N-(8-Quinolyl)pyridine-2-carbox-amide

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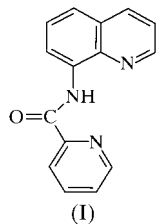Correspondence e-mail: zguo@netra.nju.edu.cn

The title compound, $C_{15}H_{11}N_3O$, is basically planar except that the pyridine ring is slightly tilted, the dihedral angle between the pyridyl and quinolyl rings being 3.55 (5)°. The crystal grows in two directions and the crystal packing is stabilized by π–π stacking interactions.
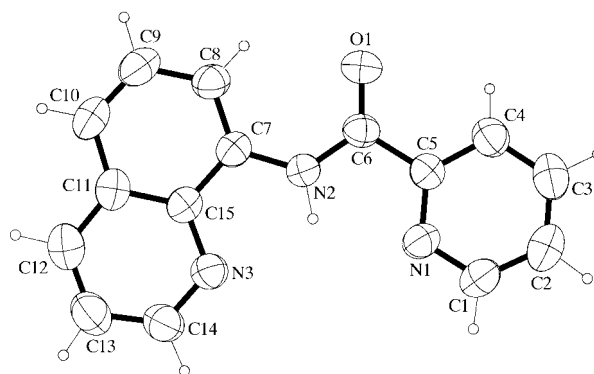
Comment

Quinoxaline derivatives, such as XK469, showed unusual solid-tumor selectivity and activity against multidrug-resistant cancer cells (Gao *et al.*, 1999). Some platinum complexes of pyridine and quinoline ligands, such as *trans*-dichlorodi-pyridineplatinum(II) and *trans*-amminedichloroquinoline-platinum(II), show comparable anticancer activity to cisplatin in cisplatin-sensitive and -resistant cell lines (Wong & Gian-domenico, 1999). Moreover, some metallo-intercalators have been widely used in DNA structural and mechanistic studies (Erkkila *et al.*, 1999). Aminoquinoline-based ligands possess a strong fluorescent property which could be used as a probe for

(I)

DNA binding (Fahrni & O'Halloran, 1999; Nasir *et al.*, 1999). Therefore, we synthesized the title compound, (I), in order to investigate the binding ability of this aminoquinoline-based ligand towards metal ions and DNA. The ligand contains pyridine, amide and quinoline N atoms which are able to coordinate to metal ions, such as Zn^{II} and Cu^{II} (Fahrni & O'Halloran, 1999; Nasir *et al.*, 1999; Amendola *et al.*, 1999). Studies of the metal complexes of (I) will be reported else-where.

The X-ray crystallographic study shows that the bond lengths and angles are within the normal ranges. The N2—C6 and N2—C7 bond distances in (I) are comparable with those in [N,N'-bis(2-pyridinecarboxamido)-1,2-benzene]copper(II) [1.337 (3) and 1.404 (2) Å; Chapman *et al.*, 1980] and N,N'-(4,5-dichloro-*o*-phenylene)bis(4-*tert*-butylpyridine-2-carbox-amide) [1.350 (4) and 1.401 (4) Å; Fun *et al.*, 1999], while the C—C and C=O bond lengths are similar to those reported in [N,N'-bis(2-pyridinecarboxamide)-1,2-benzene]nickel(II) monohydrate (Stephens & Vagg, 1986). The molecule of (I) is almost planar, except that the pyridyl ring is slightly tilted, the dihedral angle between the pyridyl and quinolyl rings being 3.55 (5)°. There are four intramolecular hydrogen bonds in the crystal (see Table 2) which could be the driving force to have N1 and N3 in the same side of the molecule.

Figure 1
The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

The crystal is a thin plate and grows in two different directions which cross each other, and the angle between these two orientations is 60.8°. There is a π–π stacking interaction between adjacent molecules packed in the same direction. The distance between two adjacent parallel aromatic rings [C1–C5/N1 and C7^i–C11^i/C15^i; symmetry code: (i) $1 - x, 2 - y, -z$] is 3.68 (2) Å, and the shortest distance is C2···C9^i of 3.481 (4) Å. This kind of interaction belongs to the face-to-face type, with a little offset, and the molecules are arranged in a head-to-tail fashion, *i.e.* the pyridyl group faces the quinoline group.

In order to understand the electron-donating ability of the three N atoms, *ab initio* calculations (HF/3-21g* method) using *GAUSSIAN*98 (Frisch *et al.*, 1998) were carried out. This gave rise to electron-distribution values of -0.731 e, -1.095 e and -0.746 e for N1, N2 and N3, respectively.

Experimental

The title compound was obtained by the reaction of one molar equivalent of pyridine-2-carboxylic acid and 8-aminoquinoline in the presence of one molar equivalent of triphenyl phosphite in pyridine at 473 K for 4 h (Leung *et al.*, 1991). Single crystals suitable for X-ray diffraction were recrystallized from pyridine and ethanol.

organic compounds

Crystal data

$C_{15}H_{11}N_3O$
$M_r = 249.27$
Monoclinic, $P2_1/n$
$a = 7.677 (2)$ Å
$b = 7.915 (3)$ Å
$c = 20.408 (5)$ Å
$\beta = 99.64 (2)°$
$V = 1222.5 (6)$ Å^3
$Z = 4$

$D_x = 1.354$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 41
    reflections
$\theta = 5.15–15.12°$
$\mu = 0.089$ mm^{-1}
$T = 293 (2)$ K
Thick plate, colorless
$0.50 \times 0.50 \times 0.40$ mm

Data collection

Siemens *P4* diffractometer
$2\theta/\omega$ scans
Absorption correction: empirical
    (North *et al.*, 1968)
    $T_{min} = 0.956$, $T_{max} = 0.965$
3082 measured reflections
2149 independent reflections
1406 reflections with $I > 2\sigma(I)$

$R_{int} = 0.026$
$\theta_{max} = 25°$
$h = -1 \rightarrow 9$
$k = -1 \rightarrow 9$
$l = -24 \rightarrow 24$
3 standard reflections
    every 97 reflections
    intensity decay: 7.12%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.122$
$S = 1.073$
2149 reflections
172 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0531P)^2 + 0.0469P]$
    where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C6	1.223 (2)	N2—C7	1.401 (2)
N1—C1	1.327 (3)	N3—C14	1.314 (2)
N1—C5	1.335 (2)	N3—C15	1.368 (2)
N2—C6	1.357 (2)	C5—C6	1.498 (3)
C1—N1—C5	116.9 (2)	C4—C5—C6	119.8 (2)
C6—N2—C7	129.3 (2)	O1—C6—N2	124.4 (2)
C14—N3—C15	117.1 (2)	O1—C6—C5	121.7 (2)
N1—C5—C6	117.0 (2)	N2—C6—C5	113.9 (2)

All H atoms were placed in geometrically calculated positions (C—H = 0.93 Å and N—H = 0.86 Å), with $U_{iso} = 1.2U_{eq}$(parent atom).

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H2B$\cdots$N1	0.86	2.21	2.653 (2)	112
N2—H2B$\cdots$N3	0.86	2.24	2.659 (2)	110
C4—H4A$\cdots$O1	0.93	2.56	2.833 (3)	97
C8—H8A$\cdots$O1	0.93	2.33	2.923 (3)	121

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