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6-Chloro-3-(4-chlorophenyl)-3,4-dihydroquinazolin-2(1*H*)-one acetone hemisolvate

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Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.003 Å
Disorder in solvent or counterion
R factor = 0.047
wR factor = 0.112
Data-to-parameter ratio = 15.8

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

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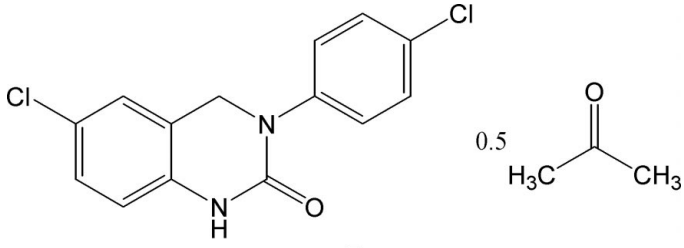
The title compound, $C_{14}H_{10}Cl_2N_2O\cdot0.5C_3H_6O$, was synthesized by the reaction of 5-chloro-*N*-(4-chlorophenyl)-2-nitrobenzylamine with triphosgene, induced by a low-valent titanium reagent ($TiCl_4$/Zn). The dihydroquinazoline ring exhibits a boat conformation. N—H···O hydrogen bonds form centrosymmetric dimers. There are also some weak C—H···O interactions.

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 anticancer 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). As part of our continuing interest in this field, the structure of the title compound, (I), has been investigated.



(I)

A molecular view of (I) is shown in Fig. 1. The bond lengths and angles have the usual values found for structurally similar molecules in the Cambridge Structural Database (CSD; Version 5.24; Allen, 2002). The heterocyclic ring has a boat conformation (Fig. 1). Atoms C3, C4, N2 and C1 are coplanar, while atoms N1 and C2 deviate from the plane by 0.085 (2) and 0.189 (2) Å, respectively. Because of the existence of a conjugated system, the N1—C4 [1.399 (2) Å] and N1—C1 [1.355 (2) Å] distances are significantly shorter than the typical Csp^2—N bond distance (1.426 Å; Lorente *et al.*, 1995). Atoms N1 and N2 are coplanar. In the crystal structure there are some acetone solvent molecules, but these solvent molecules are not involved in intermolecular hydrogen bonds with (I). However, an intermolecular hydrogen bond is formed between the amine H atom and carbonyl atom O1 (Table 1); these dimers pack along the *a* axis. In addition, some weak

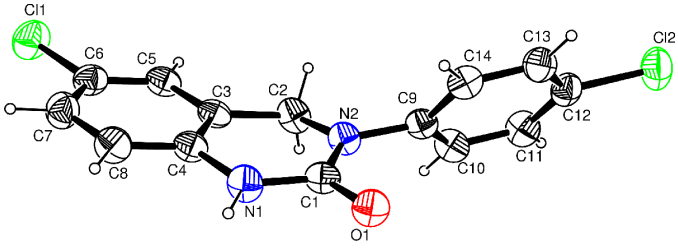


Figure 1
The molecular structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme.

intermolecular C—H···O interactions may be considered (Table 1).

Experimental

The title compound, (I), was prepared by the reaction of 5-chloro-*N*-(4′-chlorophenyl)-2-nitrobenzylamine (0.61 g) with triphosgene (0.89 g), induced by a low-valent titanium reagent ($TiCl_4$/Zn) (yield 81%; m.p.589–590 K). Single crystals suitable for X-ray diffraction were obtained by evaporation of an ethanol–acetone solution. IR (KBr, cm^{-1}): 3222 (NH), 1674 (CO), 1593, 1489, 824, 748 (phenyl ring); ^{1}H NMR (DMSO-d_6): 4.82 (2H, *s*, CH_2), 6.88 (1H, *d*, J = 8.8 Hz, ArH), 7.24–7.28 (2H, *m*, ArH), 7.38–7.46 (4H, *m*, ArH), 9.79 (1H, *s*, NH).

Crystal data

$C_{14}H_{10}Cl_2N_2O{\cdot}0.5C_3H_6O$
M_r = 322.18
Monoclinic, $P2_1/n$
a = 5.806 (2) Å
b = 13.250 (4) Å
c = 18.810 (6) Å
β = 90.802 (7)°
V = 1447.0 (8) Å^3
Z = 4
D_x = 1.479 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6413 reflections
θ = 3.1–27.5°
μ = 0.45 mm^{-1}
T = 193 (2) K
Block, colorless
0.65 × 0.35 × 0.30 mm

Data collection

Rigaku Mercury diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.759, T_{max} = 0.877
15 816 measured reflections
3315 independent reflections
3067 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 27.5°
$h = -6 \rightarrow 7$
$k = -15 \rightarrow 17$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.112
S = 1.14
3315 reflections
210 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0428P)^2 + 0.7556P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.007
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	1.99	2.847 (2)	175
C2—H2A···O1^{ii}	0.97	2.41	3.374 (2)	176

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

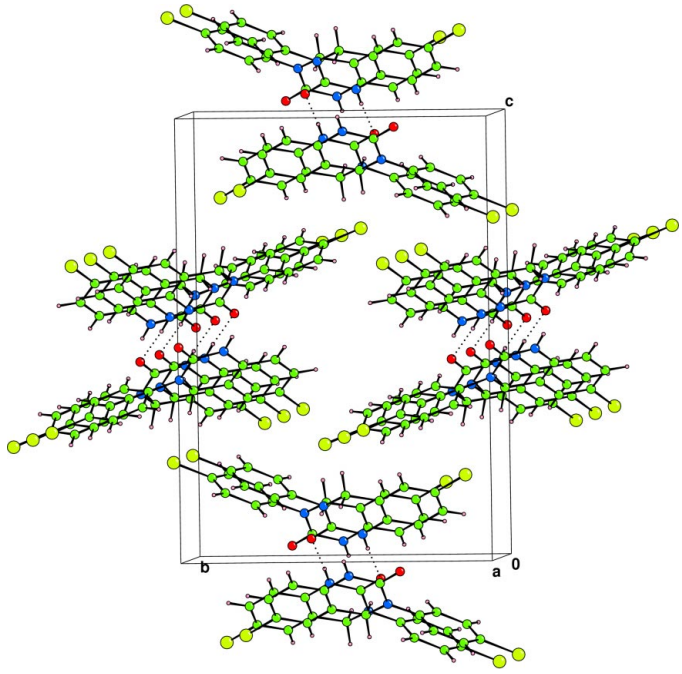


Figure 2
The crystal packing of (I). Dashed lines indicate hydrogen bonds.

All H atom were constrained to ride on their parent atoms, with N—H = 0.86 Å, C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2U_{eq}(N,C) or 1.5U_{eq}(C_{methyl}). The acetone solvent is disordered equally over an positions around the inversion center. The disorder was refine-drefined with the aid of restraints on geometry and displacement parameters.

Data collection: *CrystalClear* (Rigaku, 2000); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2003); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *SHELXL*97.

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