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5,5-Dimethyl-2,3-diphenyl-5,6-dihydroimidazo[1,2-c]quinazoline

Hui Wu,[a] Daqing Shi,[a]* Chunling Shi,[b] Juxian Wang,[b] Xiangshan Wang,[b] Hongwen Hu[c] and Kaibei Yu[d]

[a]Key Laboratory of Biotechnology of Medical Plants, Jiangsu, Xuzhou 221116, People's Republic of China, [b]Department of Chemistry, Xuzhou Normal University, Xuzhou 221116, People's Republic of China, [c]Department of Chemistry, Nanjing University, Nanjing 210093, People's Republic of China, and [d]Chengdu Institute of Organic Chemistry, Chinese Academy of Sciences, Chengdu 610041, People's Republic of China

Correspondence e-mail: dqshi@263.net

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Key indicators

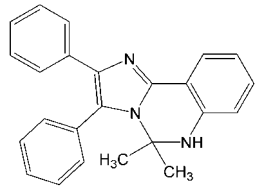
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.108
Data-to-parameter ratio = 14.1

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

The title compound, $C_{24}H_{21}N_3$, was synthesized by the reaction of 4,5-diphenyl-2-(2-nitrophenyl)imidazole with acetone, induced by a low-valent titanium reagent ($TiCl_4$/Zn). The dihydropyrimidine ring shows a skew-boat conformation.

Comment

Quinazolines are an important class of compounds found in many naturally occurring products (*e.g.* hinckdentine A; Blackman *et al.*, 1987; Billimmoria & 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 the reductive coupling of carbonyl compounds and are attracting increasing interest in organic synthesis (McMurry, 1983; Shi *et al.*, 1993, 1997, 1998, 2003). We report here the crystal structure of the title compound, (I), synthesized by the reaction of 4,5-diphenyl-2-(2-nitrophenyl)imidazole with acetone, induced by a low-valent titanium reagent ($TiCl_4$/Zn).



(I)

Atoms N1, C6, C1, C8, N2 and C7 form a dihydropyrimidine ring, with an interatomic distance of 1.454 (2) Å for N1—C7 and 1.492 (2) Å for N2—C7, showing that these C—N bonds are single. The dihydropyrimidine ring adopts a skew-boat conformation; atoms C6, C1, C8 and N1 are coplanar, while atoms N2 and C7 deviate from this plane by 0.274 (1) and 0.690 (1) Å, respectively. The dihedral angle between phenyl rings C11–C16 and C17–C22 is 76.40 (2)°. In addition, because of the existence of a conjugated system, the distances N1—C6 [1.385 (2) Å], N2—C10 [1.389 (2) Å] and N2—C8 [1.371 (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 N1—H1N···N3^{i} hydrogen bond (Table 2), forming zigzag chains along the *c* axis (Fig. 2).

Experimental

The title compound, (I), was prepared by the reaction of 4,5-diphenyl-2-(2-nitrophenyl)imidazole with acetone induced by a low-valent titanium reagent ($TiCl_4$/Zn) (m.p. 513–514 K). Single crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

