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

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Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.002 Å
R factor = 0.037
wR factor = 0.096
Data-to-parameter ratio = 16.5

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

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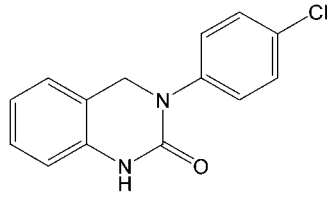
The title compound, $C_{14}H_{11}ClN_2O$, was synthesized by the reaction of *N*-(4-chlorophenyl)-2-nitrobenzylamine with triphosgene, induced by a low-valent titanium reagent ($TiCl_4$/Zn). The dihydroquinazoline ring adopts a skew-boat conformation.

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 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 reductive coupling of carbonyl compounds and are attracting increasing interest in organic synthesis (McMurry, 1983; Shi *et al.*, 1993, 1997, 1998, 2003; Shi, Wang *et al.*, 2004). We report here the synthesis and crystal structure of the title compound, (I).



(I)

In (I), the dihydropyrimidine ring adopts a skew-boat conformation; atoms C2/C3/C4/N1 are coplanar, while atoms C1 and N2 deviate from the plane by 0.391 (2) and 0.595 (2) Å, respectively. A similar conformation was observed in the structure of 5,5-dimethyl-2,3-diphenyl-5,6-dihydroimidazo[1,2-*c*]quinazoline (Wu *et al.*, 2004). The dihedral angle between the C3–C8 and C9–C14 benzene rings is 60.0 (2)°. In addition, because of the existence of a conjugated system, the N1—C4 [1.4006 (18) Å] and N1—C1 [1.3589 (17) Å] distances are significantly shorter than the

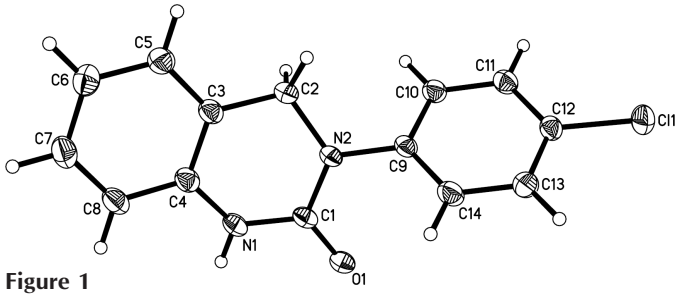


Figure 1
The molecular structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme.

typical Csp^2—N bond distance (1.426 Å; Lorente *et al.*, 1995). The molecules are linked by N—H···O hydrogen bonds (Table 2) to form dimers (Fig. 2).

Experimental

The title compound, (I), was prepared by the reaction of *N*-(4-chlorophenyl)-2-nitrobenzylamine (0.53 g, 2 mmol) with triphosgene (0.89 g, 3 mmol), induced by a low-valent titanium reagent ($TiCl_4$/Zn) (yield 83%, m.p. 552–553 K). Single crystals suitable for X-ray diffraction were obtained by evaporation of an acetone solution. ^{1}H NMR (400 MHz, DMSO-d_6, δ): 4.82 (2H, *s*, CH_2), 6.89 (1H, *d*, *J* = 8.0 Hz, ArH), 6.93 (1H, *t*, *J* = 7,6 Hz, ArH), 7.17 (1H, *d*, *J* = 6.8 Hz, ArH), 7.21 (1H, *d*, *J* = 8.0 Hz, ArH), 7.40–7.47 (4H, *m*, ArH), 9.66 (1H, *s*, NH). Elemental analysis calculated: C 65.00, H 4.29, N 10.83%; found: C 65.16, H 4.02, N 10.77%.

Crystal data

$C_{14}H_{11}ClN_2O$
M_r = 258.70
Monoclinic, $P2_1/c$
a = 9.788 (2) Å
b = 13.142 (3) Å
c = 10.114 (2) Å
β = 111.329 (4)°
V = 1211.9 (4) Å^3
Z = 4
D_x = 1.418 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5478 reflections
θ = 3.1–27.5°
μ = 0.30 mm^{-1}
T = 193 (2) K
Block, colourless
0.68 × 0.52 × 0.30 mm

Data collection

Rigaku Mercury CCD diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.821, T_{max} = 0.915
13 104 measured reflections
2767 independent reflections
2596 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 27.5°
$h = -12 \rightarrow 12$
$k = -15 \rightarrow 17$
$l = -12 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.096
S = 1.08
2767 reflections
168 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 0.4313P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.2358 (17)	N2—C9	1.4317 (16)
N1—C1	1.3589 (17)	N2—C2	1.4668 (17)
N1—C4	1.4006 (18)	C2—C3	1.4986 (19)
N2—C1	1.3775 (16)		
C1—N1—C4	124.12 (11)	O1—C1—N1	121.39 (11)
C1—N2—C9	118.77 (11)	O1—C1—N2	122.87 (12)
C1—N2—C2	121.35 (11)	N1—C1—N2	115.73 (12)
C9—N2—C2	117.61 (10)	N2—C2—C3	111.11 (11)
C4—N1—C1—N2	−9.71 (19)	N2—C2—C3—C4	−26.63 (18)
C9—N2—C1—N1	176.89 (11)	C2—C3—C4—N1	1.07 (19)
C2—N2—C1—N1	−20.61 (18)	C1—N1—C4—C8	−160.96 (13)
C1—N2—C2—C3	37.78 (18)	C1—N1—C4—C3	19.4 (2)
C9—N2—C2—C3	−159.53 (12)	C1—N2—C9—C14	−59.84 (17)
N2—C2—C3—C5	156.71 (13)	C2—N2—C9—C10	−41.07 (17)

