Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

8,8-Dimethyl-5-(4-methylphenyl)-8,9-dihydropyrimido[4,5-*b*]quinoline-2,4,6(1*H*,3*H*,7*H*)-trione *N*,*N*-dimethylformamide solvate

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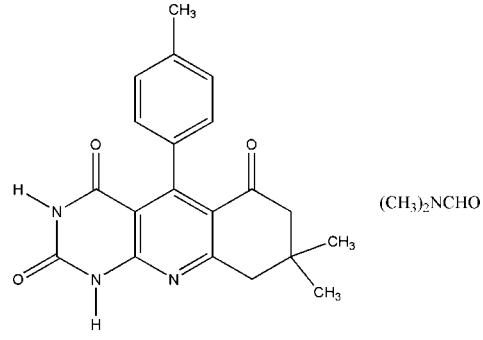
Received 16 October 2007; accepted 25 January 2008

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.046; wR factor = 0.135; data-to-parameter ratio = 15.0.

The title compound, $C_{20}H_{19}N_3O_3\cdot C_3H_7NO$, was synthesized by the reaction of 6-aminopyrimidine-2,4(1*H*,3*H*)-dione and 4-methylbenzaldehyde with 5,5-dimethyl-1,3-cyclohexanedione in 1-butyl-3-methylimidazolium bromide at 363 K. The pyrimidine ring adopts a half-chair conformation while the six-membered ring fused to the pyridine ring adopts a skew-boat conformation. The dihedral angle between the pyridine ring and the attached benzene ring is 2.38(8)°

Related literature

For related literature, see: Bhuyan *et al.* (1999); Clercq (1986); Gangjee *et al.* (1999); Griengl *et al.* (1987); Hirota *et al.* (1981); Jones *et al.* (1979); Nasr & Gineinah (2002); Pontikis & Monneret (1994); Sasaki *et al.* (1980).



Experimental

Crystal data

$C_{20}H_{19}N_3O_3\cdot C_3H_7NO$
M_r = 422.48
Triclinic, $P\overline{1}$
a = 8.8252 (16) Å
b = 10.289 (2) Å
c = 12.316 (2) Å
α = 95.898 (3)°
β = 93.115 (3)°
γ = 94.719 (3)°
V = 1106.4 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 (2) K
0.24 × 0.16 × 0.12 mm

Data collection

Bruker SMART 1000 diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.978, T_{max} = 0.989
6083 measured reflections
4285 independent reflections
2833 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.134
S = 1.00
4285 reflections
285 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O4^{i}	0.90	1.96	2.854 (2)	170
N2—H2···O1^{ii}	0.90	1.97	2.846 (2)	167

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, -y+1, -z+1$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The authors are grateful to the Foundation of the Key Laboratory of Biotechnology on Medical Plants of Jiangsu Province for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CS2057).

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  doi:10.1107/S1600536808002924

supplementary materials

Acta Cryst. (2008). E**64**, o606 [**doi:10.1107/S1600536808002924**]

8,8-Dimethyl-5-(4-methylphenyl)-8,9-dihydropyrimido[4,5-*b*]quinoline-2,4,6(1*H*,3*H*,7*H*)-trione *N*,*N*-dimethylformamide solvate

J. Wang, S.-N. Ni and D. Shi

Comment

The importance of uracil and its annelated derivatives is well recognized by synthetic (Sasaki *et al.*, 1980; Bhuyan *et al.*, 1999) as well as biological (Griengl *et al.*, 1987; Pontikis *et al.*, 1994) chemists. With the development of clinically useful anticancer and antiviral drugs (Clercq *et al.*,1986; Jones *et al.*, 1979), there has recently been remarkable interest in the synthetic manipulations of uracils (Hirota *et al.*, 1981). Pyrido[2,3-*d*]pyrimidines represent a heterocyclic ring system of considerable interest because of several biological activities associated with this scaffold. Some analogues have been found to act as anticancer agents inhibiting dihydrofolate reductases or tyrosine kinases (Gangjee *et al.*, 1999), while others are known antiviral agents (Nasr *et al.*, 2002).

The title compound was synthesized by the reaction of 6-aminopyrimidine-2,4(1*H*,3H)-dione and 4-methylbenzaldehyde with 5,5-dimethyl-1,3-cyclohexanedione using 1-butyl-3-methylimidazolium bromide ([bmim]Br) as solvent at 363 K.

