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9-Chloro-2,3-diphenyl-5,6-dihydroimidazo-[1,2-*c*]quinazolin-5-one *N*,*N*-dimethylformamide solvate

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Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.002 Å
R factor = 0.046
wR factor = 0.112
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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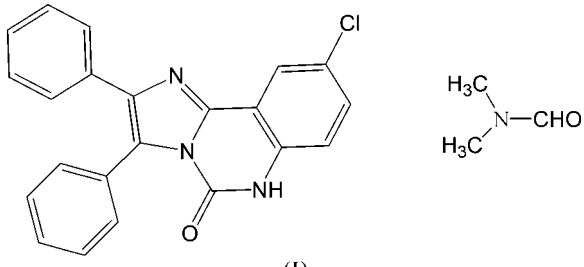
The title compound, $C_{22}H_{14}ClN_3O\cdot C_3H_7NO$, was synthesized by the reaction of 2-(5-chloro-2-nitrophenyl)-4,5-diphenylimidazole with triphosgene, induced by a low-valent titanium reagent ($TiCl_4$/Zn). The dihydropyrimidine ring adopts a skew-boat conformation.

Comment

Quinazolines are an important class of compounds found in many naturally occurring products (*e.g.* himckdentine 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 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). In the course of our work on the application of low-valent titanium reagents in the preparation of bioactive heterocyclic compounds, we have reported the synthesis of quinazoline-4(3*H*)-ones (Shi *et al.*, 2003), imidazo[1,2-*c*]quinazolines (Shi, Wang *et al.*, 2004) and pyrroles (Shi, Shi *et al.*, 2004). We report here the crystal structure of the title compound, (I).



(I)

The pyrimidine ring adopts a skew-boat conformation; atoms C1, C6, C5 and N3 are coplanar, while atoms N2 and C4 deviate from this plane by 0.111 (2) and 0.067 (2) Å, respectively. A similar conformation was observed in the structure of 5,5-dimethyl-2,3-diphenyl-5,6-dihydroimidazo[1,2-*c*]quinazole (Wu *et al.*, 2004). The benzene rings C5–C10, C11–C16 and C17–C22 form dihedral angles of 5.58 (2), 28.68 (3) and 79.41 (2)°, respectively, with the imidazole ring. The dihedral angle between the C11–C16 and C17–C22 phenyl rings is 83.57 (2)°. In addition, because of the existence of a conjugated system, the N2—C4 [1.4125 (19) Å], N3—C4 [1.356 (2) Å] and N3—C5 [1.389 (2) Å] distances are significantly shorter than the typical Csp^2—N bond distance (1.426 Å; Lorente *et al.*, 1995). An intermolecular hydrogen bond is formed between the N3/H3 amine group and atom O2 of the solvent *N*,*N*-dimethylformamide (Fig. 2 and Table 2).

Experimental

The title compound, (I), was prepared by the reaction of 2-(5-chloro-2-nitrophenyl)-4,5-diphenylimidazole (0.75 g, 2 mmol) with triphosgene (0.89 g, 3 mmol) induced by a low-valent titanium reagent ($TiCl_4$/Zn). Yield 82%, m.p. 593–595 K. Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an *N*,*N*-dimethylformamide solution. ^{1}H NMR (DMSO-d_6): δ 2.91 (3H, *s*, CH_3N), 2.98 (3H, *s*, CH_3N), 7.05 (1H, *d*, J = 8.0 Hz, ArH), 7.25–7.31 (7H, *m*, ArH), 7.45– 7.50 (3H, *m*, ArH), 7.62 (1H, *d*, J = 8.0 Hz, ArH), 8.04 (1H, *s*, CHO), 8.50 (1H, *s*, ArH), 9.58 (1H, *s*, NH).

