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3-(4-Bromophenyl)-2,2-dimethyl-2,3-dihydroquinazolin-4(1*H*)-one

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Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.003 Å
R factor = 0.027
wR factor = 0.068
Data-to-parameter ratio = 14.0

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

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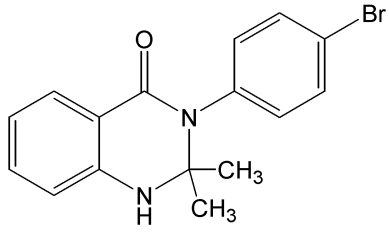
The title compound, $C_{16}H_{15}BrN_2O$, was synthesized by the reaction of *N*-(4-bromophenyl)-2-nitrobenzamide and acetone, induced by a low-valent titanium reagent ($TiCl_4$/Zn). The dihydropyrimidine ring adopts a screw-boat conformation. The molecules are connected by N—H···O hydrogen bonds, forming a linear chain along the *a* axis.

Comment

Quinazolin-4(3*H*)-one is an alkaloid (Chou *et al.*, 1948). Substituted quinazolin-4(3*H*)-ones possess a wide range of pharmacological activities, such as antibacterial (Ager *et al.*, 1977) and anticancer (Skula *et al.*, 1981). Low-valent titanium reagents have an exceedingly high ability to promote the reductive coupling of carbonyl compounds and are attracting increasing interest in organic synthesis (McMurry, 1983). In the course of our work on the applications of low-valent titanium reagents in the preparation of bioactive molecules, we have reported the syntheses of quinazolin-4(3*H*)-ones (Shi, Rong *et al.*, 2003), imidazo[1,2-*c*]quinazolines (Shi, Wang *et al.*, 2004) and pyrroles (Shi, Shi, Wang *et al.*, 2004) with the aid of low-valent titanium reagents. We report here the synthesis and the crystal structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. In the dihydropyrimidine ring (N1/C2/N2/C3/C8/C1), because of conjugation, the distances N1—C1 [1.360 (3) Å] and N2—C3 [1.361 (3) Å] are significantly shorter than the typical Csp^2—N bond distance (1.426 Å; Lorente *et al.*, 1995). This dihydropyrimidine ring adopts a screw-boat conformation; atoms C1, C8, C3 and N2 are coplanar, while N1 and C2 deviate from the plane by 0.134 (3) and 0.629 (2) Å, respectively. A similar conformation was observed in the structure of 7-chloro-2,2-dimethyl-3-(4-methylphenyl)-2,3-dihydroquinazolin-4(1*H*)-one (Shi, Shi, Zhuang *et al.*, 2004). The dihedral angle between the two substituted phenyl rings is 73.8 (2)°. The molecules are linked by N—H···O hydrogen bonds, forming a linear chain along the *a* axis (Fig. 2 and Table 2).

Experimental

The title compound, (I), was prepared by the reaction of N-(4-bromophenyl)-2-nitrobenzamide (0.96 g, 3 mmol) and acetone (0.17 g, 3 mmol), induced by a low-valent titanium reagent ($TiCl_4$/Zn) (yield 83%, m.p. 537–538 K). Single crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution. IR (cm^{-1}): 3307 (NH), 1627, 1579, 1489, 871, 815, 756, 696 (benzene ring); ^{1}H NMR (δ): 1.49 (6H, *s*, $2CH_3$), 6.68 (1H, *d*, J = 8.0 Hz, C^8-H), 6.88 (1H, *dd*, J_1 = 8.0 Hz, J_2 = 7.2 Hz, C^6-H), 7.13 (2H, *d*, J = 8.0 Hz, $C^{2'}$-H,$C^{6'}$-H), 7.34 (1H, *dd*, J_1 = 7.2 Hz, J_2 = 8.0 Hz, C^7-H), 7.56 (2H, *d*, J = 8.0 Hz, $C^{3'}$-H, $C^{5'}$-H), 7.94 (1H, *d*, J = 8.0 Hz, C^5-H); Elemental analysis calculated: C 58.02, H 4.56, N 8.46%; found: C 58.18, H 4.54, N 8.65%.

