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5-Amino-6-phenyl-1,6-dihydropyridazin-3(2*H*)-one

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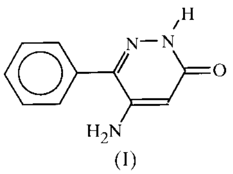
In the title compound, $C_{10}H_9N_3O$, the pyridazinone moiety is essentially planar and forms a dihedral angle of 49.5 (1)° with the phenyl substituent. The molecular packing is stabilized by van der Waals interactions and hydrogen bonds.

Comment

It is known that 6-aryl-3(2*H*)-pyridazinones and their 4,5-dihydro derivatives display several pharmacological activities, all of them related to cardiotonics, such as reduction of blood pressure, inhibition of platelet aggregation, positive inotropic activity, and others (Robertson *et al.*, 1986). Likewise, 6-arylpyridazinones with nitro and acyl substituents at the 4- and 5-positions show good antiaggregating properties (Schudt *et al.*, 1991). We have previously reported the synthesis of 5-aminomethyl-6-aryl-4,5-dihydropyridazinones and 6-aryl-5-aminomethyl-3(2*H*)-pyridazinones (Raviña *et al.*, 1990). Some of these compounds show a good *in vitro* inhibitory activity on ADP-induced rat platelet aggregation. As a continuation of this previous report on the chemistry and pharmacology of this class of compounds, we carried out the crystal structure determination of 5-amino-6-phenyl-1,6-dihydropyridazin-3(2*H*)-one, (I). This enamine-like compound can be employed in the synthesis of hetero-condensed pyridazinones. Recently, we used this compound as an intermediate in the synthesis of pyrido[2,3-*d*]pyridazines (Pita *et al.*, 2000).



(I)

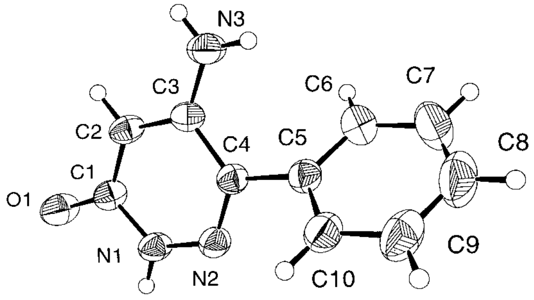


Figure 1
A plot of (I) showing the atomic numbering scheme. Displacement ellipsoids are drawn at 50% probability level, and H atoms are shown as spheres of arbitrary radii.

There are no unusual bond distances and angles in (I), and they are in the range of calculated values using the *AM*1 method in related structures (Estevez *et al.*, 1998). The bond lengths in the pyridazinone ring range from 1.304 (2) to 1.450 (3) Å. The torsion angle between the pyridazinone and the phenyl ring, found using the quantum chemical *AM*1 method in *MOPAC* (Stewart, 1990) for the lower energy conformations, is in the range 40–140°, with a heat of formation of 38.30 kcal mol^{-1} (1 kcal = 4.184 kJ). In the crystal structure this angle is −51.3 (3)°, which corresponds to the minimum in the energy calculations. The calculated favoured conformation of the enol form corresponds to torsion angles in the same range (40–140°) and a heat of formation of 34.20 kcal mol^{-1}, which shows that this enol is the predominant form at equilibrium. This is contrary to the fact that in the crystal the molecule is present in the amide form, which corresponds to a higher heat of formation. The dihedral angle between the respective least-squares planes of the pyridazinone ring and the phenyl ring is 49.5 (1)°. The mean Csp^2—Csp^2 bond length within the phenyl ring is 1.382 (1) Å. A view of (I) can be seen in Fig. 1.

The N3 atom of the amine and the N1 atom of the amide group in the pyridazinone ring are involved in two intermolecular hydrogen bonds with a neighbouring O1, forming an infinite two-dimensional network in the plane [001].

