

## 4-Hydroxy-1-methyl-2-oxo-N-(4-oxo-2-propyl-3,4-dihydroquinazolin-3-yl)-1,2-dihydroquinoline-3-carboxamide

S. V. Shishkina,<sup>a\*</sup> O. V. Shishkin,<sup>a</sup> I. V. Ukrainets,<sup>b</sup> N. A. Jaradat<sup>b</sup> and O. V. Gorokhova<sup>b</sup>

<sup>a</sup>Scientific Research Department of Alkali Halide Crystals, STC 'Institute for Single Crystals', National Academy of Sciences of Ukraine, 60 Lenina Ave., Khar'kov 310001, Ukraine, and <sup>b</sup>Department of Organic Chemistry, Ukrainian Pharmaceutical Academy, 21 Blyukhera Ave., Khar'kov 310002, Ukraine  
Correspondence e-mail: sveta@xray.isc.kharkov.com

Received 28 February 2000

Accepted 10 March 2000

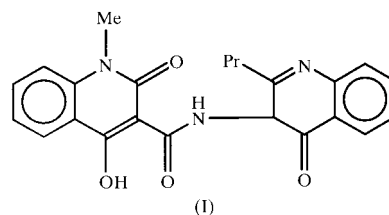
Data validation number: IUC0000074

The two bicyclic fragments of the title compound, C<sub>22</sub>H<sub>20</sub>N<sub>4</sub>O<sub>4</sub>, are individually planar and are turned with respect to each other by 77.8 (2)°. The formation of intramolecular O—H···O and N—H···O hydrogen bonds causes considerable changes in the bond lengths within the amidopyridine fragment.

### Comment

In the present paper, we report the results of an investigation of the molecular and crystal structures of the (2-propyl-4-oxoquinazolin-3-yl)amide of 1-methyl-4-hydroxy-2-oxo-1,2-dihydroquinoline-3-carboxylic acid, (I), which may be used as efficient anti-inflammatory remedies (Ukrainets *et al.*, 1993, 1994). Both bicyclic fragments of the molecule are planar. The amide group O3=C10—N2 lies in the plane of the pyridine ring [the C1—C2—C10—N2 torsion angle is 2.8 (2)°]. This is caused by formation of O2—H2O···O3 and N2—H2N···O1 hydrogen bonds [H2O···O3 1.73 (4) Å, O2—H2O···O3 151 (4)°; H2N···O1 1.83 (3) Å, N2—H2N···O1 140 (2)°]. Two planar fragments are turned with respect to each other [the C10—N2—N3—C18 torsion angle is -77.8 (2)°]. The propyl substituent at the C11 atom and the C11—N4 bond have an *sp* orientation [the N4—C11—C20—C21 torsion angle is -0.4 (2)°]. Such an arrangement of the alkyl group, apparently, results from repulsion between the H atoms of the propyl substituent and the amide fragment. This assumption is confirmed by the presence of the shortened intramolecular contacts H20A···N2 2.60 Å and H20B···N2 2.61 Å (van der Waals radii sum is 2.66 Å; Zefirov & Zorky, 1995). The formation of the intramolecular hydrogen bonds causes change of bond lengths within the amidopyridine fragment. Similar changes in the bond lengths were observed in the related structure 3-benzoyl-1-ethyl-4-hydroxy-2-quinolone

(Borowiec *et al.*, 1996). This effect may be explained by some contribution of the enole resonance form into the total structure of molecule.



### Experimental

#### Crystal data

C<sub>22</sub>H<sub>20</sub>N<sub>4</sub>O<sub>4</sub>  
M<sub>r</sub> = 404.42  
Monoclinic, P2<sub>1</sub>/c  
a = 12.474 (3) Å  
b = 15.213 (4) Å  
c = 10.684 (3) Å  
β = 105.95 (3)°  
V = 1949.4 (9) Å<sup>3</sup>  
Z = 4

D<sub>x</sub> = 1.378 Mg m<sup>-3</sup>  
Mo Kα radiation  
Cell parameters from 24 reflections  
θ = 10–11°  
μ = 0.097 mm<sup>-1</sup>  
T = 293 (2) K  
Needle, yellow  
0.40 × 0.20 × 0.10 mm

#### Data collection

Siemens P3/PC diffractometer  
q/2θ scans  
5749 measured reflections  
5477 independent reflections  
3031 reflections with I > 2σ(I)  
R<sub>int</sub> = 0.016  
θ<sub>max</sub> = 30.08°

h = -17 → 16  
k = -21 → 0  
l = 0 → 15  
2 standard reflections every 98 reflections  
intensity decay: 5%

#### Refinement

Refinement on F<sup>2</sup>  
R[F<sup>2</sup> > 2σ(F<sup>2</sup>)] = 0.064  
wR(F<sup>2</sup>) = 0.190  
S = 0.955  
5477 reflections  
351 parameters  
H atoms treated by a mixture of independent and constrained refinement

w = 1/[σ<sup>2</sup>(F<sub>o</sub><sup>2</sup>) + (0.1328P)<sup>2</sup>]  
where P = (F<sub>o</sub><sup>2</sup> + 2F<sub>c</sub><sup>2</sup>)/3  
(Δ/σ)<sub>max</sub> < 0.001  
Δρ<sub>max</sub> = 0.30 e Å<sup>-3</sup>  
Δρ<sub>min</sub> = -0.21 e Å<sup>-3</sup>

Table 1

Selected geometric parameters (Å).

O1—C1	1.244 (2)	C1—C2	1.448 (2)
O2—C3	1.326 (2)	C2—C3	1.382 (2)
O3—C10	1.2385 (19)	C3—C4	1.435 (2)

Table 2

Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2O···O3	0.83 (4)	1.72 (4)	2.500 (2)	155 (3)
N2—H2N···O1	0.90 (3)	1.82 (3)	2.567 (2)	139 (2)

All H atoms were located and their positional parameters were allowed to refine. The C—H distances are in the range 0.90 (2)–1.06 (5) Å, and N—H = 0.90 (3) Å and O—H = 0.83 (3) Å

Data collection: *P3/PC* (Siemens, 1989); cell refinement: *P3/PC*; data reduction: *XDISK* (Siemens, 1991); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXL97*; software used to prepare material for publication: *SHELXL97*.

## References

- Borowiec, H., Grochowski, J. & Serda, P. (1996). *Khim. Geterosikl. Soedin.* pp. 248–251. (In Russian.)
- Sheldrick, G. M. (1990). *Acta Cryst. A* **46**, 467–473.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Siemens (1989). *P3*. Siemens Analytical X-ray Instruments Inc., Karlsruhe, Germany.
- Siemens (1991). *XDISK*. Siemens Analytical X-ray Instruments Inc., Karlsruhe, Germany.
- Ukrainets, I. V., Gorokhova, O. V., Taran, S. G. & Turov, A. V. (1994). *Khim. Geterosikl. Soedin.* pp. 1211–1213. (In Russian.)
- Ukrainets, I. V., Taran, S. G., Evtifeeva, O. A. & Turov, A. V. (1993). *Khim. Geterosikl. Soedin.* pp. 938–940. (In Russian.)
- Zefirov, Yu. V. & Zorky, P. M. (1995). *Usp. Khim.* **64**, 446–460. (In Russian.)