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N-(4-Bromophenacyl)-4,6-dimethyl-2-oxo-1,2-dihydropyridine-2-carbonitrile

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Key indicators

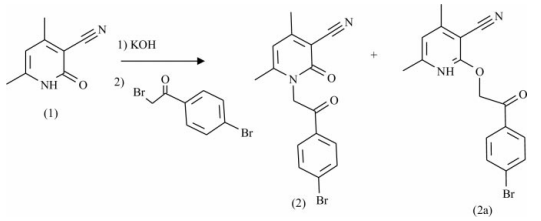
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.039
wR factor = 0.107
Data-to-parameter ratio = 15.7

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

The title compound, $C_{16}H_{13}BrN_2O_2$, was synthesized and characterized by ^{1}H NMR and X-ray diffraction techniques.

Comment

We have described earlier the crystal structure of 4,6-dimethyl-2-oxo-1,2-dihydro-pyridin-3-carbonitrile, (1) (Rybakov *et al.*, 2004). We report here the crystal structure of the product of its phenacylation, namely *N*-(4-bromophenacyl)-4,6-dimethyl-2-oxo-1,2-dihydropyridine-2-carbonitrile, (2).



In the pyridine ring of (2), the single and double bonds alternate, though allowing some degree of conjugation. This ring is planar to within 0.0128 (18) Å (Fig. 1).

Atoms attached to the pyridine moiety (O2, C31, C41 and C61) lie in its plane. The benzene ring is planar to within 0.006 (2) Å and atom Br1 lies in that plane. The torsion angle C14—C9—C8—O8 is 6.4 (5)° and the dihedral angle between the benzene and pyridine rings is 85.59 (9)°.

Experimental

Potassium hydroxide (5.6 g, 0.1 mol) and ethanol (100 ml) were placed in a flask. Compound (1) (13.4 g, 0.1 mol) was added in small quantities with rotation of the flask. This mixture was stirred for 20 min and the ethanol was evaporated. To the resulting solid, DMF (200 ml) and phenacyl bromide (0.1 mol) were added. The mixture was stirred for 2 h with heating (323 K), cooled and poured into cold water. The resulting precipitate was filtered off and dried in air. To separate the mixture of two isomers [*N*-isomer (2) and *O*-isomer (2*a*)], the precipitate was placed on a Shott filter and washed several times with chloroform. Thus, we partly isolated isomer (2). The filtrate contained both isomers and these were separated on a chromatographic column (eluant chloroform) (total yield 13.9 g, 42%; m.p. 498–499 K). ^{1}H NMR (DMSO-d$_6$): 2.32 (*s*, 3 H, 6–CH$_3$), 2.40 (*s*, 3H, 4-CH$_3$), 5.62 (*s*, 2H, CH$_2$), 6.32 (*s*, 1H, 5-CH), 7.35–7.37, 8.19–8.21 (*dd*, 4H, Ar).