In the title compound the pyridine ring (C13/C3/C4/C5/C12/N3) is a newly formed planar ring. The pyrimidine ring is less planar with atom C2 deviating from the C3/C1/C13/N1/N2 plane by −0.108 (3) Å (Fig. 1). The six-membered ring fused on to the pyridine ring adopts a skew-boat conformation; atoms C6, C5, C12 and C11 are coplanar, with atoms C7 and C8 deviating from the plane by −0.301 (2) and 0.458 (6) Å, respectively. The dihedral angle between the C13/C3/C4/C5/C12/N3 plane and the C3/C1/C13/N1/N2 plane is 2.38 (8) °, they are almost coplanar. The dihedral angle between the C13/C3/C4/C5/C12/N3 plane and the C14/C15/C16/C17/C19/C20 plane is 77.99 (5) °. The molecules are linked by N1—H1···O4 and N2—H2···O1 intermolecular hydrogen bonds (Table 1) to form dimers (Fig. 2).

Experimental

The title compound was prepared by the reaction of 6-aminopyrimidine-2,4(1*H*,3H)-dione (2 mmol) and 4-methylaldehyde (2 mmol) with 5,5-dimethyl-1,3-cyclohexanedione (2 mmol) in [bmim]Br (2 ml) at 363 K. Crystals of the title compound suitable for X-ray diffraction were obtained by slow evaporation of a *N*,*N*-dimethylformamide and water solution. ^{1}H NMR (DMSO-d_6, δ): 1.03 (6*H*, s, 2*CH_3), 2.35 (3*H*, s, CH_3), 2.40 (2*H*, s, CH_2), 2.74 (3*H*, s, CH_3), 2.90 (3*H*, s, CH_3), 3.01 (2*H*, s, CH_2), 6.89 (2*H*, d, J = 8.0 Hz, ArH), 7.08 (2*H*, d, J = 8.0 Hz, ArH), 7.96 (1*H*, s, CH), 11.12 (1*H*, s, NH), 11.88 (1*H*, s, NH).

Refinement

The amino H atoms were located in a difference map and kept riding subsequently. The C-bound H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2–1.5 U_{eq}(C).

Figures

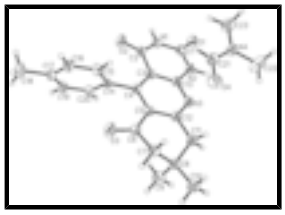

Fig. 1. The molecular structure of the title compound showing 40% probability displacement ellipsoids and the atom-numbering scheme.

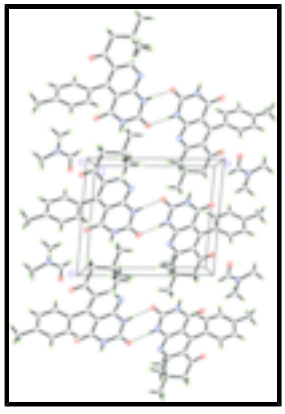

Fig. 2. The crystal packing of the title compound.

8,8-Dimethyl-5-(4-methylphenyl)-8,9-dihydropyrimido[4,5-*b*]quinoline- 2,4,6(1*H*,3H,7*H*)-trione *N*,*N*-dimethylformamide solvate

Crystal data

$C_{20}H_{19}N_3O_3 \cdot C_3H_7NO$	$Z = 2$
$M_r = 422.48$	$F_{000} = 448$
Triclinic, $P\overline{1}$	$D_x = 1.268$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.8252$ (16) Å	Cell parameters from 2318 reflections
$b = 10.289$ (2) Å	$\theta = 2.5$–26.3º
$c = 12.316$ (2) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 95.898$ (3)º	$T = 294$ (2) K
$\beta = 93.115$ (3)º	Block, colorless
$\gamma = 94.719$ (3)º	0.24 × 0.16 × 0.12 mm
$V = 1106.4$ (4) Å^3	

Data collection

Bruker SMART 1000 diffractometer	4285 independent reflections
Radiation source: fine-focus sealed tube	2833 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.018$
$T = 294$(2) K	$\theta_{max} = 26.0$º
φ and ω scans	$\theta_{min} = 1.7$º
Absorption correction: multi-scan	$h = -9 \rightarrow 10$

