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The crystal structure of 7-bromo-1-cyclopropyl-8-methyl-4-oxo-1,4-dihydroquinoline-3-carboxylic acid an intermediate of the ozenoxacin synthesis, $C_{14}H_{12}BrNO_3$

<https://doi.org/10.1515/ncrs-2017-0430>

Received January 4, 2018; accepted March 27, 2018; available online April 18, 2018

Abstract

$C_{14}H_{12}BrNO_3$, triclinic, $P\bar{1}$ (no. 2), $a = 8.4134(5)$ Å, $b = 9.5365(6)$ Å, $c = 9.6154(7)$ Å, $\alpha = 60.728(2)^\circ$, $\beta = 83.130(2)^\circ$, $\gamma = 66.331(2)^\circ$, $V = 613.52(7)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0393$, $wR_{ref}(F^2) = 0.0709$, $T = 296(2)$ K.

CCDC no.: 1832868

The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The compound we reported here, 7-bromo-1-cyclopropyl-8-methyl-4-oxo-1,4-dihydroquinoline-3-carboxylic acid, can be

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	$0.26 \times 0.21 \times 0.16$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	3.35 mm^{-1}
Diffractometer, scan mode:	PHOTON 100 CMOS, ω -scans
$\theta_{\max}$, completeness:	26.4° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	13541, 2481, 0.069
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1854
$N(\text{param})_{\text{refined}}$:	176
Programs:	Bruker programs [1], SHELX [2]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.14206(5)	1.02473(4)	0.15304(4)	0.04939(14)
C1	0.2259(4)	0.4913(3)	0.9178(3)	0.0280(6)
H1	0.2088	0.3910	0.9922	0.034*
C2	0.2661(3)	0.5808(3)	0.9744(3)	0.0278(6)
C3	0.3101(4)	0.7248(4)	0.8656(3)	0.0289(7)
C4	0.2831(3)	0.7821(3)	0.6958(3)	0.0268(6)
C5	0.2181(3)	0.6979(3)	0.6454(3)	0.0257(6)
C6	0.1596(3)	0.7751(3)	0.4819(3)	0.0272(6)
C7	0.1994(4)	0.9173(4)	0.3769(3)	0.0323(7)
C8	0.2778(4)	0.9933(4)	0.4227(4)	0.0389(8)
H8	0.3054	1.0862	0.3465	0.047*
C9	0.3136(4)	0.9286(4)	0.5822(4)	0.0349(7)
H9	0.3589	0.9829	0.6155	0.042*
C10	0.2114(4)	0.4070(4)	0.7235(3)	0.0300(7)
H10	0.0977	0.4155	0.6974	0.036*
C11	0.3573(4)	0.3455(4)	0.6366(4)	0.0379(7)
H11A	0.4448	0.3946	0.6099	0.046*
H11B	0.3320	0.3194	0.5582	0.046*
C12	0.3505(4)	0.2272(4)	0.8082(4)	0.0405(8)
H12A	0.4337	0.2046	0.8860	0.049*
H12B	0.3208	0.1293	0.8342	0.049*
C13	0.2746(4)	0.5161(4)	1.1494(4)	0.0336(7)
C14	0.0465(4)	0.7212(4)	0.4252(4)	0.0379(8)
H14A	−0.0263	0.8200	0.3307	0.057*
H14B	−0.0252	0.6793	0.5077	0.057*
H14C	0.1188	0.6298	0.4011	0.057*
N1	0.2095(3)	0.5382(3)	0.7629(3)	0.0263(5)
O1	0.2275(3)	0.4029(3)	1.2444(3)	0.0493(6)
O2	0.3394(3)	0.5939(3)	1.1972(3)	0.0467(6)
H2A	0.352(5)	0.678(5)	1.112(4)	0.056*
O3	0.3649(3)	0.8020(3)	0.9109(3)	0.0422(5)

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easily synthesized in accord with a known patent [3]. Evaporation at room temperature yielded colorless crystals of 7-bromo-1-cyclopropyl-8-methyl-4-oxo-1,4-dihydroquinoline-3-carboxylic acid.

Experimental details

The hydrogen atoms were placed on calculated positions with the help of the SHELX program (AFIX 43, 0, 13, 23 or 137 option). All H atoms were positioned geometrically and refined using a riding model: O—H = 0.87 Å, C—H = 0.93 and 0.96 Å for aromatic and CH₃ H atoms, respectively, with $U_{\text{iso}}(\text{H}) = k \times U_{\text{eq}}(\text{N}, \text{C})$, where $k = 1.5$ for CH₃ H atoms and $= 1.2$ for other H atoms.

Discussion

The title compound, we report here, is an important intermediate of the ozenoxacin synthesis. Ozenoxacin is an experimental quinolone antibiotic being developed for the treatment of impetigo and other dermatological bacterial infections. Now ozenoxacin has been approved in USA by FDA for the topical treatment of impetigo. Ozenoxacin is active against some bacteria that have developed resistance to currently used quinolone and fluoroquinolone antibiotics. The molecular structure of the title compound is shown in the figure, the bond lengths and angles are within normal ranges [4].

The crystal structure analysis of the title compound revealed that there are three rings, ring A (C4—C5—C6—C7—C8—C9), ring B (N1—C1—C2—C3—C4—C5) and ring C (C10—C11—C12). The dihedral angles between ring A and ring B is 8.8(2)°. The dihedral angles between the ring A and ring C is 82.3(3)°. The dihedral angles between the ring C and ring B is 74.4(2)°. In the crystal of the title compound there is one O—H···O intramolecular hydrogen bond (*cf.* the figure). Meanwhile, molecules are linked by C—H···O intermolecular hydrogen bonds to form a two-dimensional network.

Acknowledgements: This work is supported by the Qing Lan Project Foundation of Jiangsu Province.

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