Crystal data

$C_{22}H_{14}ClN_3O\cdot C_3H_7NO$
M_r = 444.91
Monoclinic, $P2_1/n$
a = 8.9157 (10) Å
b = 11.2560 (13) Å
c = 21.496 (3) Å
β = 97.370 (3)°
V = 2139.4 (4) Å^3
Z = 4
D_x = 1.381 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8583 reflections
θ = 3.2–27.5°
μ = 0.21 mm^{-1}
T = 193 (2) K
Block, colorless
0.58 × 0.30 × 0.24 mm

Data collection

Rigaku Mercury diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.888, T_{max} = 0.951
23 135 measured reflections
4886 independent reflections
4260 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 27.5°
$h = -11 \rightarrow 11$
$k = -13 \rightarrow 14$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.112
S = 1.10
4886 reflections
296 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 0.9166P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C4	1.2151 (18)	N2—C3	1.3963 (19)
N1—C1	1.3067 (19)	N2—C4	1.4125 (19)
N1—C2	1.3885 (19)	N3—C4	1.356 (2)
N2—C1	1.3833 (18)	N3—C5	1.389 (2)
C4—N2—C1—C6	7.3 (2)	C4—N3—C5—C6	4.8 (2)
C5—N3—C4—N2	−2.0 (2)	N3—C5—C6—C1	−1.4 (2)
C1—N2—C4—N3	−4.2 (2)	N2—C1—C6—C5	−4.1 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C20—H20···Cl1^{i}	0.95	2.90	3.685 (2)	141
C23—H23···O1^{ii}	0.95	2.50	3.283 (2)	140
N3—H3···O2^{ii}	0.92 (2)	1.86 (2)	2.770 (2)	178 (2)
N3—H3···C23^{ii}	0.92 (2)	2.57 (2)	3.400 (2)	151 (2)

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

The amine H atom was located in a differences Fourier map and refined isotropically. The C-bound H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C—H distances in the range 0.95–0.98 Å; 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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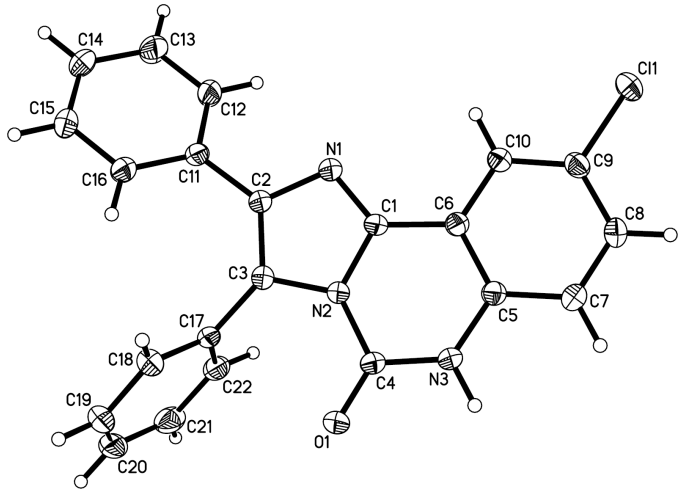

Figure 1
The molecular structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme.

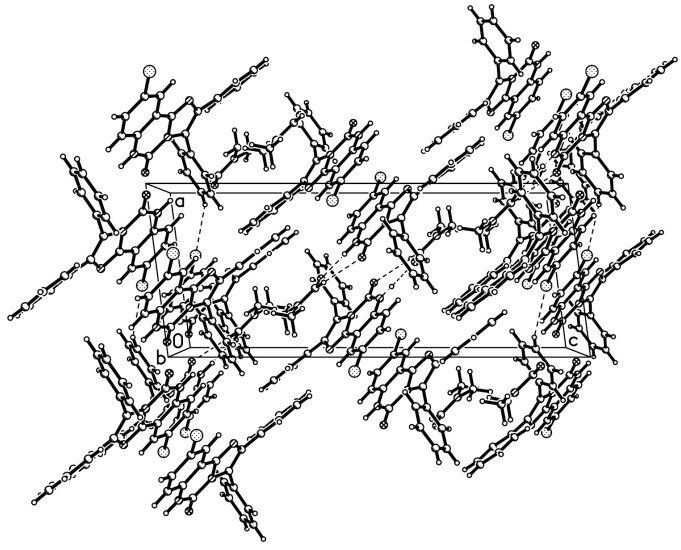

Figure 2
A molecular packing diagram for (I). The broken lines indicate hydrogen bonds and short contacts.

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