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# 6-Benzyl-6,7-dihydro-5*H*-pyrrolo[3,4-*b*]-pyridine-5,7-dione

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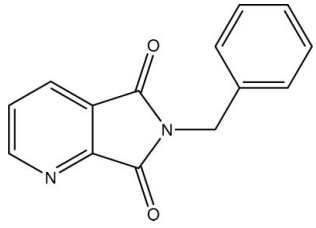
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.058; $wR$ factor = 0.174; data-to-parameter ratio = 12.8.

In the title compound, $C_{14}H_{10}N_2O_2$, the dihedral angle between the heterocyclic ring system and the phenyl ring is 45.8 (5)°. Weak intermolecular C—H···N hydrogen bonding is present in the crystal structure.

## Related literature

The title compound is a key intermediate in the synthesis of the quinolone antibiotic moxifloxacin [systematic name: 1-cyclopropyl-7-[(1*S*,6*S*)-2,8-diazabicyclo[4.3.0]non-8-yl]-6-fluoro-8-methoxy-4-oxo-quinoline-3-carboxylic acid], see: Petersen *et al.* (1993). For a related structure, see: Garduño-Beltrán *et al.* (2009).



## Experimental

### Crystal data

$C_{14}H_{10}N_2O_2$
$M_r$ = 238.24
Monoclinic, $P2_1/c$
$a$ = 11.8548 (6) Å
$b$ = 12.3969 (8) Å
$c$ = 8.1676 (4) Å
$\beta$ = 107.45 (3)°
$V$ = 1145.1 (2) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 293 K
0.20 × 0.10 × 0.10 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.981, $T_{max}$ = 0.991
2087 measured reflections
2087 independent reflections
1100 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.045
3 standard reflections every 200 reflections
intensity decay: 1%

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.174
$S$ = 1.00
2087 reflections
163 parameters
12 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.13 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C10—H10$A$···N2$^{i}$ | 0.93 | 2.46 | 3.386 (3) | 177 |

Symmetry code: (i) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors thank the Center of Testing and Analysis, Nanjing University, for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5342).

## References

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# supporting information

*Acta Cryst.* (2011). E**67**, o2895 [doi:10.1107/S1600536811041006]

## 6-Benzyl-6,7-dihydro-5*H*-pyrrolo[3,4-*b*]pyridine-5,7-dione

**Hong-Shun Sun, Long Jiang, Hong Xu, Xin-Hua Lu and Yu-Long Li**

### S1. Comment

Moxifloxacin (Petersen *et al.*, 1993) is used to treat a variety of bacterial infections. This medication belongs to a class of drugs called quinolone antibiotics. The title compound is a key intermediate to synthesize it. As part of our studies in this area, we report here its crystal structure.

In the title compound, all bond lengths and angles show normal values. The dihedral angle between the heterocycle and benzyl group is 45.8 (5)° (Fig.1), similar to that found in a related strcture (Garduño-Beltrán *et al.*, 2009). There is a intermolecular C—H···N hydrogen bond (Table 1) in the crystal structure.

### S2. Experimental

Benzylamine (3.85 ml, 35.2 mmol) was added to a suspension of 2,3-pyridinedicarboxylic anhydride (5 g, 33.5 mmol) in acetic acid (50 ml), and the mixture was heated under reflux for 18 h. It was then cooled to room temperature, concentrated *in vacuo* and the residue was triturated with diethyl ether to afford the title compound as a white solid in 57% yield. Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an acetone solution.

### S3. Refinement

All H atoms were positioned geometrically and refined using a riding model with C—H = 0.93–0.97 Å and $U_{iso}$(H) = 1.2 $U_{eq}$(C).

