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2,6-Dimethyl-4-(1,3,4-oxadiazol-2-yl)-quinoline

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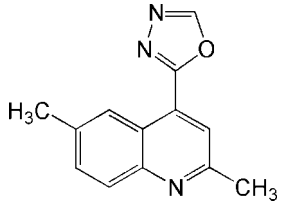
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.002 Å; R factor = 0.042; wR factor = 0.130; data-to-parameter ratio = 14.3.

The title compound, $C_{13}H_{11}N_3O$, a potential chemotherapeutic agent, contains a essential planar [maximum deviation = 0.0144 (14) Å] quinoline moiety. The quinoline ring system and the five-membered heterocycle form a dihedral angle of 7.81 (6)°. In the crystal, intermolecular non-classical C—H···N hydrogen bonding is present.

Related literature

For general background to the use of compounds containing a quinoline fragment as chemotherapeutical agents, see: Kaila *et al.* (2007); Vaitilingam *et al.* (2004).



Experimental

Crystal data

$C_{13}H_{11}N_3O$
M_r = 225.25
Monoclinic, $P2_1/c$
a = 15.5372 (14) Å
b = 9.7546 (7) Å
c = 7.3984 (5) Å
β = 100.64 (1)°
V = 1102.02 (15) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.73 mm^{-1}
T = 295 K
0.20 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: refined from ΔF (Walker & Stuart, 1983)
T_{min} = 0.391, T_{max} = 0.865
2236 measured reflections
2236 independent reflections
1855 reflections with $I > 2\sigma(I)$
1 standard reflections every 60 min
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.130
S = 1.07
2236 reflections
156 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13···N15^{i}	0.93	2.59	3.523 (2)	178

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: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors are indebted to Russian Foundation for Basic Research for covering the licence fee for use of the Cambridge Structural Database (Allen, 2002).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DS2065).

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supporting information

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2,6-Dimethyl-4-(1,3,4-oxadiazol-2-yl)quinoline

Artyom G. Kashaev, Anatoliy V. Zimichev, Victor B. Rybakov, Yurij N. Klimochkin and Margarita N. Zemtsova

S1. Comment

The compounds containing a fragment of quinoline ring, are widely used as chemotherapeutical agents (Vaitilingam *et al.*, 2004; Kaila *et al.*, 2007). Synthesis of new quinoline derivatives and study of its properties to be of interest in theoretic and practical aspects as well. The 2,6-dimethyl-4-(1,3,4-oxadiazol-2-yl)quinoline **II** was synthesized from triethyl orthoformate and hydrazide 2,6-dimethyl-4-quinoline carboxylic acid **I** (Fig. 1).

In the crystal structure is found non-classical intermolecular hydrogen bond - C13–H13···N15^{i}, where contacts H13···N15^{i} = 2.594 Å, C13···N15^{i} = 3.523 (2)Å and angle C13–H13···N15^{i} = 178°. Symmetry code:(i) -*x*, *y* + 1/2, -*z* + 1/2. The short intramolecular contacts C6–H6···N15 (H6···N15 = 2.370 Å) and C3–H3···O12 (H3···O12 = 2.372 Å) are obliged by conjugation of oxadiazol and guinoline moieties - in title molecule, guinoline moiety is planar (max deviation of C7 = 0.0144 (14) Å) and essential planar oxadiazol moiety form dihedral angle 7.81 (6)° (Fig. 2).

