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7-Chloro-2,2-dimethyl-3-(4-methylphenyl)-1,2-dihydroquinazolin-4(3*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 = 0.051
wR factor = 0.137
Data-to-parameter ratio = 14.1

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

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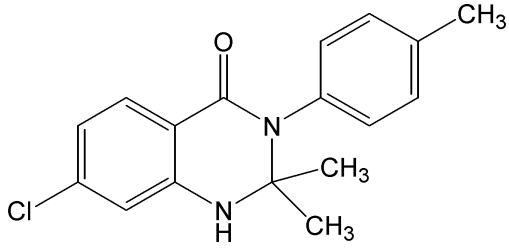
The title compound, $C_{17}H_{17}ClN_2O$, was synthesized by the reaction of *N*-(4-methylphenyl)-4-chloro-2-nitrobenzamide and acetone induced by a low-valent titanium reagent ($TiCl_4$/Zn). The dihydropyrimidine ring in the molecule adopts a screw-boat conformation. The molecules are connected by N—H···O 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 (Shukla *et al.*, 1981). Low-valent titanium reagents have an exceedingly high ability to promote 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 synthesis of quinazolin-4(3*H*)-ones with the aid of a low-valent titanium reagent (Shi *et al.*, 2003). We report here the crystal structure of the title compound, (I), which has been synthesized by a reaction induced by a low-valent titanium reagent.



(I)

The molecular structure of (I) shown in Fig. 1. In the dihydropyrimidine ring, because of the existence of conjugation, the distances N1—C1 [1.359 (3) Å] and N2—C3 [1.368 (3) Å] are significantly shorter than the typical Csp^2—N bond distance (1.426 Å; Lorente *et al.*, 1995). The dihydropyrimidine ring adopts a screw-boat conformation, atoms C1, C8, C3 and N2 being coplanar, while atoms N1 and C2 deviate from the plane by 0.144 (3) and 0.589 (2) Å, respectively. The dihedral angle between the two substituted phenyl rings is 71.4 (2)°. The sum of bond angles around N1 (358.6°) and N2 (350.6°) indicate an approximately planar geometry. 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 4-chloro-*N*-(4-methylphenyl)-2-nitrobenzamide (0.87 g, 3 mmol) and acetone (0.17 g, 3 mmol) in the presence of a low-valent titanium reagent ($TiCl_4$/Zn) (yield 89%, m.p. 551–553 K). IR: 3300(NH), 1633, 1510, 1484, 849, 786, 765 (phenyl ring); ^{1}H NMR: 1.49 (6H, *s*, $2CH_3$), 2.38 (3H, *s*, CH_3), 6.67 (1H, *s*, C4—H), 6.82 (1H, *d*, *J* = 8.0 Hz, C6—H), 7.10 (2H, *d*, *J* = 7.2 Hz, C10—H, C14—H), 7.23 (2H, *d*, *J* = 7.2 Hz, C11—H, C13—H), 7.87 (1H, *d*, *J* = 8.0 Hz, C7—H); Analysis calculated for $C_{17}H_{17}ClN_2O$: C 67.88, H 5.70, N 9.31%; found: C 67.93, H 5.54, N 9.49%. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{17}H_{17}ClN_2O$
M_r = 300.78
Triclinic, $P\overline{1}$
a = 6.886 (1) Å
b = 10.017 (2) Å
c = 11.272 (2) Å
α = 89.258 (9)°
β = 84.168 (8)°
γ = 85.447 (8)°
V = 771.0 (2) Å^3
Z = 2
D_x = 1.296 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2181 reflections
θ = 3.3–25.3°
μ = 0.25 mm^{-1}
T = 193 (2) K
Block, colorless
0.30 × 0.26 × 0.25 mm