(SADABS; Sheldrick, 1996)
T_{min} = 0.978, T_{max} = 0.989
6083 measured reflections
$k = -10 \rightarrow 12$
$l = -15 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.134$
$S = 1.00$
4285 reflections
285 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0627P)^2 + 0.2466P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.66314 (15)	0.41510 (14)	0.49822 (12)	0.0587 (4)
O2	0.61458 (17)	0.01747 (15)	0.62264 (14)	0.0726 (5)
O3	0.1315 (2)	−0.03738 (19)	0.88397 (19)	0.1117 (8)
N1	0.63982 (17)	0.22017 (16)	0.56995 (13)	0.0485 (4)
H1	0.7300	0.2042	0.5431	0.058*
N2	0.45129 (17)	0.36219 (16)	0.58594 (14)	0.0503 (4)
H2	0.4112	0.4358	0.5704	0.060*
N3	0.24137 (17)	0.32697 (15)	0.68299 (13)	0.0475 (4)
C1	0.5892 (2)	0.33743 (19)	0.54821 (16)	0.0456 (5)
C2	0.5624 (2)	0.1225 (2)	0.62047 (16)	0.0474 (5)
C3	0.42031 (19)	0.15976 (18)	0.66859 (14)	0.0411 (4)
C4	0.3337 (2)	0.08197 (18)	0.73401 (14)	0.0404 (4)
C5	0.1967 (2)	0.12798 (18)	0.76963 (15)	0.0438 (4)
C6	0.0920 (2)	0.0536 (2)	0.83849 (18)	0.0556 (5)
C7	−0.0623 (2)	0.10073 (19)	0.85164 (18)	0.0533 (5)
H7A	−0.1091	0.0604	0.9108	0.064*
H7B	−0.1259	0.0737	0.7851	0.064*
C8	−0.0549 (2)	0.25028 (19)	0.87620 (16)	0.0473 (5)
C9	0.0437 (3)	0.2941 (2)	0.98100 (19)	0.0691 (6)
H9A	0.0456	0.3875	0.9972	0.104*
H9B	0.1455	0.2703	0.9716	0.104*
H9C	0.0023	0.2520	1.0402	0.104*
C10	−0.2145 (2)	0.2942 (2)	0.8905 (2)	0.0650 (6)
H10A	−0.2567	0.2574	0.9520	0.098*
H10B	−0.2785	0.2647	0.8256	0.098*
H10C	−0.2088	0.3882	0.9029	0.098*

