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9-Chloro-5,5-dimethyl-2,3-diphenyl-2,3-dihydroimidazo[1,2-*c*]quinazoline

Daqing Shi,[a]* Juxian Wang,[a] Chunling Shi,[a] Liangce Rong,[a] Xiangshan Wang[a] and Hongwen Hu[b]

[a]Department of Chemistry, Xuzhou Normal University, Xuzhou 221116, People's Republic of China, and [b]Department of Chemistry, Nanjing University, Nanjing 210093, People's Republic of China

Correspondence e-mail: dqshi@163.com

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.109
Data-to-parameter ratio = 14.7

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

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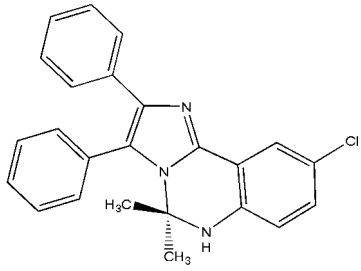
The title compound, $C_{24}H_{20}ClN_3$, (I), was synthesized by the reaction of 4,5-diphenyl-2-(2-nitro-5-chlorophenyl)imidazole with acetone, induced by a low-valent titanium reagent ($TiCl_4$/Zn). X-ray analysis reveals that (I) contains a pyrimidine ring in a distorted boat conformation.

Comment

Quinazolines are an important class of compounds 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.*, 1998). 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.*, 2003). We report here the crystal structure of 9-chloro-5,5-dimethyl-2,3-diphenyl-2,3-dihydroimidazo[1,2-*c*]quinazoline, (I), synthesized by the reaction of 4,5-diphenyl-2-(2-nitro-5-chlorophenyl)-imidazole with acetone, induced by a low-valent titanium reagent ($TiCl_4$/Zn).



(I)

In (I), atoms N1, C5, C6, C7, N1 and C10 form a pyrimidine ring, with an interatomic distance of 1.440 (3) Å for N1—C10 and 1.487 (2) Å for N2—C10, which show that these C—N bonds are single. The pyrimidine ring adopts a distorted boat conformation (Figs. 1 and 2); atoms C5, C6, C7 and N2 are coplanar, while atoms N1 and C10 deviate from this plane by 0.157 (1) and 0.347 (1) Å, respectively. The dihedral angle between the C13—C18 and C19—C24 phenyl rings is 79.31 (2)°. In addition, because of conjugation, the distances N1—C5 [1.378 (2) Å], N2—C7 [1.364 (2) Å] and N2—C8 [1.388 (2) Å] are significantly shorter than the typical Csp^2—N bond distance (1.426 Å; Lorente *et al.*, 1995). The molecules are linked by an N—H···N hydrogen bond (Table 2), forming a chain along the *c* axis (Fig. 2).

Experimental

The title compound (I), was prepared by the reaction of 4,5-diphenyl-2-(2-nitro-5-chlorophenyl)imidazole with acetone induced by low-valent titanium reagent ($TiCl_4$/Zn) (m.p. 528–529 K). Single crystals

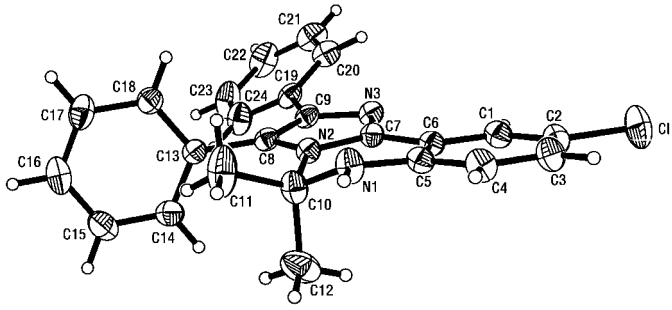


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

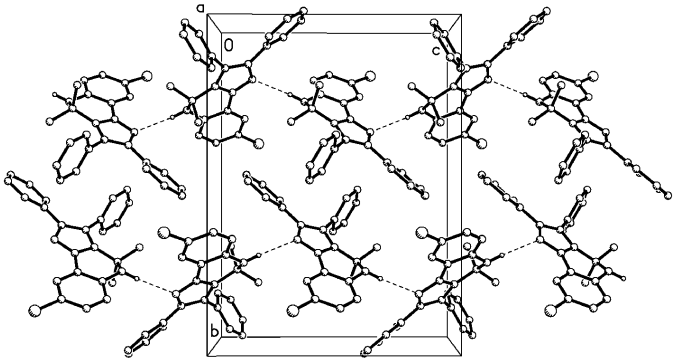


Figure 2
A molecular packing diagram of (I). H atoms have been omitted for clarity.

suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{24}H_{20}ClN_3$
M_r = 385.88
Monoclinic, $P2_1/c$
a = 8.955 (1) Å
b = 17.568 (2) Å
c = 13.075 (2) Å
β = 90.26 (1)°
V = 2056.9 (4) Å^3
Z = 4
D_x = 1.246 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 29 reflections
θ = 3.0–14.2°
μ = 0.20 mm^{-1}
T = 296 (2) K
Block, colorless
0.50 × 0.50 × 0.30 mm

Data collection

Siemens $P4$ diffractometer
ω scans
Absorption correction: ψ scan (*XSCANS*; Siemens, 1994)
T_{min} = 0.893, T_{max} = 0.945
4393 measured reflections
3833 independent reflections
2274 reflections with $I > 2\sigma(I)$
R_{int} = 0.012
θ_{max} = 25.5°
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 21$
$l = -15 \rightarrow 15$
3 standard reflections
every 97 reflections
intensity decay: 0.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.109
S = 0.88
3833 reflections
260 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0609P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0099 (11)

Table 1
Selected geometric parameters (Å, °).

N1—C5	1.378 (2)	N3—C7	1.328 (2)
N1—C10	1.440 (3)	N3—C9	1.386 (2)
N2—C7	1.364 (2)	C6—C7	1.448 (3)
N2—C8	1.388 (2)	C8—C9	1.378 (2)
N2—C10	1.487 (2)		
C5—N1—C10	122.50 (16)	N1—C5—C6	118.52 (19)
C7—N2—C8	107.12 (14)	N3—C7—N2	111.86 (17)
C7—N2—C10	121.96 (16)	N3—C7—C6	127.74 (17)
C8—N2—C10	129.94 (15)	N2—C7—C6	120.40 (16)
C7—N3—C9	105.19 (15)	N2—C8—C13	123.84 (15)
N1—C5—C4	122.17 (18)		
C10—N1—C5—C4	−155.6 (2)	C5—N1—C10—N2	−42.6 (3)
C1—C6—C7—N3	−6.6 (3)	C9—C8—C13—C18	−83.6 (3)
C1—C6—C7—N2	173.91 (18)	N2—C8—C13—C18	95.8 (2)
N2—C8—C9—N3	1.1 (2)	C8—C9—C19—C20	152.8 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···N3^{i}	0.90 (2)	2.26 (2)	3.147 (2)	172.3 (19)

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

The H atom on N was refined isotropically, with the N—H bond length restrained to 0.90 Å; other H atoms were positioned geometrically and refined as riding [C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2 U_{eq}(C)].

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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