Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Amino-4-phenyl-5,6-dihydrobenzo[*h*]quinazoline

Xiangshan Wang,[a]* Daqing Shi,[a] Shujiang Tu[a] and Kaibei Yu[b]

[a]Department of Chemistry, Xuzhou Normal University, Xuzhou 221116, People's Republic of China, and [b]Institute of Organic Chemistry, Chinese Academy of Sciences, Chengdu Sichuan, 610041, People's Republic of China

Correspondence e-mail: xswang@xznu.edu.cn

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.102
Data-to-parameter ratio = 12.3

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

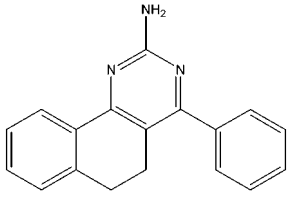
The title compound, $C_{18}H_{15}N_3$, was synthesized by the reaction of 2-phenylmethylidene-3,4-dihydronaphthalen-1(2*H*)-one with guanidine carbonate in ethylene glycol under microwave irradiation. X-ray analysis revealed the formation of a pyrimidine ring. The partially saturated six-membered ring adopts a distorted boat conformation.

Received 24 February 2003
Accepted 27 February 2003
Online 7 March 2003

This paper is dedicated to Professor Daqing Shi on the occasion of his 40th birthday.

Comment

Pyrimidine derivatives occupy a unique position as functional agents, both as essential components of nucleic acids and therapeutic agents (Mochida Pharmaceutical Co. Ltd, 1982). Since microwave heating, with the resulting mass heating effect, much faster temperature rise, and attainment of the high temperatures required for reactions, was used for organic synthesis by Gedye *et al.* (1986), microwave chemistry has been a topic of continuing interest. Rate enhancement in many reactions has been reported in recent years. We report here the crystal structure of the title compound, (I), which was synthesized by the reaction of 2-phenylmethylidene-3,4-dihydronaphthalen-1(2*H*)-one with guanidine carbonate in ethylene glycol under microwave irradiation.



(I)

In (I), atoms C1, C2, N1, N2, C11 and C12 form a pyrimidine ring (Fig. 1). The N1—C1, N1—C2, N2—C1 and N2—C12 bond lengths of 1.3431 (19), 1.3471 (17), 1.3442 (18) and 1.3469 (17) Å, respectively, are slightly longer than those of ethyl 2-amino-4-(3-nitrophenyl)-1,4-dihydro-2*H*-pyrano[3,2-*h*]quinoline-3-carboxylate (Wang *et al.*, 2003). In addition, the N3—C1 bond length of 1.3603 (19) Å is shorter than the typical Csp^2—N bond distance (1.426 Å; Lorente *et al.*, 1995). The six-membered ring C2/C3/C8/C9/C10/C11 adopts a distorted boat conformation; atoms C2, C3, C8 and C9 are coplanar, while C10 and C11 deviate from the plane by 0.857 (3) and 0.437 (3) Å, respectively. The C8—C9—C10—C11 torsion angle is −52.98 (18)°. The dihedral angles between this plane and the C3–C8 benzo ring, the C13–C18 phenyl ring and the pyrimidine ring are 1.6 (1), 68.39 (6) and 22.6 (1)°, respectively.

In the crystal structure, molecules are linked by N—H···N hydrogen bonds, forming polymers (Fig. 2 and Table 2). The hydrogen bonds are formed between the amino group and

atoms N1 and N2 of the pyrimidine rings of adjacent molecules.

Experimental

The title compound, (I), was prepared by the reaction of 2-phenylmethylidene-3,4-dihydronaphthalen-1(2*H*)-one with guanidine carbonate in ethylene glycol under microwave irradiation (m.p. 447–448 K). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an acetone and petroleum ether solution.