Experimental

A suspension of 5-bromo-6-phenyl-3(2*H*)-pyridazinone (0.5 g, 1.9 mmol), ammonium chloride (0.3 g, 5.6 mmol) and ammonium hydroxide (50 ml) was heated at 458 K at pressure of 374 psi (1 psi $\simeq$ 6.895 kPa) for 3 h in a Parr reactor. The mixture was evaporated *in vacuo* and washed with ammonium hydroxide, and the solid obtained, (I), was recrystallized from ethanol (yield 70%; m.p. 517 K). Spectroscopic analysis: IR (KBr), cm^{-1}: 3480–3425 (NH), 1670 (CO); ^{1}H NMR (DMSO-δ_6, p.p.m.): δ 12.12 (*s*, 1H, NH, deuterium oxide exchangeable), 7.50–7.43 (*m*, 5H, aromatics), 5.71 (*s*, 1H, CH—CO), 5.96 (*s*, 2H, NH_2); ^{13}C NMR (p.p.m.): δ 162.5 (C3), 99.1 (C4), 149.2 (C5), 140.1 (C6), 134.4 (C1′), 129.1 (C2′, C6′), 128.8 (C3′, C5′), 129.1 (C4′); analysis, calculated: C 64.16, H 4.85, N 22.45%; found: C 64.20, H 4.78, N 22.43%.

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Table 1
Selected geometric parameters (Å, °).

O1—C1	1.265 (2)	N3—C3	1.346 (3)
N1—N2	1.344 (2)	C1—C2	1.395 (3)
N1—C1	1.367 (2)	C2—C3	1.376 (3)
N2—C4	1.304 (2)	C3—C4	1.450 (3)
N2—N1—C1	125.86 (15)	N3—C3—C2	122.12 (17)
N1—N2—C4	118.06 (15)	N3—C3—C4	120.82 (16)
O1—C1—C2	126.04 (17)	N2—C4—C5	114.71 (16)
N1—C1—C2	115.88 (16)	N2—C4—C3	121.97 (16)
O1—C1—N1	118.07 (16)		

Crystal data

$C_{10}H_9N_3O$
M_r = 187.20
Orthorhombic, *Pbca*
a = 8.752 (2) Å
b = 10.525 (5) Å
c = 20.619 (5) Å
V = 1899.3 (11) Å^3
Z = 8
D_x = 1.309 Mg m^{-3}

Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10.78–28.12°
μ = 0.73 mm^{-1}
T = 293 (2) K
Prism, light green
0.48 × 0.20 × 0.14 mm

Data collection

Siemens *P*4 four-circle diffractometer
$2\theta/\omega$ scans
Absorption correction: ψ-scan (North *et al.*, 1968)
$T_{\min}$ = 0.626, $T_{\max}$ = 0.903
1733 measured reflections
1266 independent reflections
1102 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.024
$\theta_{\max}$ = 57.19°
$h = -1 \rightarrow 9$
$k = -1 \rightarrow 11$
$l = -1 \rightarrow 22$
3 standard reflections every 100 reflections intensity decay: <1.0%

Refinement

Refinement on F^2
$R(F)$ = 0.038
$wR(F^2)$ = 0.104
S = 1.100
1266 reflections
128 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0532P)^2 + 0.757P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.002
$\Delta\rho_{\max}$ = 0.14 e Å^{-3}
$\Delta\rho_{\min}$ = −0.14 e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.0110 (8)

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97; molecular graphics: *DIAMOND* (Bergerhoff, 1996); software used to prepare material for publication: *PLATON* (Spek, 1990), *PARST* (Nardelli, 1983, 1995) and *PARSTCIF* (Nardelli, 1992).

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1$^{\rm i}$	0.86	1.96	2.818 (2)	180
N3—H3A···O1$^{\rm ii}$	0.86	2.06	2.880 (3)	159

Symmetry codes: (i) $-x, -y, -z$; (ii) $\frac{1}{2}+x, \frac{1}{2}-y, -z$.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1336). Services for accessing these data are described at the back of the journal.

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