C11	0.0129 (2)	0.3077 (2)	0.77834 (18)	0.0544 (5)
H11A	−0.0631	0.2944	0.7174	0.065*
H11B	0.0350	0.4016	0.7966	0.065*
C12	0.1560 (2)	0.25081 (18)	0.74199 (16)	0.0458 (5)
C13	0.36838 (19)	0.27982 (18)	0.64708 (15)	0.0420 (4)
C14	0.38754 (19)	−0.04296 (18)	0.76792 (14)	0.0398 (4)
C15	0.3251 (2)	−0.16362 (19)	0.71966 (16)	0.0503 (5)
H15	0.2520	−0.1684	0.6618	0.060*
C16	0.3704 (2)	−0.2778 (2)	0.75669 (18)	0.0558 (5)
H16	0.3266	−0.3584	0.7235	0.067*
C17	0.4790 (2)	−0.2743 (2)	0.84175 (17)	0.0530 (5)
C18	0.5257 (3)	−0.4007 (3)	0.8817 (2)	0.0871 (8)
H18A	0.6009	−0.4355	0.8362	0.131*
H18B	0.4381	−0.4632	0.8783	0.131*
H18C	0.5675	−0.3833	0.9559	0.131*
C19	0.5433 (2)	−0.1537 (2)	0.88808 (17)	0.0548 (5)
H19	0.6181	−0.1493	0.9448	0.066*
C20	0.4988 (2)	−0.0388 (2)	0.85185 (16)	0.0485 (5)
H20	0.5442	0.0417	0.8842	0.058*
O4	−0.06364 (17)	0.16079 (19)	0.51009 (14)	0.0800 (5)
N4	0.09249 (19)	0.25425 (17)	0.39330 (15)	0.0582 (5)
C21	0.0462 (2)	0.1599 (3)	0.45278 (19)	0.0658 (6)
H21	0.1016	0.0868	0.4510	0.079*
C22	0.0147 (3)	0.3718 (2)	0.3888 (3)	0.0941 (10)
H22A	−0.0687	0.3694	0.4357	0.141*
H22B	−0.0234	0.3772	0.3151	0.141*
H22C	0.0845	0.4472	0.4128	0.141*
C23	0.2201 (3)	0.2428 (3)	0.3249 (2)	0.0797 (8)
H23A	0.2674	0.1643	0.3368	0.120*
H23B	0.2928	0.3176	0.3431	0.120*
H23C	0.1846	0.2390	0.2495	0.120*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0489 (8)	0.0557 (9)	0.0800 (10)	0.0090 (6)	0.0278 (7)	0.0317 (7)
O2	0.0714 (10)	0.0658 (10)	0.0973 (12)	0.0368 (8)	0.0439 (9)	0.0436 (9)
O3	0.1149 (15)	0.0955 (14)	0.1588 (19)	0.0597 (12)	0.0936 (14)	0.0921 (14)
N1	0.0396 (8)	0.0558 (10)	0.0574 (10)	0.0151 (7)	0.0189 (7)	0.0236 (8)
N2	0.0446 (9)	0.0477 (9)	0.0676 (11)	0.0153 (7)	0.0225 (8)	0.0298 (8)
N3	0.0434 (8)	0.0466 (9)	0.0597 (10)	0.0131 (7)	0.0181 (7)	0.0249 (8)
C1	0.0395 (10)	0.0480 (11)	0.0535 (11)	0.0077 (8)	0.0124 (9)	0.0168 (9)
C2	0.0473 (11)	0.0521 (12)	0.0497 (11)	0.0165 (9)	0.0143 (9)	0.0232 (9)
C3	0.0396 (10)	0.0441 (10)	0.0441 (10)	0.0110 (8)	0.0097 (8)	0.0163 (8)
C4	0.0429 (10)	0.0408 (10)	0.0412 (10)	0.0100 (8)	0.0087 (8)	0.0132 (8)
C5	0.0440 (10)	0.0443 (11)	0.0478 (11)	0.0098 (8)	0.0142 (8)	0.0162 (8)
C6	0.0633 (13)	0.0447 (11)	0.0665 (13)	0.0131 (10)	0.0296 (11)	0.0229 (10)
C7	0.0485 (11)	0.0497 (12)	0.0646 (13)	0.0020 (9)	0.0198 (10)	0.0141 (10)

C8	0.0420 (10)	0.0464 (11)	0.0575 (12)	0.0090 (8)	0.0173 (9)	0.0138 (9)
C9	0.0749 (15)	0.0634 (15)	0.0693 (15)	0.0109 (12)	0.0075 (12)	0.0036 (12)
C10	0.0516 (12)	0.0669 (15)	0.0824 (16)	0.0141 (11)	0.0280 (11)	0.0164 (12)
C11	0.0459 (11)	0.0571 (13)	0.0687 (13)	0.0190 (9)	0.0227 (10)	0.0263 (10)
C12	0.0432 (10)	0.0462 (11)	0.0533 (11)	0.0113 (8)	0.0145 (9)	0.0194 (9)
C13	0.0376 (9)	0.0447 (11)	0.0484 (11)	0.0088 (8)	0.0120 (8)	0.0190 (8)
C14	0.0409 (9)	0.0410 (10)	0.0422 (10)	0.0103 (8)	0.0136 (8)	0.0163 (8)
C15	0.0527 (11)	0.0477 (12)	0.0514 (12)	0.0066 (9)	−0.0011 (9)	0.0113 (9)
C16	0.0631 (13)	0.0389 (11)	0.0670 (14)	0.0073 (9)	0.0069 (11)	0.0095 (10)
C17	0.0564 (12)	0.0500 (13)	0.0601 (13)	0.0194 (10)	0.0158 (10)	0.0236 (10)
C18	0.0965 (19)	0.0673 (17)	0.109 (2)	0.0288 (14)	0.0107 (16)	0.0448 (15)
C19	0.0550 (12)	0.0627 (14)	0.0508 (12)	0.0155 (10)	−0.0006 (10)	0.0198 (10)
C20	0.0520 (11)	0.0463 (11)	0.0483 (11)	0.0042 (9)	0.0019 (9)	0.0116 (9)
O4	0.0493 (9)	0.1184 (15)	0.0802 (11)	0.0195 (9)	0.0241 (8)	0.0278 (10)
N4	0.0482 (10)	0.0595 (11)	0.0705 (12)	0.0154 (8)	0.0172 (9)	0.0086 (9)
C21	0.0501 (12)	0.0872 (18)	0.0675 (15)	0.0244 (12)	0.0143 (11)	0.0229 (13)
C22	0.0639 (15)	0.0549 (15)	0.166 (3)	0.0120 (12)	0.0356 (17)	0.0052 (16)
C23	0.0733 (16)	0.0849 (18)	0.0918 (19)	0.0286 (14)	0.0400 (14)	0.0257 (15)