Crystal data

$C_{18}H_{15}N_3$
M_r = 273.33
Monoclinic, $P2_1/c$
a = 10.902 (2) Å
b = 6.906 (1) Å
c = 18.577 (3) Å
β = 94.85 (1)°
V = 1393.6 (4) Å^3
Z = 4
D_x = 1.303 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 3.0–12.6°
μ = 0.08 mm^{-1}
T = 296 (2) K
Block, yellow
0.58 × 0.54 × 0.24 mm

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: none
2919 measured reflections
2448 independent reflections
1810 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.0°
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 8$
$l = -22 \rightarrow 22$
3 standard reflections
every 97 reflections
intensity decay: 1.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.102
S = 1.01
2448 reflections
199 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0602P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.043 (3)

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.3431 (19)	N3—C1	1.3603 (19)
N1—C2	1.3471 (17)	C2—C11	1.392 (2)
N2—C1	1.3442 (18)	C9—C10	1.517 (2)
N2—C12	1.3469 (17)	C11—C12	1.394 (2)
C1—N1—C2	115.74 (12)	N1—C2—C11	122.67 (13)
C1—N2—C12	116.21 (12)	N1—C2—C3	117.81 (12)
N1—C1—N2	126.45 (12)	N2—C12—C11	122.05 (13)
N1—C1—N3	116.72 (13)	N2—C12—C13	115.43 (13)
N2—C1—N3	116.83 (14)		
C11—C2—C3—C8	−21.5 (2)	C8—C9—C10—C11	−52.98 (18)
C2—C3—C8—C9	0.5 (2)	C3—C2—C11—C10	2.2 (2)
C3—C8—C9—C10	36.76 (19)	C9—C10—C11—C2	35.1 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3B···N1^{i}	0.876 (9)	2.304 (10)	3.162 (2)	166.2 (17)
N3—H3A···N2^{ii}	0.880 (9)	2.370 (12)	3.1868 (19)	154.4 (15)

Symmetry codes: (i) $2-x, \frac{1}{2}+y, \frac{3}{2}-z$; (ii) $2-x, y-\frac{1}{2}, \frac{3}{2}-z$.

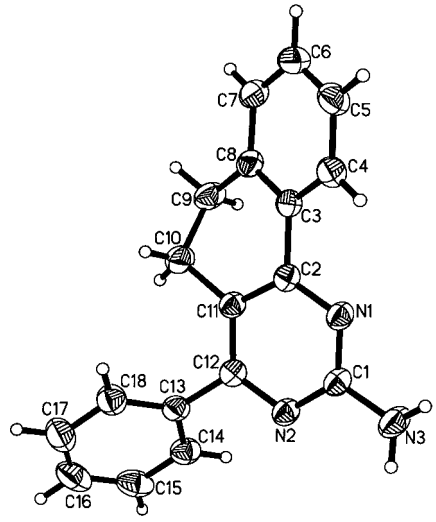


Figure 1
The molecular structure of (I), showing the atom-numbering scheme, with displacement ellipsoids drawn at the 50% probability level.

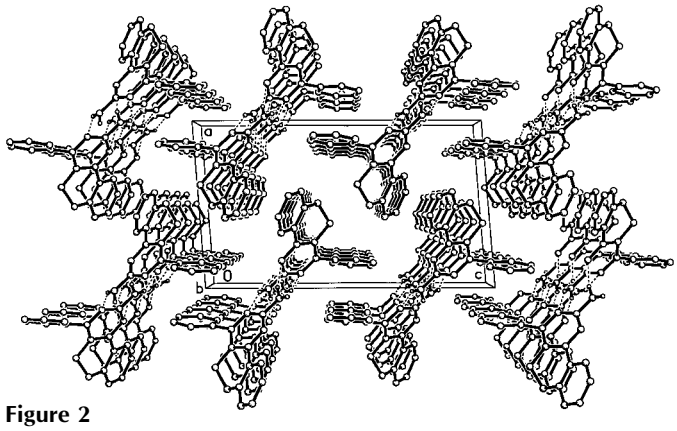


Figure 2
The molecular packing in the crystal of (I), viewed down the *b* axis.

Atoms H3*A* and H3*B* were refined isotropically. The positions of the other H atoms were fixed geometrically, with C—H distances of 0.93 (CH) or 0.97 Å (CH_2).

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

We thank the Foundation of the 'Surpassing Project' of Jiangsu Province for financial support.

References

Gedye, R., Smith, F., Westaway, K., Humera, A., Baldisern, L., Laberge, L. & Rousell, J. (1986). *Tetrahedron Lett.* **27**, 279–282.
Lorente, A., Galan, C. & Fonseca, I. (1995). *Can. J. Chem.* **73**, 1546–1555.
Mochida Pharmaceutical Co. Ltd (1981). Japan Patent 81 127 383.
Sheldrick, G. M. (1997). *SHELXTL.* Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1994). *XSCANS*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Wang, X. S., Shi, D. Q. & Tu, S. J. (2003). *Chin. J. Struct. Chem.* In the press.