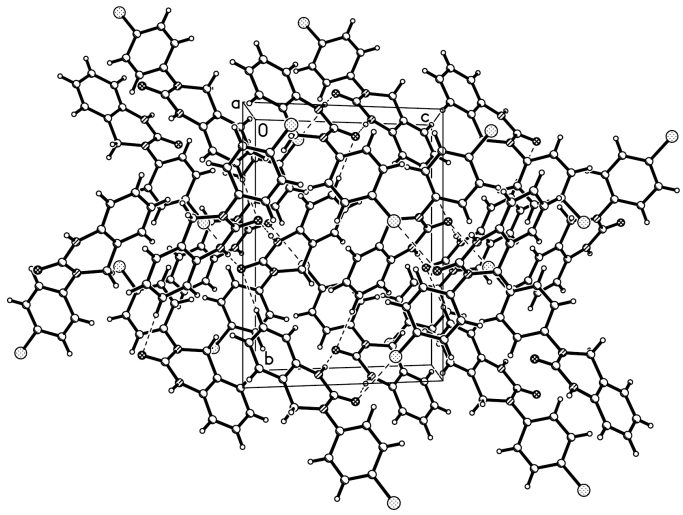


Figure 2
The molecular packing in the crystal structure of (I). Dashed lines indicate N—H···O and C—H···O interactions.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.861 (18)	2.012 (19)	2.8699 (15)	174.5 (16)
C7—H7···O1^{ii}	0.95	2.51	3.4477 (18)	169

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

The H atom on atom N1 was refined isotropically, with the N—H bond length restrained to 0.86 (2) Å; other H atoms were positioned geometrically and refined as riding, with C—H = 0.95–0.99 Å and U_{iso}(H) = 1.2U_{eq}(C).

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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References

Billimoria, A. D. & Cava, M. P. (1994). *J. Org. Chem.* **59**, 6772–6782.
Blackman, A., Hambley, T. W., Picker, R., Taylor, W. C. & Thirasana, N. (1987). *Tetrahedron Lett.* **28**, 5561–5564.
Brana, M. F., Castellano, J. M., Keilahauer, G., Machuca, A., Martin, Y., Redondo, C., Schlick, E. & Walker, N. (1994). *Anti-Cancer Drugs Des.* **9**, 527–538.
Helissey, P., Cros, S. & Giorgi-Renault, S. (1994). *Anti-Cancer Drugs Des.* **9**, 51–57.
Ibrahim, E. S., Montgomerie, A. M., Sneddon, A. H., Proctor, G. R. & Green, B. (1988). *Eur. J. Med. Chem.* **23**, 183–188.
Jacobson, R. (1998). Private communication to Rigaku Corporation.
Lorente, A., Galan, C., Fonseea, I. & Sanz-Aparicio, J. (1995). *Can. J. Chem*, **73**, 1546–1555.
McMurry, J. E. (1983). *Acc. Chem. Res.* **16**, 405–411.
Rigaku (2000). *CrystalClear.* Rigaku Corporation, Tokyo, Japan.

Rigaku/MSC (2003). *CrystalStructure.* Rigaku/MSC, 9009 New Trails Drive, The Woodlands, TX 77381–5209, USA.
Riou, J. F., Helissey, P., Grondard, L. & Giorgi-Renault, S. (1991). *Mol. Pharmacol.* **40**, 699–706.
Sheldrick, G. M. (1997). *SHELXTL.* Version 5.1. Bruker AXS Inc. Madison, Wisconsin, USA.
Shi, D. Q., Chen, J. X., Chai, W. Y., Chen, W. X. & Kao, T. Y. (1993). *Tetrahedron Lett.* **34**, 2963–2964.
Shi, D. Q., Lu, Z. S. & Dai, G. Y. (1998). *Synth. Commun.* **28**, 1073–1078.
Shi, D. Q., Mu, L. L., Lu, Z. S. & Dai, G. Y. (1997). *Synth. Commun.* **27**, 4121–4129.
Shi, D. Q., Wang, J. X., Shi, C. L., Rong, L. C. & Hu, H. W. (2004). *Synlett*, pp. 1098–1100.
Wu, H., Shi, D.-Q., Shi, C.-L., Wang, X.-S., Hu, H.-W. & Yu, K.-B. (2004). *Acta Cryst.* E**60**, o9–o11.