Geometric parameters (Å, °)

O1—C1	1.226 (2)	C10—H10C	0.9600
O2—C2	1.211 (2)	C11—C12	1.506 (2)
O3—C6	1.205 (2)	C11—H11A	0.9700
N1—C1	1.367 (2)	C11—H11B	0.9700
N1—C2	1.388 (2)	C14—C15	1.378 (3)
N1—H1	0.8997	C14—C20	1.382 (3)
N2—C1	1.360 (2)	C15—C16	1.383 (3)
N2—C13	1.382 (2)	C15—H15	0.9300
N2—H2	0.8952	C16—C17	1.377 (3)
N3—C13	1.337 (2)	C16—H16	0.9300
N3—C12	1.338 (2)	C17—C19	1.375 (3)
C2—C3	1.478 (2)	C17—C18	1.516 (3)
C3—C13	1.398 (2)	C18—H18A	0.9600
C3—C4	1.405 (2)	C18—H18B	0.9600
C4—C5	1.409 (2)	C18—H18C	0.9600
C4—C14	1.497 (2)	C19—C20	1.384 (3)
C5—C12	1.411 (2)	C19—H19	0.9300
C5—C6	1.503 (2)	C20—H20	0.9300
C6—C7	1.495 (3)	O4—C21	1.230 (2)
C7—C8	1.533 (3)	N4—C21	1.326 (3)
C7—H7A	0.9700	N4—C22	1.444 (3)
C7—H7B	0.9700	N4—C23	1.447 (3)
C8—C9	1.523 (3)	C21—H21	0.9300
C8—C11	1.526 (3)	C22—H22A	0.9600
C8—C10	1.528 (3)	C22—H22B	0.9600
C9—H9A	0.9600	C22—H22C	0.9600
C9—H9B	0.9600	C23—H23A	0.9600
C9—H9C	0.9600	C23—H23B	0.9600