S2. Experimental

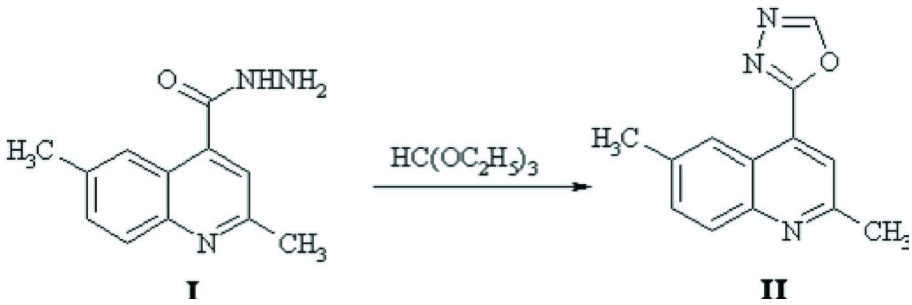
A solution of triethyl orthoformate (60 mmol) and 2,6-dimethyl-4-(1,3,4-oxadiazol-2-yl)quinoline (5 mmol) was refluxed for 20 h. Ester was removed in a vacuum. Recrystallization of the crude product from ethanol gave 0.89 g of colourless crystals. Yield 79%, mp 448-449 K.

IR, *v*, cm^{-1}: 3116 (C–H, oxadiazol), 1751 (CO), 1600 (C═C), 1508 (C═ N). MS, m/*z*: 225 (100) [*M*]$^{+}$, 210 (17), 184 (11), 156 (32), 115 (15), 89 (8), 63 (9). ^{1}H NMR, *δ*: 2.51 s (3*H*, 6-CH_3), 2.71 s (3*H*, 2-CH_3), 7.63 d (1*H*, J = 8.80, 7-H), 7.89 s (1*H*, 3-H), 7.92 d (1*H*, J = 8.80, 8-H), 8.71 s (1*H*, 5-H), 9.52 s (1*H*, C–H oxadiazol). Anal. calc. for $C_{13}H_{11}N_3O$, %: C 69.32; H 4.92; N 18.66. Found, %: C 69.27; H 4.83; N 18.61.

Single crystals for *X*-ray analysis were obtained by slow evaporation of an ethanol. IR spectrum was recorded (in KBr) on Shimadzu FTIR-8400S. Mass spectrum was measured on Finnigan Trance DSQ spectrometer. ^{1}H NMR spectrum was obtained in *DMSO*-d_6 on Bruker AM 300 (300 MHz), using *TMS* as internal standard. Elemental composition was determined on Euro Vector EA-3000 elemental analyzer.

S3. Refinement

C-bound H-atoms were placed in calculated positions (C–H 0.93 Å & 0.97 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C).



I II

Figure 1

Synthesis of the title compound.

