

Dmitry V. Albov,* Eugenia I. Turubanova, Victor B. Rybakov, Eugene V. Babaev and Leonid A. Aslanov

Department of Chemistry, Moscow State University, 119992 Moscow, Russian Federation

Correspondence e-mail: albov@biocryst.phys.msu.su

Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(C-C)$ = 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>.

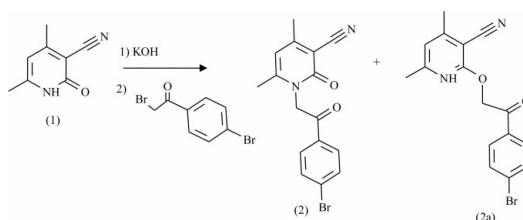
N-(4-Bromophenacyl)-4,6-dimethyl-2-oxo-1,2-dihydropyridine-2-carbonitrile

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

Received 8 June 2004
Accepted 11 June 2004
Online 26 June 2004

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 (2a)], 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). 1H NMR (DMSO- d_6): 2.32 (s, 3 H, 6-CH₃), 2.40 (s, 3H, 4-CH₃), 5.62 (s, 2H, CH₂), 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) Å³
 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
Non-profiled $\omega/2\theta$ scans
Absorption correction: none
3007 measured reflections
3007 independent reflections
2444 reflections with $I > 2\sigma(I)$

$\theta_{\max} = 74.9^\circ$
 $h = -11 \rightarrow 11$
 $k = 0 \rightarrow 9$
 $l = 0 \rightarrow 26$
1 standard reflection
every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.107$
 $S = 1.03$
3007 reflections
192 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 0.9586P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.34 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.39 \text{ e } \text{\AA}^{-3}$
Extinction correction: none

Table 1

Selected geometric parameters (\AA , $^\circ$).

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 \AA . For methyl H atoms, U_{iso} values were set equal to $1.5U_{\text{eq}}$ of the carrier atoms; for other H atoms, U_{iso} values were set at $1.2U_{\text{eq}}$ of the carrier atoms.

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, 1997); program(s) used to refine structure: *SHELXL97*

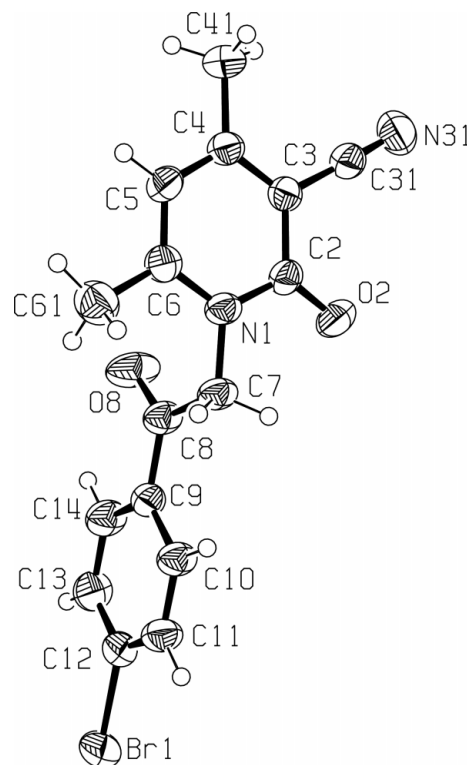


Figure 1

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

(Sheldrick, 1997); molecular graphics: *ORTEP-3* for Windows (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

The authors are indebted to the Russian Foundation for Basic Research for covering the licence fee for use of the Cambridge Structural Database (Allen, 2002) (project No. 02-07-90322).

References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
 Enraf–Nonius (1994). *CAD-4 EXPRESS*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
 Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
 Rybakov, V. B., Bush, A. A., Babaev, E. V. & Aslanov, L. A. (2004). *Acta Cryst.* **E60**, 160–o161.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.