C10—H10A	0.9600	C23—H23C	0.9600
C10—H10B	0.9600		
C1—N1—C2	127.04 (15)	C12—C11—H11B	108.5
C1—N1—H1	114.5	C8—C11—H11B	108.5
C2—N1—H1	118.3	H11A—C11—H11B	107.5
C1—N2—C13	123.74 (15)	N3—C12—C5	123.18 (16)
C1—N2—H2	118.6	N3—C12—C11	114.43 (16)
C13—N2—H2	117.6	C5—C12—C11	122.38 (16)
C13—N3—C12	116.77 (15)	N3—C13—N2	114.21 (15)
O1—C1—N2	122.24 (17)	N3—C13—C3	125.32 (15)
O1—C1—N1	122.30 (16)	N2—C13—C3	120.46 (15)
N2—C1—N1	115.46 (16)	C15—C14—C20	118.52 (17)
O2—C2—N1	119.28 (17)	C15—C14—C4	121.53 (17)
O2—C2—C3	125.96 (17)	C20—C14—C4	119.91 (17)
N1—C2—C3	114.75 (16)	C14—C15—C16	120.51 (19)
C13—C3—C4	117.92 (15)	C14—C15—H15	119.7
C13—C3—C2	117.88 (15)	C16—C15—H15	119.7
C4—C3—C2	124.20 (16)	C17—C16—C15	121.3 (2)
C3—C4—C5	117.59 (16)	C17—C16—H16	119.4
C3—C4—C14	121.21 (15)	C15—C16—H16	119.4
C5—C4—C14	121.17 (15)	C19—C17—C16	117.98 (18)
C4—C5—C12	119.14 (16)	C19—C17—C18	121.7 (2)
C4—C5—C6	123.10 (16)	C16—C17—C18	120.4 (2)
C12—C5—C6	117.74 (16)	C17—C18—H18A	109.5
O3—C6—C7	121.12 (18)	C17—C18—H18B	109.5
O3—C6—C5	121.92 (19)	H18A—C18—H18B	109.5
C7—C6—C5	116.92 (17)	C17—C18—H18C	109.5
C6—C7—C8	111.96 (16)	H18A—C18—H18C	109.5
C6—C7—H7A	109.2	H18B—C18—H18C	109.5
C8—C7—H7A	109.2	C17—C19—C20	121.29 (19)
C6—C7—H7B	109.2	C17—C19—H19	119.4
C8—C7—H7B	109.2	C20—C19—H19	119.4
H7A—C7—H7B	107.9	C14—C20—C19	120.40 (19)
C9—C8—C11	111.19 (18)	C14—C20—H20	119.8
C9—C8—C10	108.88 (18)	C19—C20—H20	119.8
C11—C8—C10	109.94 (16)	C21—N4—C22	122.15 (19)
C9—C8—C7	109.80 (17)	C21—N4—C23	121.93 (19)
C11—C8—C7	106.70 (16)	C22—N4—C23	115.86 (19)
C10—C8—C7	110.32 (16)	O4—C21—N4	125.7 (2)
C8—C9—H9A	109.5	O4—C21—H21	117.2
C8—C9—H9B	109.5	N4—C21—H21	117.2
H9A—C9—H9B	109.5	N4—C22—H22A	109.5
C8—C9—H9C	109.5	N4—C22—H22B	109.5
H9A—C9—H9C	109.5	H22A—C22—H22B	109.5
H9B—C9—H9C	109.5	N4—C22—H22C	109.5
C8—C10—H10A	109.5	H22A—C22—H22C	109.5
C8—C10—H10B	109.5	H22B—C22—H22C	109.5
H10A—C10—H10B	109.5	N4—C23—H23A	109.5
C8—C10—H10C	109.5	N4—C23—H23B	109.5

H10A—C10—H10C	109.5	H23A—C23—H23B	109.5
H10B—C10—H10C	109.5	N4—C23—H23C	109.5
C12—C11—C8	114.94 (16)	H23A—C23—H23C	109.5
C12—C11—H11A	108.5	H23B—C23—H23C	109.5
C8—C11—H11A	108.5		
C13—N2—C1—O1	−176.75 (19)	C13—N3—C12—C11	−178.87 (17)
C13—N2—C1—N1	3.2 (3)	C4—C5—C12—N3	0.0 (3)
C2—N1—C1—O1	−176.3 (2)	C6—C5—C12—N3	178.69 (19)
C2—N1—C1—N2	3.8 (3)	C4—C5—C12—C11	−179.04 (18)
C1—N1—C2—O2	171.4 (2)	C6—C5—C12—C11	−0.4 (3)
C1—N1—C2—C3	−9.2 (3)	C8—C11—C12—N3	−159.06 (18)
O2—C2—C3—C13	−172.7 (2)	C8—C11—C12—C5	20.1 (3)
N1—C2—C3—C13	8.0 (3)	C12—N3—C13—N2	179.51 (17)
O2—C2—C3—C4	7.0 (3)	C12—N3—C13—C3	−1.7 (3)
N1—C2—C3—C4	−172.41 (18)	C1—N2—C13—N3	175.18 (18)
C13—C3—C4—C5	2.6 (3)	C1—N2—C13—C3	−3.7 (3)
C2—C3—C4—C5	−176.97 (18)	C4—C3—C13—N3	−0.6 (3)
C13—C3—C4—C14	−175.28 (17)	C2—C3—C13—N3	179.01 (18)
C2—C3—C4—C14	5.1 (3)	C4—C3—C13—N2	178.07 (17)
C3—C4—C5—C12	−2.4 (3)	C2—C3—C13—N2	−2.3 (3)
C14—C4—C5—C12	175.53 (18)	C3—C4—C14—C15	−104.4 (2)
C3—C4—C5—C6	179.03 (18)	C5—C4—C14—C15	77.8 (2)
C14—C4—C5—C6	−3.1 (3)	C3—C4—C14—C20	77.8 (2)
C4—C5—C6—O3	13.9 (4)	C5—C4—C14—C20	−100.1 (2)
C12—C5—C6—O3	−164.7 (2)	C20—C14—C15—C16	1.7 (3)
C4—C5—C6—C7	−168.34 (19)	C4—C14—C15—C16	−176.17 (17)
C12—C5—C6—C7	13.1 (3)	C14—C15—C16—C17	−0.4 (3)
O3—C6—C7—C8	132.9 (3)	C15—C16—C17—C19	−1.0 (3)
C5—C6—C7—C8	−45.0 (3)	C15—C16—C17—C18	179.4 (2)
C6—C7—C8—C9	−59.3 (2)	C16—C17—C19—C20	1.0 (3)
C6—C7—C8—C11	61.3 (2)	C18—C17—C19—C20	−179.3 (2)
C6—C7—C8—C10	−179.26 (18)	C15—C14—C20—C19	−1.6 (3)
C9—C8—C11—C12	70.9 (2)	C4—C14—C20—C19	176.28 (17)
C10—C8—C11—C12	−168.42 (18)	C17—C19—C20—C14	0.3 (3)
C7—C8—C11—C12	−48.8 (2)	C22—N4—C21—O4	−0.6 (4)
C13—N3—C12—C5	2.0 (3)	C23—N4—C21—O4	−177.7 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O4^{i}	0.90	1.96	2.854 (2)	170
N2—H2···O1^{ii}	0.90	1.97	2.846 (2)	167

