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N-(1-Adamantyl)-4-hydroxy-2-(2-methylpropyl)-2-oxo-1,2-dihydroquinoline-3-carboxamide

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Key indicators

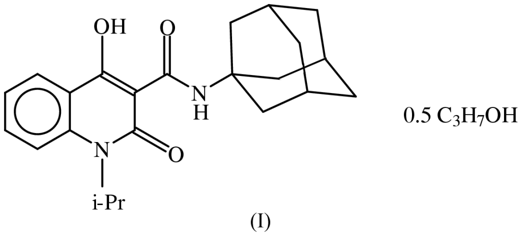
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
Disorder in solvent or counterion
R factor = 0.069
wR factor = 0.212
Data-to-parameter ratio = 23.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{24}H_{30}N_2O_3 \cdot 0.5C_3H_8O$, the formation of intramolecular O—H···O and N—H···O hydrogen bonds causes considerable changes in the bond lengths within the amidopyridine fragment. This fact probably results in the unusual reactivity of the ether group in this type of compound.

Comment

Earlier, we demonstrated that intramolecular hydrogen bonds in the ethyl ethers of 1(*R*)-4-hydroxy-2-oxo-1,2-dihydroquinoline-3-carboxylic acid result in the marked lengthening of the C═O bond of the 3-carboethoxy group (Ukrainets, Djaradat *et al.*, 2000; Ukrainets, Taran, Likhaneva, Djaradat & Shishkin, 2000; Ukrainets, Taran, Likhaneva, Ribakov *et al.*, 2000). This fact, probably, determines an unusual reactivity of these compounds. A similar situation is observed for heteryl (Shishkina *et al.*, 2000) and arylalkyl (Ukrainets, Djaradat *et al.*, 2000; Ukrainets, Taran, Likhaneva, Djaradat & Shishkin, 2000; Ukrainets, Taran, Likhaneva, Ribakov *et al.*, 2000) amides of this acid. In this paper, we report the results of the X-ray diffraction study of the adamantyl-1-amide of 1-isobutyl-4-hydroxy-2-oxo-1,2-dihydroquinoline-3-carboxylic acid, (I).



(I)

The quinolone fragment (Fig. 1) and the C10, O1, O3, C14, O2, N2 and C15 atoms are coplanar within 0.04 Å. The isopropyl group of the alkyl substituent at the N1 atom is arranged nearly orthogonal with respect to the pyridone ring [the C5—N1—C10—C11 torsion angle is −79.1 (2)°] and has a +*ac* orientation relatively to the N1—C10 bond (the N1—C10—C11—H11 torsion angle is 64.3°). There are some differences between values of the C—C bonds within isopropyl group. The C11—C12 bond [1.553 (4) Å] is longer than the C11—C13 bond [1.540 (4) Å].

The adamantane fragment is arranged with respect to the amide group in such a way that the C14—N2 and C16—C15 bonds have an *ap* orientation [the C14—N2—C15—C16 torsion angle is 178.9 (2)°]. Repulsion between the bulky adamantane substituent and the carbonyl group probably causes some elongation of the N2—C15 bond [1.489 (3) Å] with respect to its mean value 1.469 Å (Bürgi & Dunitz, 1994).

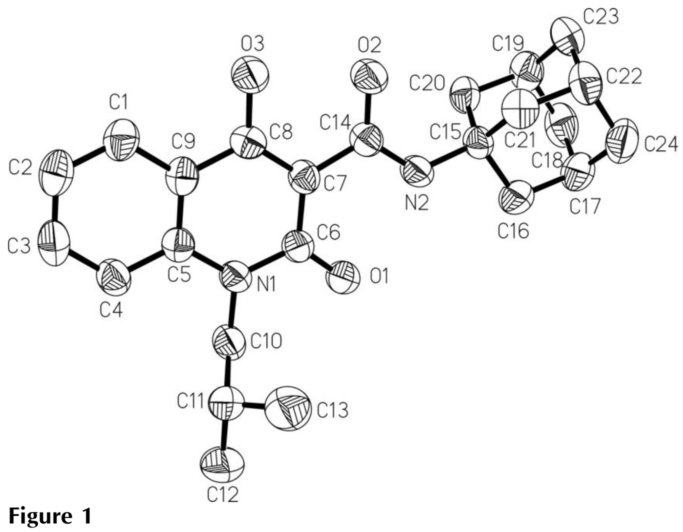


Figure 1
View of the title compound. The non-H atoms are shown with displacement ellipsoids drawn at the 50% probability level and H atoms have been omitted for clarity.