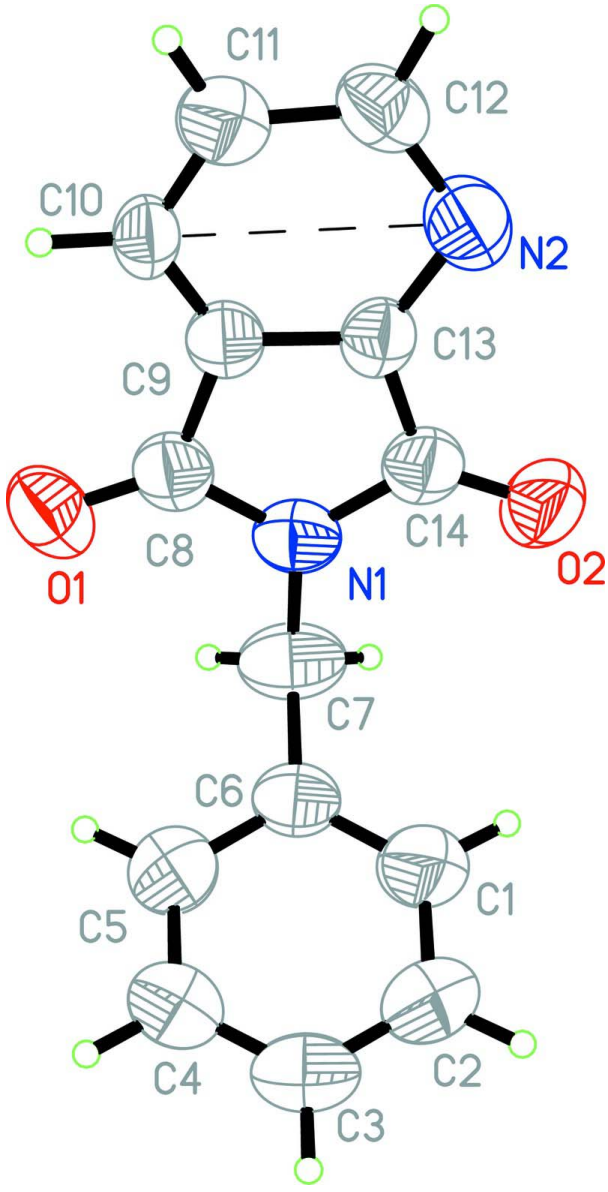


**Figure 1**

The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids at the 50% probability level.

### 6-Benzyl-6,7-dihydro-5*H*-pyrrolo[3,4-*b*]pyridine-5,7-dione

*Crystal data*

$C_{14}H_{10}N_2O_2$
$M_r = 238.24$
Monoclinic, $P2_1/c$
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$a = 11.8548$ (6) Å
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$\beta = 107.45$ (3)°
$V = 1145.1$ (2) Å$^3$
$Z = 4$
$F(000) = 496$
$D_x = 1.382$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–13°
$\mu = 0.10$ mm$^{-1}$

$T$ = 293 K
Block, colorless

0.20 × 0.10 × 0.10 mm

*Data collection*

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Radiation source: fine-focus sealed tube
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$\omega/2\theta$ scans
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.981, $T_{max}$ = 0.991
2087 measured reflections

2087 independent reflections
1100 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.045
$\theta_{max}$ = 25.4°, $\theta_{min}$ = 1.8°
$h$ = −14→13
$k$ = 0→14
$l$ = 0→9
3 standard reflections every 200 reflections
intensity decay: 1%

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.174
$S$ = 1.00
2087 reflections
163 parameters
12 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.082P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.13 e Å$^{-3}$

*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 $F^2 > \sigma(F^2)$ 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 (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.2267 (2) | 0.6223 (2) | 0.0923 (3) | 0.0602 (7) |
| O1 | 0.20765 (18) | 0.43903 (17) | 0.1329 (3) | 0.0753 (7) |
| C1 | 0.4977 (3) | 0.7166 (3) | 0.2368 (5) | 0.0896 (11) |
| H1A | 0.4640 | 0.7810 | 0.1871 | 0.108* |
| N2 | −0.0141 (2) | 0.74370 (19) | 0.2194 (3) | 0.0711 (7) |
| O2 | 0.19962 (18) | 0.80705 (17) | 0.0885 (3) | 0.0782 (7) |
| C2 | 0.6005 (3) | 0.7199 (3) | 0.3733 (5) | 0.0957 (12) |
| H2B | 0.6327 | 0.7861 | 0.4170 | 0.115* |
| C3 | 0.6539 (3) | 0.6287 (3) | 0.4429 (4) | 0.0763 (9) |
| H3A | 0.7242 | 0.6307 | 0.5324 | 0.092* |
| C4 | 0.6038 (3) | 0.5337 (3) | 0.3807 (5) | 0.0980 (13) |
| H4A | 0.6398 | 0.4698 | 0.4289 | 0.118* |
| C5 | 0.5005 (3) | 0.5297 (3) | 0.2473 (5) | 0.0919 (12) |