Data collection

Rigaku Mercury diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.929, T_{max} = 0.941
7618 measured reflections
2795 independent reflections
2013 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 25.4°
$h = -8 \rightarrow 8$
$k = -11 \rightarrow 12$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.137
S = 1.07
2795 reflections
198 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0655P)^2 + 0.1194P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.237 (2)	N2—C3	1.368 (3)
N1—C1	1.359 (3)	N2—C2	1.453 (3)
N1—C9	1.445 (3)	C1—C8	1.468 (3)
N1—C2	1.500 (2)		
C1—N1—C9	117.95 (16)	N1—C1—C8	116.86 (17)
C1—N1—C2	122.49 (18)	N2—C2—N1	107.53 (18)
C9—N1—C2	118.16 (18)	N1—C2—C16	109.60 (17)
C3—N2—C2	120.63 (18)	N2—C3—C4	121.58 (19)
O1—C1—N1	121.46 (19)	N2—C3—C8	119.0 (2)
O1—C1—C8	121.6 (2)		
C9—N1—C1—O1	5.0 (3)	C1—N1—C2—N2	36.2 (2)
C2—N1—C1—O1	171.19 (18)	C9—N1—C2—N2	−157.60 (17)
C9—N1—C1—C8	−178.86 (16)	C1—N1—C2—C15	152.4 (2)
C2—N1—C1—C8	−12.6 (3)	C9—N1—C2—C15	−41.4 (3)
C3—N2—C2—N1	−42.8 (3)	C1—N1—C2—C16	−85.0 (3)

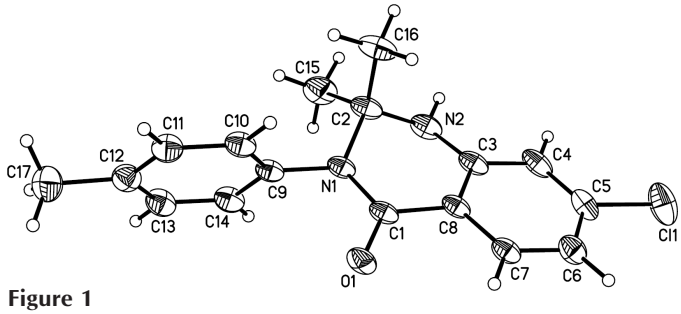


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

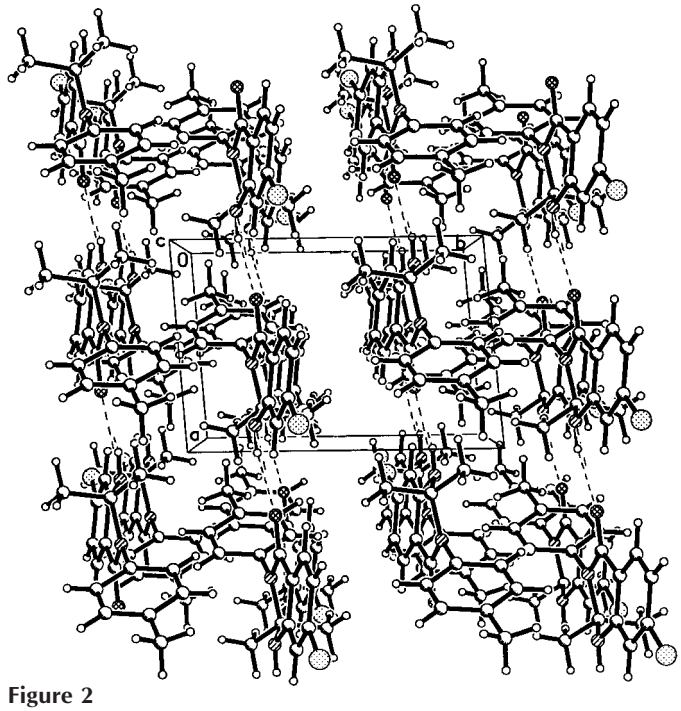


Figure 2
A packing diagram for (I). Dashed lines indicate hydrogen bonds.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1^{i}	0.88 (3)	2.05 (3)	2.912 (2)	168 (2)

Symmetry code: (i) $1 + x, y, z$.

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) for CH and U_{iso}(H) = 1.5U_{eq}(C) for the methyl group.

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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