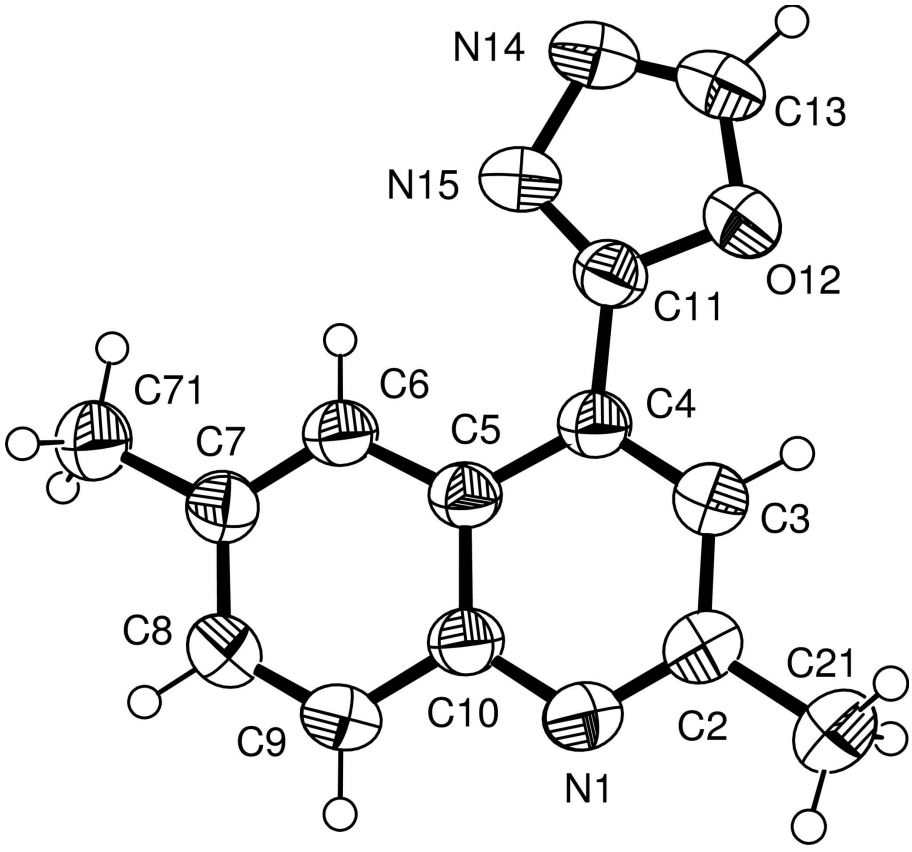


Figure 2

ORTEP-3 (Farrugia, 1997) plot of molecular structure of the title compound showing the atom-numbering scheme. Thermal displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

2,6-Dimethyl-4-(1,3,4-oxadiazol-2-yl)quinoline

Crystal data

$C_{13}H_{11}N_3O$
$M_r = 225.25$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 15.5372\ (14)$ Å
$b = 9.7546\ (7)$ Å
$c = 7.3984\ (5)$ Å
$\beta = 100.64\ (1)°$
$V = 1102.02\ (15)$ Å^3
$Z = 4$

$F(000) = 472$
$D_x = 1.358$ Mg m^{-3}
Melting point = 448–449 K
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 25 reflections
$\theta = 36.3$–$39.9°$
$\mu = 0.73$ mm^{-1}
$T = 295$ K
Prism, colourless
0.20 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: Fine-focus sealed tube
Graphite monochromator
Non–profiled ω scans
Absorption correction: part of the refinement model (ΔF) (Walker & Stuart, 1983)
$T_{min} = 0.391$, $T_{max} = 0.865$
2236 measured reflections
2236 independent reflections
1855 reflections with $I > 2\sigma(I)$
$R_{int} = 0.000$
$\theta_{max} = 74.9°$, $\theta_{min} = 2.9°$
$h = 0 \rightarrow 19$
$k = 0 \rightarrow 12$
$l = -8 \rightarrow 9$
1 standard reflections every 60 min
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.130$
$S = 1.07$
2236 reflections
156 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0669P)^2 + 0.1662P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.005$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.39891 (8)	0.63577 (13)	0.05252 (18)	0.0557 (3)
C2	0.36926 (10)	0.75870 (16)	0.0823 (2)	0.0549 (4)
C21	0.42870 (12)	0.87852 (19)	0.0735 (3)	0.0720 (5)
H21A	0.4875	0.8464	0.0781	0.108*
H21B	0.4271	0.9383	0.1758	0.108*
H21C	0.4096	0.9276	−0.0393	0.108*
C3	0.28466 (10)	0.77894 (16)	0.1206 (2)	0.0537 (4)
H3	0.2663	0.8673	0.1417	0.064*
C4	0.22912 (9)	0.67122 (14)	0.12717 (19)	0.0479 (3)
C5	0.25819 (9)	0.53629 (15)	0.09414 (18)	0.0468 (3)