The formation of the N2—H2N$\cdots$O1 and O3—H3O$\cdots$O2 intramolecular hydrogen bonds (H2N$\cdots$O1 1.98 Å, N2—H2N$\cdots$O1 138°; H3O$\cdots$O2 1.75 Å and O3—H3O$\cdots$O2 148°) results in an elongation of the O1—C6, O2—C14, C7—C8 and C7—C14 bonds (Table 1) compared with their mean values of 1.210, 1.330 and 1.465 Å (Burgi & Dunitz, 1994), respectively.

The presence of shortened intramolecular contacts (Table 2) O1$\cdots$H10*A* = 2.29 Å (the van der Waals radii sum is 2.45 Å; Zefirov & Zorky, 1995), H10*B*$\cdots$C4 = 2.66 Å (2.87 Å), H10*B*$\cdots$H4 = 2.17 Å (2.32 Å), H11$\cdots$C4 = 2.82 Å, H11$\cdots$H4 = 2.25 Å, probably causes the elongation of the N1—C5, N1—C6 and N1—C10 bonds (mean values are 1.371, 1.355 and 1.469 Å, respectively).

In the crystal phase, compound (I) exists as the solvate with 2-propanol in a 2:1 ratio. The molecules of the solvent are disordered over two positions with equal populations with respect to the centre of symmetry.

Experimental

Crystals of (I) were grown by slow evaporation from a 2-propanol solution.

Crystal data

$C_{24}H_{30}N_2O_3{\cdot}0.5C_3H_8O$
$M_r = 424.55$
Monoclinic, $P2_1/c$
$a = 15.140\ (4)$ Å
$b = 13.655\ (3)$ Å
$c = 12.286\ (3)$ Å
$\beta = 110.51\ (3)°$
$V = 2379\ (1)$ Å^3
$Z = 4$
$D_x = 1.185$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 24 reflections
$\theta = 10–11°$
$\mu = 0.08$ mm^{-1}
$T = 293\ (2)$ K
Needle, colourless
0.40 × 0.20 × 0.20 mm

Data collection

Siemens *P*3/*PC* diffractometer
$\theta–2\theta$ scans
6801 measured reflections
6505 independent reflections
2175 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.038$
$\theta_{\rm max} = 30.1°$
$h = -21 \rightarrow 20$
$k = -19 \rightarrow 0$
$l = 0 \rightarrow 15$
2 standard reflections every 98 reflections intensity decay: 5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.212$
$S = 0.91$
6505 reflections
280 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0991P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.042$
$\Delta\rho_{\rm max} = 0.41$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.27$ e Å^{-3}

Table 1
Selected bond lengths (Å).

N1—C5	1.408 (3)	O2—C14	1.282 (3)
N1—C6	1.417 (3)	O3—C8	1.340 (2)
N1—C10	1.495 (3)	C6—C7	1.472 (3)
N2—C14	1.351 (3)	C7—C8	1.394 (3)
N2—C15	1.489 (2)	C7—C14	1.498 (3)
O1—C6	1.250 (3)	C8—C9	1.461 (3)

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

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
N2—H2N$\cdots$O1	0.86	1.98	2.687 (2)	138
O3—H3O$\cdots$O2	0.82	1.75	2.489 (2)	148

Data collection: *P*3 (Siemens, 1989); cell refinement: *P*3; data reduction: *XDISK* (Siemens, 1991); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1991); software used to prepare material for publication: *SHELXL*97.

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