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

Fig. 1

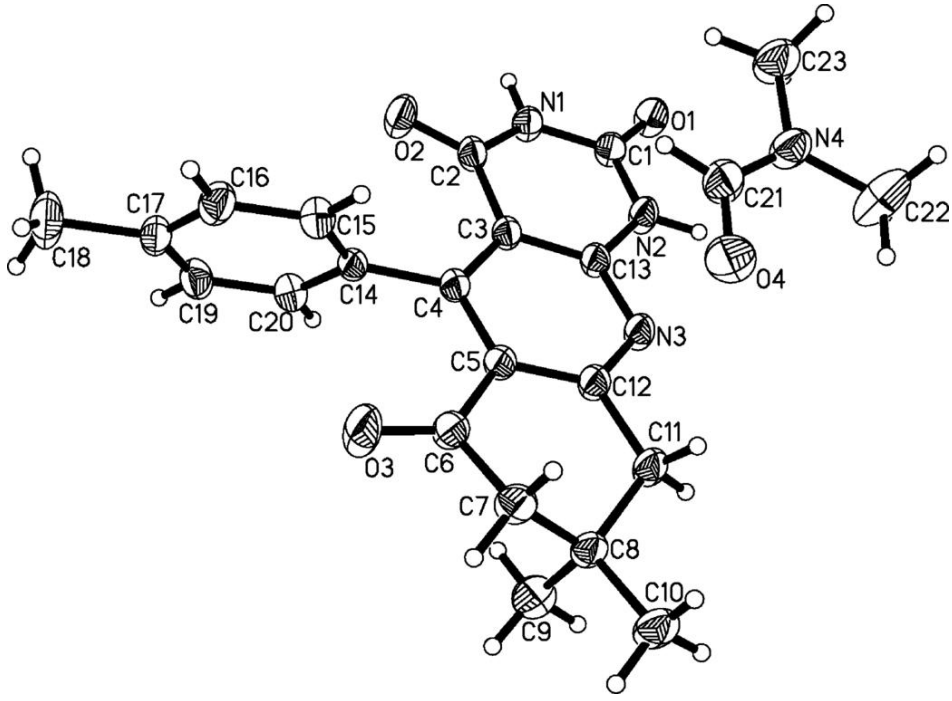
C23
N1
O1
N4
O2
C2
C1
C21
C22
C16
C17
C15
C3
N2
C18
C13
O4
C19
C20
C14
C4
N3
C5
C12
C11
O3
C6
C7
C8
C10
C9

Fig. 2