| | | | | |
|---|---|---|---|---|
| H5A | 0.4681 | 0.4630 | 0.2066 | 0.110* |
| C6 | 0.4446 (2) | 0.6219 (2) | 0.1734 (3) | 0.0577 (8) |
| C7 | 0.3308 (3) | 0.6186 (3) | 0.0307 (4) | 0.0758 (10) |
| H7A | 0.3282 | 0.6793 | −0.0454 | 0.091* |
| H7B | 0.3282 | 0.5530 | −0.0351 | 0.091* |
| C8 | 0.1751 (2) | 0.5306 (2) | 0.1379 (3) | 0.0570 (8) |
| C9 | 0.0735 (2) | 0.5705 (2) | 0.1955 (3) | 0.0505 (6) |
| C10 | −0.0010 (2) | 0.5161 (2) | 0.2533 (3) | 0.0493 (7) |
| H10A | 0.0040 | 0.4415 | 0.2653 | 0.059* |
| C11 | −0.0833 (3) | 0.5716 (2) | 0.2939 (4) | 0.0655 (8) |
| H11A | −0.1372 | 0.5342 | 0.3353 | 0.079* |
| C12 | −0.0940 (3) | 0.6819 (2) | 0.2785 (3) | 0.0633 (8) |
| H12A | −0.1550 | 0.7164 | 0.3075 | 0.076* |
| C13 | 0.0710 (2) | 0.6813 (2) | 0.1768 (3) | 0.0504 (6) |
| C14 | 0.1696 (3) | 0.7163 (2) | 0.1142 (3) | 0.0581 (8) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0490 (14) | 0.0711 (16) | 0.0568 (14) | 0.0019 (13) | 0.0100 (11) | 0.0026 (13) |
| O1 | 0.0755 (15) | 0.0661 (14) | 0.0803 (15) | 0.0207 (12) | 0.0173 (12) | −0.0042 (11) |
| C1 | 0.063 (2) | 0.075 (2) | 0.118 (3) | −0.0018 (18) | 0.007 (2) | 0.021 (2) |
| N2 | 0.0711 (18) | 0.0628 (14) | 0.0755 (17) | 0.0089 (11) | 0.0162 (13) | −0.0014 (12) |
| O2 | 0.0702 (15) | 0.0627 (13) | 0.0928 (16) | −0.0110 (11) | 0.0108 (12) | 0.0161 (11) |
| C2 | 0.067 (2) | 0.084 (3) | 0.120 (3) | −0.014 (2) | 0.003 (2) | −0.005 (2) |
| C3 | 0.0509 (18) | 0.101 (3) | 0.076 (2) | −0.001 (2) | 0.0185 (16) | 0.001 (2) |
| C4 | 0.068 (2) | 0.079 (3) | 0.129 (3) | 0.009 (2) | 0.003 (2) | 0.014 (2) |
| C5 | 0.073 (3) | 0.072 (2) | 0.113 (3) | −0.0044 (19) | 0.000 (2) | −0.010 (2) |
| C6 | 0.0490 (16) | 0.071 (2) | 0.0572 (16) | 0.0014 (16) | 0.0227 (14) | 0.0005 (16) |
| C7 | 0.0562 (19) | 0.114 (3) | 0.0586 (18) | −0.0033 (18) | 0.0195 (16) | 0.0010 (18) |
| C8 | 0.0532 (18) | 0.0573 (19) | 0.0503 (17) | 0.0024 (15) | −0.0002 (13) | −0.0016 (13) |
| C9 | 0.0483 (15) | 0.0511 (12) | 0.0446 (15) | 0.0002 (12) | 0.0027 (12) | −0.0005 (12) |
| C10 | 0.0542 (17) | 0.0359 (12) | 0.0503 (15) | 0.0001 (10) | 0.0040 (12) | 0.0023 (11) |
| C11 | 0.0599 (18) | 0.0689 (14) | 0.0665 (19) | 0.0027 (14) | 0.0171 (15) | 0.0000 (16) |
| C12 | 0.0601 (19) | 0.0673 (15) | 0.0618 (18) | 0.0126 (14) | 0.0171 (14) | −0.0092 (16) |
| C13 | 0.0525 (15) | 0.0481 (12) | 0.0442 (14) | −0.0032 (12) | 0.0049 (12) | 0.0002 (12) |
| C14 | 0.0516 (18) | 0.060 (2) | 0.0516 (17) | −0.0023 (15) | −0.0014 (13) | 0.0026 (14) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C14 | 1.385 (4) | C4—H4A | 0.9300 |
| N1—C8 | 1.394 (3) | C5—C6 | 1.367 (4) |
| N1—C7 | 1.467 (4) | C5—H5A | 0.9300 |
| O1—C8 | 1.203 (3) | C6—C7 | 1.496 (4) |
| C1—C6 | 1.359 (4) | C7—H7A | 0.9700 |
| C1—C2 | 1.384 (5) | C7—H7B | 0.9700 |
| C1—H1A | 0.9300 | C8—C9 | 1.503 (4) |
| N2—C13 | 1.395 (3) | C9—C10 | 1.306 (3) |

| | | | |
|---|---|---|---|
| N2—C12 | 1.411 (4) | C9—C13 | 1.382 (4) |
| O2—C14 | 1.217 (3) | C10—C11 | 1.315 (4) |
| C2—C3 | 1.336 (4) | C10—H10A | 0.9300 |
| C2—H2B | 0.9300 | C11—C12 | 1.376 (4) |
| C3—C4 | 1.347 (4) | C11—H11A | 0.9300 |
| C3—H3A | 0.9300 | C12—H12A | 0.9300 |
| C4—C5 | 1.374 (4) | C13—C14 | 1.474 (4) |