Crystal data

$C_{16}H_{13}BrN_2O_2$
M_r = 345.19
Monoclinic, $P2_1/c$
a = 9.5667 (16) Å
b = 7.3784 (12) Å
c = 20.850 (4) Å
β = 95.34 (1)°
V = 1465.4 (4) Å^3
Z = 4
D_x = 1.565 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 33–35°
μ = 3.88 mm^{-1}
T = 293 (2) K
Prism, orange
0.15 × 0.15 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	$\theta_{max} = 74.9°$
Non-profiled $\omega/2\theta$ scans	$h = -11 \rightarrow 11$
Absorption correction: none	$k = 0 \rightarrow 9$
3007 measured reflections	$l = 0 \rightarrow 26$
3007 independent reflections	1 standard reflection
2444 reflections with $I > 2\sigma(I)$	every 200 reflections
	intensity decay: 1%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 0.9586P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.039$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.107$	$\Delta\rho_{max} = 0.34$ e Å^{-3}
$S = 1.03$	$\Delta\rho_{min} = -0.39$ e Å^{-3}
3007 reflections	Extinction correction: none
192 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Br1—C12	1.896 (3)	C5—C6	1.362 (4)
N1—C6	1.376 (3)	C6—C61	1.493 (4)
N1—C2	1.394 (3)	C7—C8	1.526 (4)
N1—C7	1.460 (3)	C8—O8	1.209 (3)
C2—O2	1.232 (3)	C8—C9	1.485 (4)
C2—C3	1.451 (3)	C9—C14	1.388 (4)
C3—C4	1.372 (4)	C9—C10	1.391 (4)
C3—C31	1.436 (4)	C10—C11	1.390 (4)
C31—N31	1.142 (4)	C11—C12	1.374 (4)
C4—C5	1.397 (4)	C12—C13	1.384 (4)
C4—C41	1.507 (4)	C13—C14	1.376 (4)
C6—N1—C2	123.0 (2)	N1—C6—C61	119.4 (2)
C6—N1—C7	120.4 (2)	N1—C7—C8	109.6 (2)
C2—N1—C7	116.2 (2)	O8—C8—C9	121.0 (2)
O2—C2—N1	121.2 (2)	O8—C8—C7	119.8 (2)
O2—C2—C3	123.7 (3)	C9—C8—C7	119.1 (2)
N1—C2—C3	115.1 (2)	C14—C9—C10	119.3 (3)
C4—C3—C31	121.1 (2)	C14—C9—C8	118.1 (2)
C4—C3—C2	122.1 (2)	C10—C9—C8	122.6 (2)
C31—C3—C2	116.8 (2)	C11—C10—C9	120.1 (3)
N31—C31—C3	177.3 (3)	C12—C11—C10	119.3 (3)
C3—C4—C5	118.7 (2)	C11—C12—C13	121.3 (3)
C3—C4—C41	122.2 (3)	C11—C12—Br1	120.0 (2)
C5—C4—C41	119.1 (3)	C13—C12—Br1	118.7 (2)
C6—C5—C4	121.2 (3)	C14—C13—C12	119.2 (3)
C5—C6—N1	119.9 (3)	C13—C14—C9	120.8 (3)
C5—C6—C61	120.6 (3)		

H atoms bonded to C atoms were included in calculated positions and refined as riding atoms. Calculated C—H bond lengths are in the range 0.93–0.99 Å. For methyl H atoms, U_{iso} values were set equal to $1.5U_{eq}$ of the carrier atoms; for other H atoms, U_{iso} values were set at $1.2U_{eq}$ of the carrier atoms.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 for Windows (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

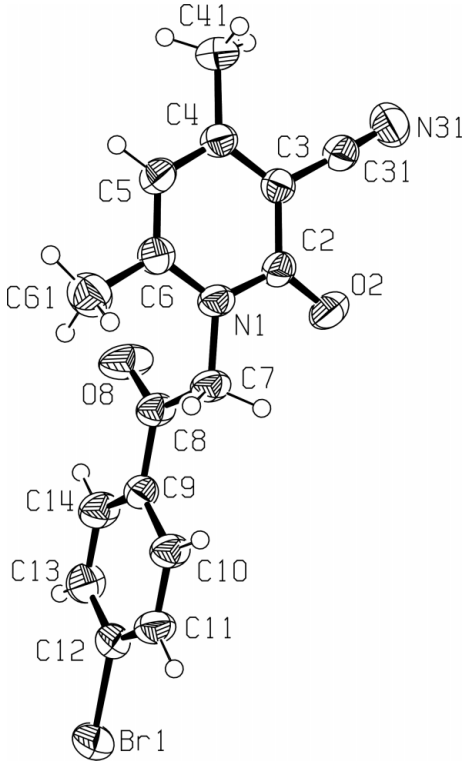


Figure 1
ORTEP-3 (Farrugia, 1997) plot of the title molecule and the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

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