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5,6-Dihydrobenzo[*h*]quinazoline

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.070
wR factor = 0.245
Data-to-parameter ratio = 12.6

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

5,6-Dihydrobenzo[*h*]quinazoline, $C_{12}H_{10}N_2$, has been synthesized from α-tetralone and formamide using different palladium complexes as catalysts. There are two molecules in the asymmetric unit and the least-squares planes through the two molecules show that they are almost perpendicular to each other, resulting in a zigzag packing pattern in the crystal structure.

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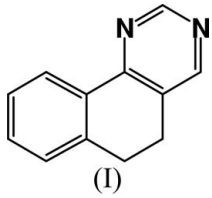
Comment

Pyrimidines are widely found in nature, *e.g.* in pyrimidine bases and purines in nucleic acids, and in the vitamin thiamin. They have also attracted interest as potential drugs and are available in the nucleoside analogue AZT used in AIDS therapy, Acyclovir used in treatment of herpes infections and the prodrug Capecitabine used in cancer therapy. The interesting chemical and physiological properties of pyrimidines have led to a number of syntheses being developed [see von Angerer (2004) for a comprehensive review]. In our procedure, good yields of the expected products are formed from the appropriate ketone when reacted with formamide, catalyzed by different palladium complexes (Ingebrigtsen *et al.*, 2005). The title compound, (I), crystallizes in the monoclinic centrosymmetric space group $P2_1/c$, with two molecules in the asymmetric unit. The atomic numbering scheme of the title compound is shown in Fig. 1. The bond lengths are within the normal range of such bonds (Allen *et al.*, 1987). There are no significant C—H···N or C—H···π(ring) interactions between the molecules in the crystal packing. The benzene and pyrazine rings in each independent molecule are essentially planar, while they make a dihedral angle with each other of 17.5 (2) and 19.4 (2)° for molecules 1 (N11) and 2 (N21), respectively. The total puckering amplitude parameter Q_T for the central six-membered ring system is 0.470 (3) and 0.468 (3) Å, respectively, for molecule 1 and 2 (Cremer & Pople, 1975; Iulek & Zukerman-Schpector, 1997). This ring puckering is described as 50% half-chair and 44% twisted-boat for molecule 1, and 47% half-chair and 45% twisted-boat for molecule 2. Fig. 2 shows the zigzag packing of the molecules in the crystal structure.



(I)

Experimental

To a 10 ml flask charged with $Pd(OAc)_2$ (0.05 equivalents) and PPh_3 (0.10 equivalents) were added formamide (5.0 g), PhI (2.0 g) and α-tetralone (1.0 equivalents). The resulting mixture was heated at 433 K for 8 h. The reaction mixture was diluted with diethyl ether and extracted three times with 2 *M* HCl. The combined aqueous layers were basified with 4 *M* NaOH and extracted with diethyl ether. The organic layer was washed with water and brine, and dried over Na_2CO_3. Evaporation of the solvent gave the crude product as a white solid. Purification by silica-gel column chromatography (EtOAc) gave crystals that were dissolved in a small amount of diethyl ether. Heptane was added and crystals of the title compound were grown by slow evaporation of the solvent at room temperature.

Crystal data

$C_{12}H_{10}N_2$
M_r = 182.22
Monoclinic, $P2_1/c$
a = 25.118 (5) Å
b = 7.3522 (9) Å
c = 10.0830 (17) Å
β = 101.664 (11)°
V = 1823.6 (5) Å^3
Z = 8
D_x = 1.327 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 12–16°
μ = 0.08 mm^{-1}
T = 298 (2) K
Block, brown
0.5 × 0.3 × 0.3 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan [*ABSCALC* in *OSCAIL;* McArdle & Daly (1999) and North *et al.* (1968)]
T_{min} = 0.961, T_{max} = 0.976
3334 measured reflections
3184 independent reflections
1703 reflections with $I > 2\sigma(I)$
R_{int} = 0.006
θ_{max} = 25.0°
$h = -12 \rightarrow 29$
$k = 0 \rightarrow 8$
$l = -11 \rightarrow 11$
3 standard reflections
frequency: 120 min
intensity decay: 10%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.245
S = 1.07
3184 reflections
253 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1448P)^2 + 0.3821P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

All the H atoms were positioned geometrically and refined using a riding model, fixing the aromatic C—H distances at 0.93 Å and the CH_2 C—H distances at 0.97 Å, with $U_{iso}(H) = 1.3U_{eq}(C)$. The quality of the crystal was rather poor and accordingly data were collected only to θ_{max} = 25.0°.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992); cell refinement: *CELDIM* in *CAD-4-PC Software*; data reduction: *XCAD4* (McArdle & Higgins, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1993*a*); software used to prepare material for publication: *OSCAIL* (McArdle, 1993*b*).

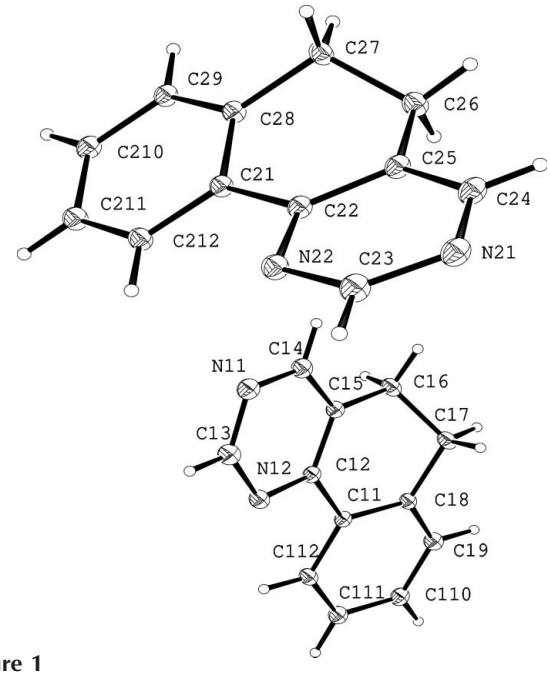


Figure 1
A view of the asymmetric unit of the title compound, (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

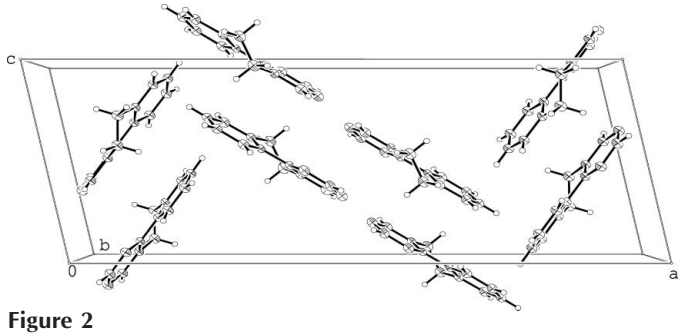


Figure 2
A view of the crystal packing in the title compound, (I). Displacement ellipsoids are drawn at the 30% probability level.

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