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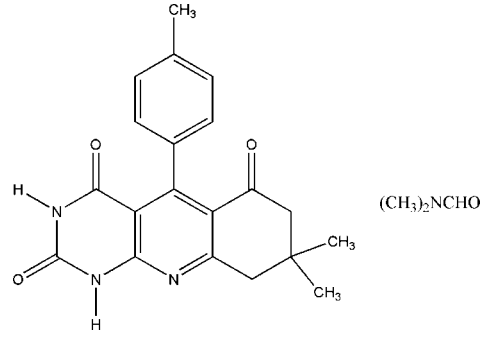
Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.046; $wR$ factor = 0.135; data-to-parameter ratio = 15.0.

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
$\alpha$ = 95.898 (3)°
$\beta$ = 93.115 (3)°
$\gamma$ = 94.719 (3)°
$V$ = 1106.4 (4) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 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*.

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

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# supporting information

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## 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

**Jing Wang, Sai-Nan Ni and Daqing Shi**

### S1. 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-methyl-benzaldehyde 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).

### S2. Experimental

The title compound was prepared by the reaction of 6-aminopyrimidine-2,4(1*H*,3H)-dione (2 mmol) and 4-methyl-aldehyde (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-$d_6$, $\delta$): 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).

### S3. 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).

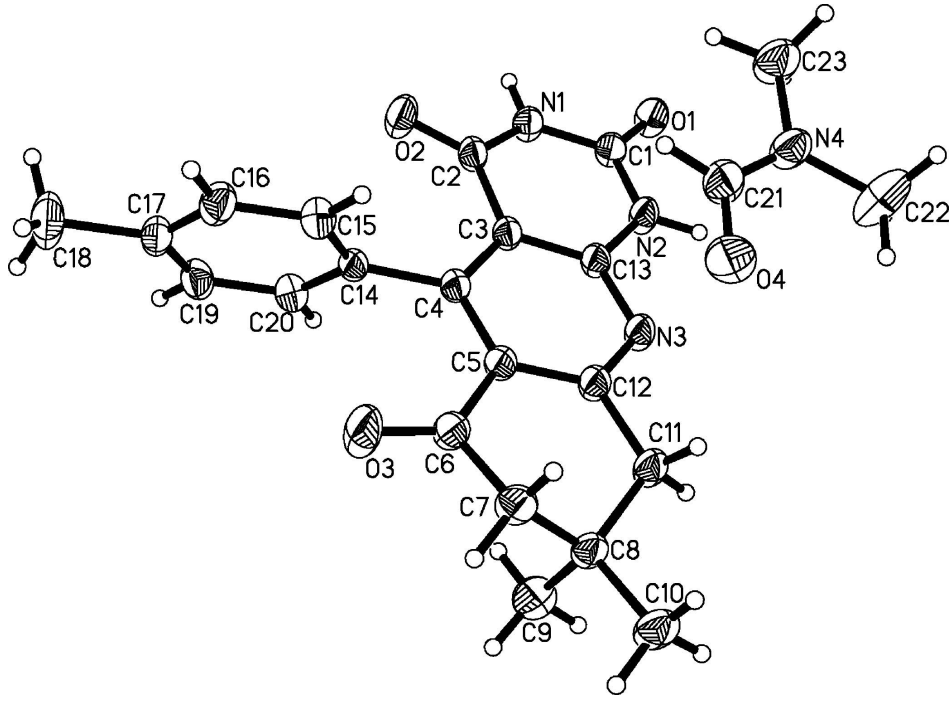


**Figure 1**

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

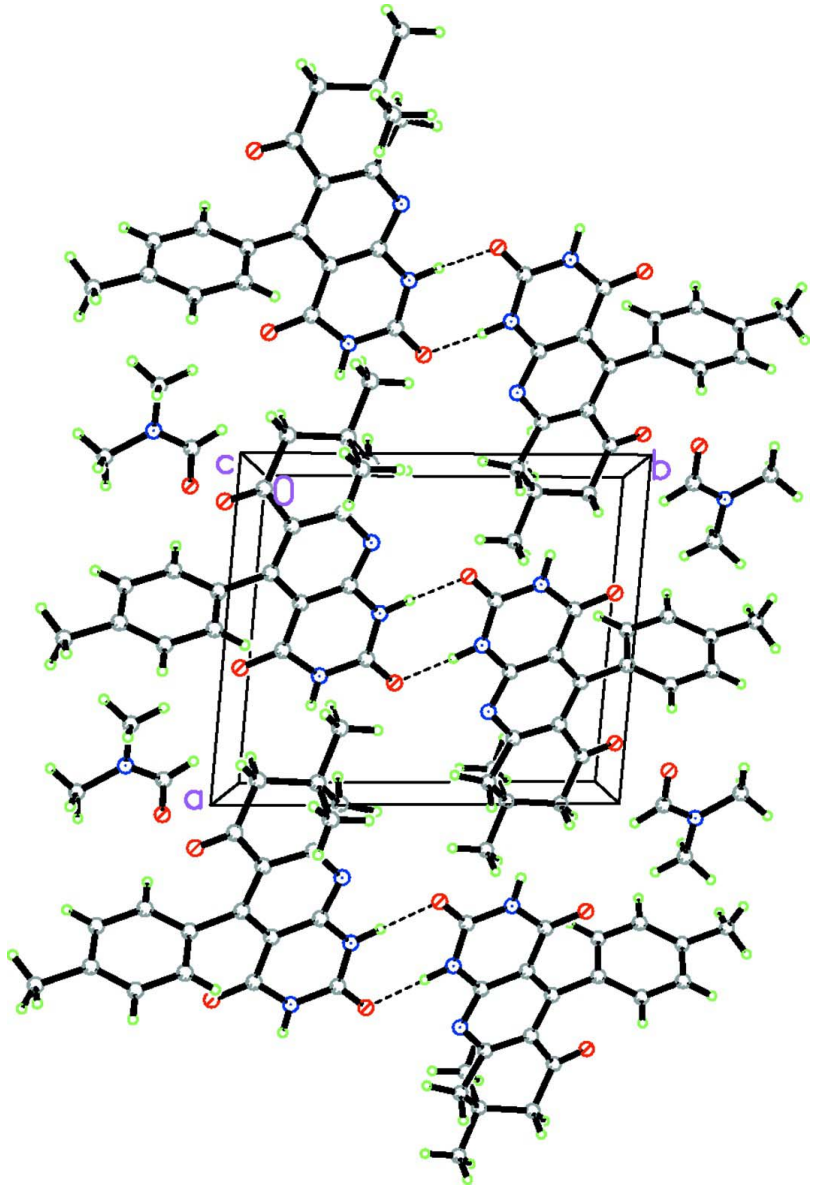


**Figure 2**

The crystal packing of the title compound.

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*Crystal data*

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

$T$ = 294 K
Block, colorless

0.24 × 0.16 × 0.12 mm

*Data collection*

Bruker SMART 1000 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
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
$\theta_{max}$ = 26.0°, $\theta_{min}$ = 1.7°
$h$ = −9→10
$k$ = −10→12
$l$ = −15→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$^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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 (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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, º)*

| | | | |
|---|---|---|---|
| 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 |

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| 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 | | |

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| 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 |

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|---|---|---|---|
| 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 | | |

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| 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, º)*

| *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) $-x+1, -y+1, -z+1$.