

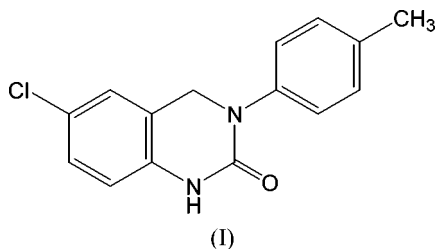
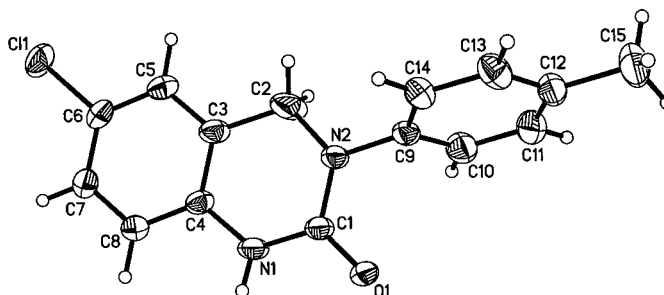
6-Chloro-3-(*p*-tolyl)-3,4-dihydroquinazolin-2(1*H*)-oneDaqing Shi,<sup>a,b\*</sup> Chunling Shi,<sup>a</sup>  
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## Key indicators

Single-crystal X-ray study  
*T* = 193 K  
Mean  $\sigma$ (C–C) = 0.004 Å  
*R* factor = 0.065  
*wR* factor = 0.169  
Data-to-parameter ratio = 17.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The title compound, C<sub>15</sub>H<sub>13</sub>ClN<sub>2</sub>O, was synthesized by the reaction of 5-chloro-*N*-(*p*-tolyl)-2-nitrobenzylamine with triphosgene, induced by a low-valent titanium reagent (TiCl<sub>4</sub>/Zn). The dihydropyrimidine ring adopts a boat conformation.Received 8 July 2004  
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## Comment

Quinazolines are an important class of compound found in many naturally occurring products (*e.g.* hinckdentine A; Blackman *et al.*, 1987; Billimoria & Cava, 1994) and employed as anticancer potent agents (Helissey *et al.*, 1994; Brana *et al.*, 1994; Riou *et al.*, 1991; Ibrahim *et al.*, 1988). 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; Shi *et al.*, 1993, 1997, 1998, 2003, 2004). We report here the crystal structure of the title compound, (I).In (I), the dihydropyrimidine ring adopts a boat conformation (Fig. 1 and Table 1). Atoms C3/C4/N2/C1 are coplanar, while atoms N1 and C2 deviate from the plane by 0.065 (2) and 0.184 (2) Å, respectively. The dihedral angle between the C3–C8 and C9–C14 benzene rings is 72.3 (2)°. In addition, because of the existence of a conjugated system, the N1–C4 [1.395 (3) Å] and N1–C1 [1.355 (3) Å] distances are significantly shorter than the typical Csp<sup>2</sup>–N bond distance**Figure 1**  
The molecular structure of (I), showing 35% probability displacement ellipsoids and the atom-numbering scheme.

(1.426 Å; Lorente *et al.*, 1995). Molecules are linked by N—H···O hydrogen bonds (Table 2), forming dimers (Fig. 2).

### Experimental

The title compound, (I), was prepared by the reaction of 5-chloro-*N*-(4-*p*-tolyl)-2-nitrobenzylamine (0.55 g) with triphosgene (0.89 g), induced by a low-valent titanium reagent (TiCl<sub>4</sub>/Zn) (yield 91%; m.p. 487–488 K). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an acetone solution.

