 (3)	0.028 (3)	0.008 (2)	0.010 (2)	0.006 (2)
C15	0.049 (3)	0.077 (4)	0.049 (3)	−0.002 (3)	0.026 (3)	0.012 (3)
C16	0.042 (3)	0.067 (3)	0.046 (3)	−0.004 (2)	0.018 (3)	0.006 (3)
C17	0.046 (3)	0.050 (3)	0.028 (3)	0.003 (2)	0.008 (2)	0.000 (2)
C18	0.037 (2)	0.037 (3)	0.028 (2)	0.0010 (19)	0.010 (2)	−0.0007 (19)
C19	0.039 (3)	0.047 (3)	0.038 (3)	−0.003 (2)	0.017 (2)	0.000 (2)
C20	0.038 (3)	0.055 (3)	0.042 (3)	0.006 (2)	0.017 (2)	−0.008 (2)
C21	0.039 (3)	0.041 (3)	0.042 (3)	0.007 (2)	0.006 (2)	−0.008 (2)
C22	0.053 (3)	0.046 (3)	0.036 (3)	0.010 (2)	0.010 (2)	0.008 (2)
C23	0.045 (3)	0.053 (3)	0.031 (3)	0.011 (2)	0.017 (2)	0.003 (2)
C24	0.065 (4)	0.053 (3)	0.060 (4)	−0.016 (3)	0.018 (3)	−0.019 (3)
C25	0.052 (3)	0.047 (3)	0.066 (4)	0.005 (2)	0.010 (3)	0.004 (3)
C26	0.133 (5)	0.091 (5)	0.142 (6)	0.002 (5)	0.012 (5)	−0.029 (5)
C27	0.181 (7)	0.156 (7)	0.143 (7)	−0.010 (6)	0.015 (7)	−0.042 (6)
C28	0.158 (7)	0.105 (6)	0.166 (7)	0.002 (5)	0.027 (6)	0.015 (6)

Geometric parameters (Å, °)

Cl1—C21	1.745 (5)	C11—C16	1.389 (6)
N1—C1	1.398 (5)	C11—C12	1.395 (6)
N1—C12	1.413 (5)	C12—C13	1.395 (6)
N1—C9	1.417 (5)	C13—C14	1.380 (6)
N2—C10	1.352 (5)	C13—H13	0.93
N2—C1	1.388 (5)	C14—C15	1.387 (6)
N2—H2	0.86	C14—H14	0.93
N3—C17	1.142 (6)	C15—C16	1.357 (7)
N4—C26	1.286 (10)	C15—H15	0.93
N4—C27	1.414 (11)	C16—H16	0.93
N4—C28	1.473 (10)	C18—C23	1.387 (6)
O1—C5	1.214 (5)	C18—C19	1.398 (6)