C6	0.20838 (9)	0.41454 (15)	0.09503 (19)	0.0505 (3)
H6	0.1513	0.4206	0.1158	0.061*
C7	0.24197 (10)	0.28864 (15)	0.0662 (2)	0.0532 (4)
C71	0.19028 (12)	0.15983 (16)	0.0748 (3)	0.0656 (4)
H71A	0.1315	0.1829	0.0868	0.098*
H71B	0.2171	0.1062	0.1789	0.098*
H71C	0.1891	0.1078	−0.0358	0.098*
C8	0.32817 (11)	0.28112 (16)	0.0302 (2)	0.0599 (4)
H8	0.3515	0.1959	0.0097	0.072*
C9	0.37777 (10)	0.39518 (17)	0.0249 (2)	0.0587 (4)
H9	0.4340	0.3871	−0.0003	0.070*
C10	0.34470 (9)	0.52589 (15)	0.0574 (2)	0.0508 (3)
C11	0.14273 (9)	0.69987 (15)	0.1721 (2)	0.0500 (3)
O12	0.11965 (7)	0.83379 (11)	0.18110 (16)	0.0631 (3)
C13	0.03932 (11)	0.8249 (2)	0.2255 (3)	0.0688 (5)
H13	0.0057	0.9013	0.2419	0.083*
N14	0.01403 (9)	0.70333 (17)	0.2429 (2)	0.0739 (4)
N15	0.08253 (9)	0.61910 (15)	0.2072 (2)	0.0651 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0469 (6)	0.0603 (8)	0.0626 (8)	−0.0012 (5)	0.0168 (5)	0.0026 (6)
C2	0.0506 (8)	0.0585 (9)	0.0568 (8)	−0.0052 (6)	0.0135 (6)	0.0015 (6)
C21	0.0661 (10)	0.0651 (10)	0.0887 (13)	−0.0142 (8)	0.0241 (9)	0.0008 (9)
C3	0.0542 (8)	0.0518 (8)	0.0570 (9)	0.0007 (6)	0.0150 (6)	−0.0010 (6)
C4	0.0464 (7)	0.0516 (8)	0.0468 (8)	0.0019 (6)	0.0117 (6)	0.0008 (6)
C5	0.0456 (7)	0.0520 (8)	0.0442 (7)	0.0029 (6)	0.0119 (5)	0.0018 (5)
C6	0.0472 (7)	0.0558 (8)	0.0508 (8)	0.0007 (6)	0.0150 (6)	0.0008 (6)
C7	0.0561 (8)	0.0517 (8)	0.0532 (8)	0.0012 (6)	0.0134 (6)	0.0002 (6)
C71	0.0701 (10)	0.0550 (9)	0.0745 (11)	−0.0045 (8)	0.0207 (8)	−0.0047 (7)
C8	0.0602 (9)	0.0530 (8)	0.0695 (10)	0.0098 (7)	0.0195 (7)	0.0008 (7)
C9	0.0491 (8)	0.0616 (9)	0.0693 (10)	0.0082 (7)	0.0209 (7)	0.0009 (7)
C10	0.0463 (7)	0.0557 (8)	0.0522 (8)	0.0019 (6)	0.0142 (6)	0.0033 (6)
C11	0.0501 (7)	0.0510 (8)	0.0506 (8)	0.0051 (6)	0.0138 (6)	−0.0015 (6)
O12	0.0570 (6)	0.0550 (6)	0.0804 (8)	0.0078 (5)	0.0207 (5)	−0.0039 (5)
C13	0.0575 (9)	0.0735 (11)	0.0795 (12)	0.0173 (8)	0.0235 (8)	−0.0056 (9)
N14	0.0573 (8)	0.0778 (10)	0.0940 (11)	0.0120 (7)	0.0337 (7)	0.0009 (8)
N15	0.0538 (7)	0.0647 (8)	0.0840 (10)	0.0053 (6)	0.0315 (7)	0.0014 (7)

Geometric parameters (Å, º)

N1—C2	1.3177 (19)	C7—C8	1.415 (2)
N1—C10	1.3677 (18)	C7—C71	1.499 (2)
C2—C3	1.408 (2)	C71—H71A	0.9600
C2—C21	1.499 (2)	C71—H71B	0.9600
C21—H21A	0.9600	C71—H71C	0.9600
C21—H21B	0.9600	C8—C9	1.358 (2)

C21—H21C	0.9600	C8—H8	0.9300
C3—C4	1.366 (2)	C9—C10	1.412 (2)
C3—H3	0.9300	C9—H9	0.9300
C4—C5	1.4271 (19)	C11—N15	1.286 (2)
C4—C11	1.4680 (19)	C11—O12	1.3596 (17)
C5—C6	1.4182 (19)	O12—C13	1.3508 (19)
C5—C10	1.4233 (19)	C13—N14	1.263 (2)
C6—C7	1.366 (2)	C13—H13	0.9300
C6—H6	0.9300	N14—N15	1.4075 (18)