| | | | |
|---|---|---|---|
| C14—N1—C8 | 112.4 (2) | N1—C7—H7B | 109.0 |
| C14—N1—C7 | 124.4 (3) | C6—C7—H7B | 109.0 |
| C8—N1—C7 | 123.2 (3) | H7A—C7—H7B | 107.8 |
| C6—C1—C2 | 121.9 (3) | O1—C8—N1 | 126.2 (3) |
| C6—C1—H1A | 119.1 | O1—C8—C9 | 128.0 (3) |
| C2—C1—H1A | 119.1 | N1—C8—C9 | 105.8 (2) |
| C13—N2—C12 | 113.2 (2) | C10—C9—C13 | 124.0 (3) |
| C3—C2—C1 | 120.5 (3) | C10—C9—C8 | 129.5 (3) |
| C3—C2—H2B | 119.7 | C13—C9—C8 | 106.5 (3) |
| C1—C2—H2B | 119.7 | C9—C10—C11 | 117.1 (3) |
| C2—C3—C4 | 118.7 (3) | C9—C10—H10A | 121.5 |
| C2—C3—H3A | 120.7 | C11—C10—H10A | 121.5 |
| C4—C3—H3A | 120.7 | C10—C11—C12 | 123.4 (3) |
| C3—C4—C5 | 121.2 (3) | C10—C11—H11A | 118.3 |
| C3—C4—H4A | 119.4 | C12—C11—H11A | 118.3 |
| C5—C4—H4A | 119.4 | C11—C12—N2 | 121.3 (3) |
| C6—C5—C4 | 121.2 (3) | C11—C12—H12A | 119.4 |
| C6—C5—H5A | 119.4 | N2—C12—H12A | 119.4 |
| C4—C5—H5A | 119.4 | C9—C13—N2 | 121.1 (3) |
| C1—C6—C5 | 116.5 (3) | C9—C13—C14 | 109.8 (3) |
| C1—C6—C7 | 121.8 (3) | N2—C13—C14 | 129.1 (2) |
| C5—C6—C7 | 121.7 (3) | O2—C14—N1 | 125.2 (3) |
| N1—C7—C6 | 112.7 (2) | O2—C14—C13 | 129.4 (3) |
| N1—C7—H7A | 109.0 | N1—C14—C13 | 105.4 (2) |
| C6—C7—H7A | 109.0 | | |

| | | | |
|---|---|---|---|
| C6—C1—C2—C3 | 2.8 (6) | C13—C9—C10—C11 | 0.7 (4) |
| C1—C2—C3—C4 | −1.9 (6) | C8—C9—C10—C11 | −179.4 (2) |
| C2—C3—C4—C5 | 0.6 (6) | C9—C10—C11—C12 | −0.1 (4) |
| C3—C4—C5—C6 | −0.3 (6) | C10—C11—C12—N2 | −1.1 (4) |
| C2—C1—C6—C5 | −2.4 (5) | C13—N2—C12—C11 | 1.4 (4) |
| C2—C1—C6—C7 | 177.4 (3) | C10—C9—C13—N2 | −0.2 (4) |
| C4—C5—C6—C1 | 1.1 (5) | C8—C9—C13—N2 | 179.9 (2) |
| C4—C5—C6—C7 | −178.7 (3) | C10—C9—C13—C14 | 178.1 (2) |
| C14—N1—C7—C6 | 91.4 (3) | C8—C9—C13—C14 | −1.8 (3) |
| C8—N1—C7—C6 | −87.6 (3) | C12—N2—C13—C9 | −0.8 (4) |
| C1—C6—C7—N1 | −88.5 (4) | C12—N2—C13—C14 | −178.8 (2) |
| C5—C6—C7—N1 | 91.3 (4) | C8—N1—C14—O2 | 177.7 (3) |
| C14—N1—C8—O1 | −179.2 (3) | C7—N1—C14—O2 | −1.4 (4) |
| C7—N1—C8—O1 | −0.1 (4) | C8—N1—C14—C13 | −1.1 (3) |

| | | | |
|---|---|---|---|
| C14—N1—C8—C9 | 0.0 (3) | C7—N1—C14—C13 | 179.8 (2) |
| C7—N1—C8—C9 | 179.2 (2) | C9—C13—C14—O2 | −176.9 (3) |
| O1—C8—C9—C10 | 0.5 (5) | N2—C13—C14—O2 | 1.2 (5) |
| N1—C8—C9—C10 | −178.7 (3) | C9—C13—C14—N1 | 1.8 (3) |
| O1—C8—C9—C13 | −179.6 (3) | N2—C13—C14—N1 | 179.9 (2) |
| N1—C8—C9—C13 | 1.1 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C10—H10*A*···N2$^{i}$ | 0.93 | 2.46 | 3.386 (3) | 177 |

Symmetry code: (i) −*x*, *y*−1/2, −*z*+1/2.