O2—C10	1.235 (5)	C19—C20	1.368 (6)
O3—C26	1.266 (10)	C19—H19	0.9300
C1—C2	1.360 (6)	C20—C21	1.376 (6)
C2—C17	1.424 (6)	C20—H20	0.9300
C2—C3	1.512 (6)	C21—C22	1.388 (6)
C3—C4	1.506 (6)	C22—C23	1.370 (6)
C3—C18	1.511 (6)	C22—H22	0.93
C3—H3	0.98	C23—H23	0.93
C4—C9	1.349 (5)	C24—H24A	0.96
C4—C5	1.488 (6)	C24—H24B	0.96
C5—C6	1.493 (6)	C24—H24C	0.96
C6—C7	1.524 (6)	C25—H25A	0.96
C6—H6A	0.97	C25—H25B	0.96
C6—H6B	0.97	C25—H25C	0.96
C7—C24	1.533 (6)	C26—H26	0.93
C7—C8	1.536 (6)	C27—H27A	0.96
C7—C25	1.537 (7)	C27—H27B	0.96
C8—C9	1.510 (6)	C27—H27C	0.96
C8—H8A	0.97	C28—H28A	0.96
C8—H8B	0.97	C28—H28B	0.96
C10—C11	1.462 (6)	C28—H28C	0.96
C1—N1—C12	120.0 (3)	C14—C13—H13	120.6
C1—N1—C9	116.9 (3)	C12—C13—H13	120.6
C12—N1—C9	123.0 (3)	C13—C14—C15	121.9 (5)
C10—N2—C1	125.9 (4)	C13—C14—H14	119.1
C10—N2—H2	117.0	C15—C14—H14	119.1
C1—N2—H2	117.0	C16—C15—C14	118.9 (4)
C26—N4—C27	120.6 (9)	C16—C15—H15	120.5
C26—N4—C28	122.0 (9)	C14—C15—H15	120.5
C27—N4—C28	117.3 (8)	C15—C16—C11	121.1 (5)
C2—C1—N2	123.9 (4)	C15—C16—H16	119.5
C2—C1—N1	120.3 (4)	C11—C16—H16	119.5
N2—C1—N1	115.8 (3)	N3—C17—C2	173.8 (5)
C1—C2—C17	121.7 (4)	C23—C18—C19	117.0 (4)
C1—C2—C3	121.3 (4)	C23—C18—C3	122.8 (4)
C17—C2—C3	117.0 (4)	C19—C18—C3	120.1 (4)
C4—C3—C18	115.7 (3)	C20—C19—C18	122.3 (4)
C4—C3—C2	107.7 (3)	C20—C19—H19	118.9
C18—C3—C2	111.7 (3)	C18—C19—H19	118.9
C4—C3—H3	107.1	C19—C20—C21	118.7 (4)
C18—C3—H3	107.1	C19—C20—H20	120.6
C2—C3—H3	107.1	C21—C20—H20	120.6
C9—C4—C5	120.2 (4)	C20—C21—C22	121.1 (4)
C9—C4—C3	122.6 (4)	C20—C21—Cl1	119.4 (3)
C5—C4—C3	117.0 (3)	C22—C21—Cl1	119.5 (4)
O1—C5—C4	119.2 (4)	C23—C22—C21	118.8 (4)
O1—C5—C6	122.9 (4)	C23—C22—H22	120.6
C4—C5—C6	117.9 (4)	C21—C22—H22	120.6
C5—C6—C7	113.8 (4)	C22—C23—C18	122.1 (4)