Crystal data

$C_{16}H_{15}BrN_2O$	Z = 2
M_r = 331.21	D_x = 1.516 Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
a = 6.8865 (6) Å	Cell parameters from 2946 reflections
b = 9.9508 (6) Å	θ = 3.5–25.3°
c = 11.6702 (8) Å	μ = 2.83 mm^{-1}
α = 65.397 (4)°	T = 193 (2) K
β = 88.452 (6)°	Block, colorless
γ = 86.194 (6)°	0.50 × 0.30 × 0.16 mm
V = 725.51 (9) $Å^3$	

Data collection

Rigaku Mercury CCD diffractometer	2623 independent reflections
ω scans	2325 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (Jacobson, 1998)	R_{int} = 0.026
T_{min} = 0.332, T_{max} = 0.660	θ_{max} = 25.4°
7155 measured reflections	$h = -7 \rightarrow 8$
	$k = -11 \rightarrow 11$
	$l = -12 \rightarrow 14$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.027	$w = 1/[\sigma^2(F_o^2) + (0.0402P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.069	$(\Delta/\sigma)_{max}$ = 0.002
S = 1.03	$\Delta\rho_{max}$ = 0.36 e $Å^{-3}$
2623 reflections	$\Delta\rho_{min}$ = −0.50 e $Å^{-3}$
188 parameters	

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.238 (2)	N1—C2	1.502 (2)
N1—C1	1.360 (3)	N2—C3	1.361 (3)
N1—C9	1.443 (2)	N2—C2	1.459 (3)
C1—N1—C9	117.44 (15)	O1—C1—N1	121.48 (18)
C1—N1—C2	122.19 (15)	O1—C1—C8	121.87 (18)
C9—N1—C2	118.55 (16)	N1—C1—C8	116.55 (16)
C3—N2—C2	120.15 (17)	N2—C2—N1	106.34 (16)
C9—N1—C1—O1	−3.9 (3)	C1—N1—C2—N2	−40.1 (2)
C2—N1—C1—O1	−168.31 (18)	C9—N1—C2—N2	155.65 (16)
C9—N1—C1—C8	179.59 (16)	C2—N2—C3—C8	−27.3 (3)
C2—N1—C1—C8	15.2 (3)	C2—N2—C3—C4	157.40 (19)
C3—N2—C2—N1	46.0 (2)		

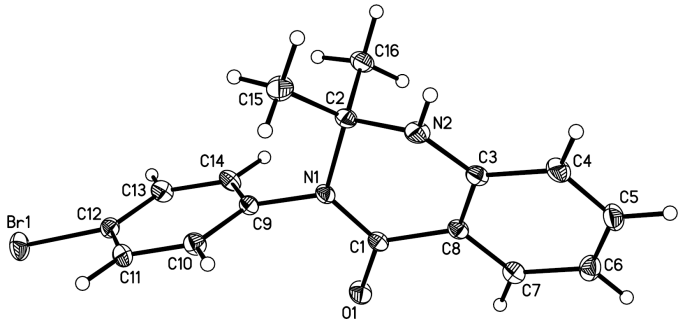


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

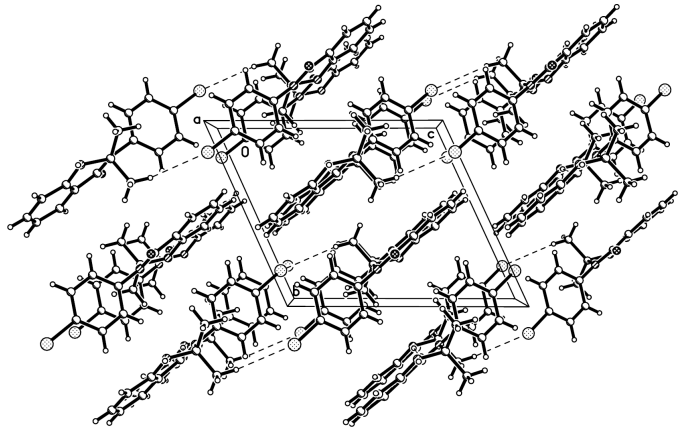


Figure 2
A molecular packing diagram of the crystal structure of (I). Dashed lines indicate the C—H···Br interactions.

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

D—H···A	D—H	H···A	D···A	D—H···A
C16—H16B···Br1^{i}	0.98	3.02	3.894 (2)	149
N2—H2···O1^{ii}	0.75 (2)	2.18 (3)	2.925 (2)	173 (2)

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

The H atom bonded to N2 was refined isotropically. The other H atoms were positioned geometrically and refined as riding, with C—H = 0.95–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl}).

Data collection: *CrystalClear* (Rigaku, 2000); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2003); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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