C2—N1—C10	118.21 (12)	C7—C71—H71A	109.5
N1—C2—C3	121.95 (14)	C7—C71—H71B	109.5
N1—C2—C21	117.71 (14)	H71A—C71—H71B	109.5
C3—C2—C21	120.33 (14)	C7—C71—H71C	109.5
C2—C21—H21A	109.5	H71A—C71—H71C	109.5
C2—C21—H21B	109.5	H71B—C71—H71C	109.5
H21A—C21—H21B	109.5	C9—C8—C7	121.66 (14)
C2—C21—H21C	109.5	C9—C8—H8	119.2
H21A—C21—H21C	109.5	C7—C8—H8	119.2
H21B—C21—H21C	109.5	C8—C9—C10	120.58 (13)
C4—C3—C2	121.23 (14)	C8—C9—H9	119.7
C4—C3—H3	119.4	C10—C9—H9	119.7
C2—C3—H3	119.4	N1—C10—C9	117.26 (13)
C3—C4—C5	118.75 (13)	N1—C10—C5	123.86 (13)
C3—C4—C11	118.15 (13)	C9—C10—C5	118.88 (13)
C5—C4—C11	123.09 (12)	N15—C11—O12	111.76 (13)
C6—C5—C10	118.46 (13)	N15—C11—C4	131.21 (14)
C6—C5—C4	125.54 (12)	O12—C11—C4	117.03 (12)
C10—C5—C4	116.00 (13)	C13—O12—C11	102.38 (12)
C7—C6—C5	121.84 (13)	N14—C13—O12	113.79 (14)
C7—C6—H6	119.1	N14—C13—H13	123.1
C5—C6—H6	119.1	O12—C13—H13	123.1
C6—C7—C8	118.56 (14)	C13—N14—N15	105.60 (13)
C6—C7—C71	121.59 (13)	C11—N15—N14	106.47 (14)
C8—C7—C71	119.84 (14)		

C10—N1—C2—C3	0.7 (2)	C2—N1—C10—C5	−0.4 (2)
C10—N1—C2—C21	−178.99 (14)	C8—C9—C10—N1	179.04 (14)
N1—C2—C3—C4	−0.4 (2)	C8—C9—C10—C5	−0.6 (2)
C21—C2—C3—C4	179.24 (15)	C6—C5—C10—N1	179.88 (13)
C2—C3—C4—C5	−0.1 (2)	C4—C5—C10—N1	−0.1 (2)
C2—C3—C4—C11	178.58 (13)	C6—C5—C10—C9	−0.5 (2)
C3—C4—C5—C6	−179.61 (13)	C4—C5—C10—C9	179.53 (13)
C11—C4—C5—C6	1.8 (2)	C3—C4—C11—N15	−171.28 (16)
C3—C4—C5—C10	0.3 (2)	C5—C4—C11—N15	7.4 (3)
C11—C4—C5—C10	−178.28 (13)	C3—C4—C11—O12	8.1 (2)
C10—C5—C6—C7	1.6 (2)	C5—C4—C11—O12	−173.22 (12)
C4—C5—C6—C7	−178.43 (14)	N15—C11—O12—C13	0.07 (17)

C5—C6—C7—C8	−1.6 (2)	C4—C11—O12—C13	−179.46 (13)
C5—C6—C7—C71	177.53 (14)	C11—O12—C13—N14	0.0 (2)
C6—C7—C8—C9	0.4 (2)	O12—C13—N14—N15	−0.1 (2)
C71—C7—C8—C9	−178.70 (15)	O12—C11—N15—N14	−0.13 (18)
C7—C8—C9—C10	0.7 (3)	C4—C11—N15—N14	179.32 (15)
C2—N1—C10—C9	179.94 (13)	C13—N14—N15—C11	0.14 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13···N15^{i}	0.93	2.59	3.523 (2)	178

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