C5—C6—H6A	108.8	C22—C23—H23	118.9
C7—C6—H6A	108.8	C18—C23—H23	118.9
C5—C6—H6B	108.8	C7—C24—H24A	109.5
C7—C6—H6B	108.8	C7—C24—H24B	109.5
H6A—C6—H6B	107.7	H24A—C24—H24B	109.5
C6—C7—C24	110.0 (4)	C7—C24—H24C	109.5
C6—C7—C8	107.1 (4)	H24A—C24—H24C	109.5
C24—C7—C8	108.7 (4)	H24B—C24—H24C	109.5
C6—C7—C25	111.5 (4)	C7—C25—H25A	109.5
C24—C7—C25	108.5 (4)	C7—C25—H25B	109.5
C8—C7—C25	111.0 (4)	H25A—C25—H25B	109.5
C9—C8—C7	111.7 (3)	C7—C25—H25C	109.5
C9—C8—H8A	109.3	H25A—C25—H25C	109.5
C7—C8—H8A	109.3	H25B—C25—H25C	109.5
C9—C8—H8B	109.3	O3—C26—N4	124.8 (10)
C7—C8—H8B	109.3	O3—C26—H26	117.6
H8A—C8—H8B	107.9	N4—C26—H26	117.6
C4—C9—N1	119.3 (4)	N4—C27—H27A	109.5
C4—C9—C8	121.6 (4)	N4—C27—H27B	109.5
N1—C9—C8	118.5 (3)	H27A—C27—H27B	109.5
O2—C10—N2	121.7 (4)	N4—C27—H27C	109.5
O2—C10—C11	122.3 (4)	H27A—C27—H27C	109.5
N2—C10—C11	115.9 (4)	H27B—C27—H27C	109.5
C16—C11—C12	119.9 (4)	N4—C28—H28A	109.5
C16—C11—C10	120.2 (4)	N4—C28—H28B	109.5
C12—C11—C10	119.8 (4)	H28A—C28—H28B	109.5
C11—C12—C13	119.4 (4)	N4—C28—H28C	109.5
C11—C12—N1	118.6 (4)	H28A—C28—H28C	109.5
C13—C12—N1	122.0 (4)	H28B—C28—H28C	109.5
C14—C13—C12	118.8 (4)		
C10—N2—C1—C2	175.0 (4)	C7—C8—C9—N1	−144.2 (4)
C10—N2—C1—N1	−8.0 (6)	C1—N2—C10—O2	176.6 (4)
C12—N1—C1—C2	−160.9 (4)	C1—N2—C10—C11	−6.9 (6)
C9—N1—C1—C2	22.9 (5)	O2—C10—C11—C16	10.0 (7)
C12—N1—C1—N2	22.0 (5)	N2—C10—C11—C16	−166.4 (4)
C9—N1—C1—N2	−154.3 (3)	O2—C10—C11—C12	−175.2 (4)
N2—C1—C2—C17	1.7 (7)	N2—C10—C11—C12	8.3 (6)
N1—C1—C2—C17	−175.2 (4)	C16—C11—C12—C13	1.4 (6)
N2—C1—C2—C3	−175.5 (4)	C10—C11—C12—C13	−173.4 (4)
N1—C1—C2—C3	7.6 (6)	C16—C11—C12—N1	179.8 (4)
C1—C2—C3—C4	−30.2 (5)	C10—C11—C12—N1	5.0 (6)
C17—C2—C3—C4	152.5 (4)	C1—N1—C12—C11	−20.8 (5)
C1—C2—C3—C18	97.9 (5)	C9—N1—C12—C11	155.2 (4)
C17—C2—C3—C18	−79.5 (5)	C1—N1—C12—C13	157.6 (4)
C18—C3—C4—C9	−99.9 (5)	C9—N1—C12—C13	−26.4 (6)
C2—C3—C4—C9	25.8 (5)	C11—C12—C13—C14	−3.1 (6)
C18—C3—C4—C5	84.3 (5)	N1—C12—C13—C14	178.6 (4)
C2—C3—C4—C5	−150.0 (4)	C12—C13—C14—C15	2.0 (7)
C9—C4—C5—O1	173.9 (4)	C13—C14—C15—C16	0.8 (8)