#### Crystal data

C <sub>15</sub> H <sub>13</sub> ClN <sub>2</sub> O	$D_x = 1.358 \text{ Mg m}^{-3}$
$M_r = 272.72$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 5465 reflections
$a = 19.006 (6) \text{ \AA}$	$\theta = 3.3\text{--}27.5^\circ$
$b = 12.836 (3) \text{ \AA}$	$\mu = 0.28 \text{ mm}^{-1}$
$c = 11.484 (4) \text{ \AA}$	$T = 193 (2) \text{ K}$
$\beta = 107.764 (7)^\circ$	Block, colorless
$V = 2668.1 (13) \text{ \AA}^3$	$0.80 \times 0.52 \times 0.41 \text{ mm}$
$Z = 8$	

#### Data collection

Rigaku Mercury diffractometer	2786 reflections with $I > 2\sigma(I)$
$\omega$ scans	$R_{\text{int}} = 0.019$
Absorption correction: multi-scan (Jacobson, 1998)	$\theta_{\text{max}} = 27.5^\circ$
$T_{\text{min}} = 0.808$ , $T_{\text{max}} = 0.894$	$h = -24 \rightarrow 24$
14355 measured reflections	$k = -16 \rightarrow 14$
3048 independent reflections	$l = -14 \rightarrow 12$

#### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0745P)^2 + 3.41P]$
$R[F^2 > 2\sigma(F^2)] = 0.065$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.169$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.08$	$\Delta\rho_{\text{max}} = 0.54 \text{ e \AA}^{-3}$
3048 reflections	$\Delta\rho_{\text{min}} = -0.47 \text{ e \AA}^{-3}$
178 parameters	
H atoms treated by a mixture of independent and constrained refinement	

**Table 1**

Selected geometric parameters (Å, °).

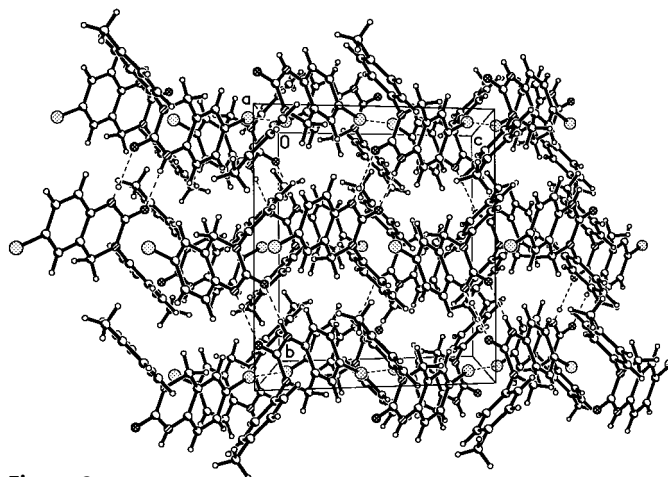
O1—C1	1.235 (2)	N2—C9	1.435 (3)
N1—C1	1.355 (3)	N2—C2	1.442 (3)
N1—C4	1.395 (3)	C2—C3	1.500 (4)
N2—C1	1.365 (2)		
C1—N1—C4	124.80 (18)	N1—C1—N2	116.91 (18)
C1—N2—C9	117.82 (16)	N2—C2—C3	113.4 (2)
C1—N2—C2	124.71 (18)	C8—C4—N1	120.48 (18)
C9—N2—C2	117.17 (17)	N1—C4—C3	118.85 (19)
C4—N1—C1—O1	173.0 (2)	C1—N2—C2—C3	16.1 (5)
C4—N1—C1—N2	−6.6 (3)	C9—N2—C2—C3	−170.3 (2)
C9—N2—C1—O1	1.0 (3)	N2—C2—C3—C5	169.7 (3)
C2—N2—C1—O1	174.6 (3)	N2—C2—C3—C4	−15.1 (5)
C9—N2—C1—N1	−179.4 (2)	C1—N1—C4—C8	−174.9 (2)
C2—N2—C1—N1	−5.9 (4)	C1—N1—C4—C3	6.7 (4)

**Table 2**

Hydrogen-bonding geometry (Å, °).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
N1—H1···O1 <sup>i</sup>	0.82 (3)	2.00 (3)	2.815 (2)	177 (3)

Symmetry code: (i)  $\frac{1}{2} - x, \frac{3}{2} - y, 1 - z$ .



**Figure 2**  
Projection of the crystal structure of (I) along the *a* axis. Dashed lines indicate hydrogen bonds.

The H atom on N1 was refined isotropically, with the N—H bond length restrained to 0.82 (3) Å; other H atoms were positioned geometrically and refined as riding, with C—H = 0.95–0.99 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ , except for the methyl H atoms, for which  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ .

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

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