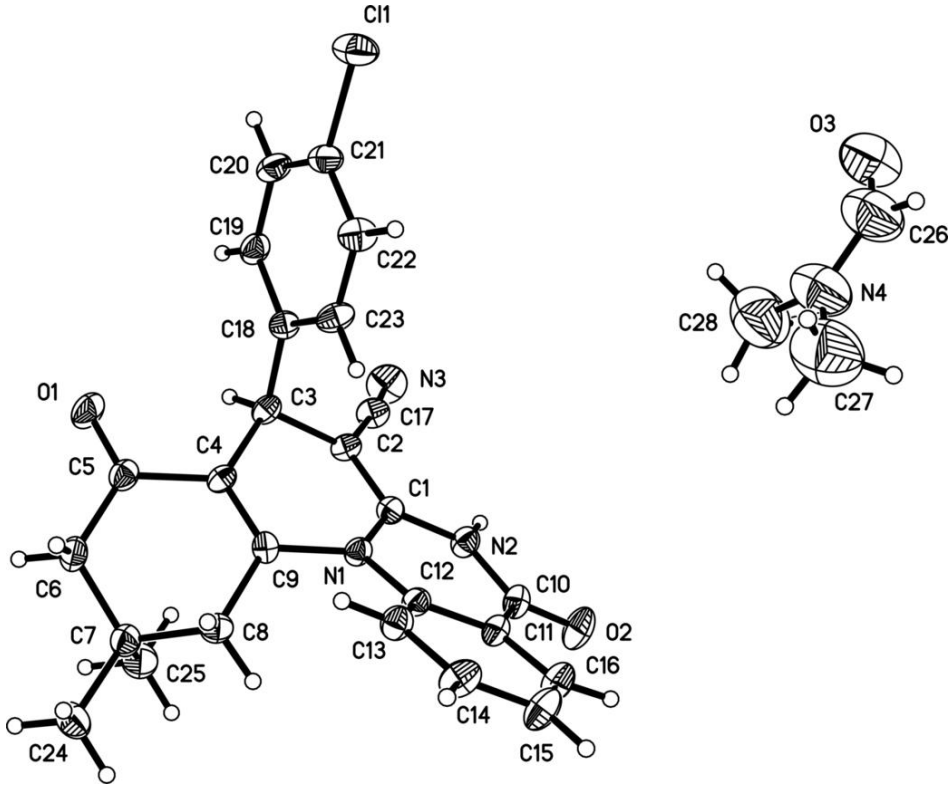
C3—C4—C5—O1	−10.1 (6)	C14—C15—C16—C11	−2.6 (8)
C9—C4—C5—C6	−6.7 (6)	C12—C11—C16—C15	1.5 (7)
C3—C4—C5—C6	169.3 (4)	C10—C11—C16—C15	176.2 (5)
O1—C5—C6—C7	154.3 (4)	C4—C3—C18—C23	57.6 (5)
C4—C5—C6—C7	−25.1 (6)	C2—C3—C18—C23	−66.1 (5)
C5—C6—C7—C24	173.0 (4)	C4—C3—C18—C19	−125.7 (4)
C5—C6—C7—C8	55.0 (5)	C2—C3—C18—C19	110.7 (4)
C5—C6—C7—C25	−66.6 (5)	C23—C18—C19—C20	1.2 (7)
C6—C7—C8—C9	−55.7 (5)	C3—C18—C19—C20	−175.7 (4)
C24—C7—C8—C9	−174.6 (4)	C18—C19—C20—C21	−0.3 (7)
C25—C7—C8—C9	66.2 (5)	C19—C20—C21—C22	−0.6 (7)
C5—C4—C9—N1	176.8 (4)	C19—C20—C21—Cl1	178.3 (4)
C3—C4—C9—N1	1.1 (6)	C20—C21—C22—C23	0.5 (7)
C5—C4—C9—C8	5.0 (6)	Cl1—C21—C22—C23	−178.3 (4)
C3—C4—C9—C8	−170.6 (4)	C21—C22—C23—C18	0.5 (7)
C1—N1—C9—C4	−27.3 (5)	C19—C18—C23—C22	−1.3 (7)
C12—N1—C9—C4	156.5 (4)	C3—C18—C23—C22	175.6 (4)
C1—N1—C9—C8	144.7 (4)	C27—N4—C26—O3	−179.7 (9)
C12—N1—C9—C8	−31.4 (5)	C28—N4—C26—O3	3.3 (15)
C7—C8—C9—C4	27.6 (6)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2^{i}	0.86	2.04	2.849 (5)	157
C20—H20···O1^{ii}	0.93	2.38	3.269 (6)	160
C13—H13···Cg1^{iii}	0.93	2.84	3.530 (5)	132
C26—H26···Cg2^{iv}	0.93	2.96	3.720 (10)	140

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

Fig. 1


Cl1
C21
C20
C19
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C6
C9
N1
C12
C10
C7
C8
C11
O2
C13
C16
C25
C14
C24
C15
O3
C26
N4
C28
C27

Fig. 2