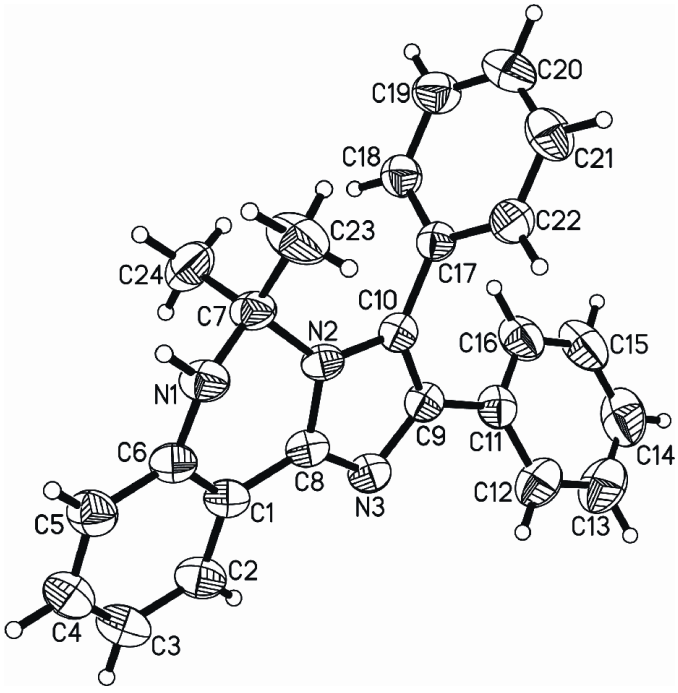


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

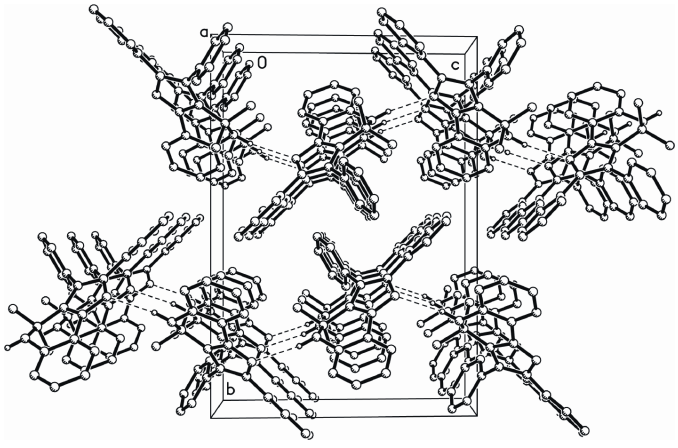


Figure 2
The molecular packing diagram for (I).

Crystal data

$C_{24}H_{21}N_3$
M_r = 351.44
Monoclinic, $P2_1/c$
a = 8.140 (1) Å
b = 18.522 (2) Å
c = 12.963 (2) Å
β = 94.32 (1)°
V = 1948.8 (3) Å^3
Z = 4
D_x = 1.198 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 35 reflections
θ = 3.8–14.8°
μ = 0.07 mm^{-1}
T = 296 (2) K
Block, colorless
0.58 × 0.56 × 0.50 mm

Data collection

Siemens $P4$ diffractometer
ω scans
Absorption correction: none
4071 measured reflections
3527 independent reflections
2148 reflections with $I > 2\sigma(I)$
R_{int} = 0.010
θ_{max} = 25.3°
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 22$
$l = -15 \rightarrow 15$
3 standard reflections
every 97 reflections
intensity decay: 3.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.108
S = 0.96
3527 reflections
251 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0558P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0177 (15)

Table 1
Selected geometric parameters (Å, °).

N1—C6	1.385 (2)	N3—C9	1.382 (2)
N1—C7	1.454 (2)	C1—C8	1.454 (2)
N2—C8	1.3714 (19)	C9—C10	1.369 (2)
N2—C10	1.389 (2)	C9—C11	1.480 (2)
N2—C7	1.492 (2)	C10—C17	1.482 (2)
N3—C8	1.326 (2)		
C6—N1—C7	120.62 (14)	N2—C7—C23	112.12 (16)
C8—N2—C10	107.00 (13)	N1—C7—C24	112.39 (16)
C8—N2—C7	121.77 (14)	N2—C7—C24	107.24 (14)
C10—N2—C7	130.31 (13)	N3—C8—N2	111.56 (15)
C8—N3—C9	105.11 (13)	N3—C8—C1	128.48 (15)
N1—C6—C5	122.29 (16)	N2—C8—C1	119.94 (14)
N1—C6—C1	118.19 (17)	N3—C9—C11	120.30 (14)
N1—C7—N2	105.71 (13)	C9—C10—N2	105.26 (14)
N1—C7—C23	106.98 (15)	N2—C10—C17	124.83 (13)
C7—N1—C6—C5	−150.22 (17)	C10—N2—C7—C23	−43.1 (2)
C7—N1—C6—C1	34.7 (2)	C8—N2—C7—C24	−86.93 (18)
C4—C5—C6—N1	−176.38 (17)	C9—N3—C8—N2	−1.72 (18)
C2—C1—C6—N1	176.95 (16)	C9—N3—C8—C1	176.59 (16)
C8—C1—C6—N1	−1.3 (2)	C10—N2—C8—N3	2.48 (18)
C6—N1—C7—N2	−48.1 (2)	C7—N2—C8—N3	172.58 (14)
C6—N1—C7—C23	−167.78 (17)	C6—C1—C8—N3	169.57 (16)
C6—N1—C7—C24	68.5 (2)	C6—C1—C8—N2	−12.2 (2)
C8—N2—C7—N1	33.2 (2)	C7—N2—C10—C9	−171.07 (16)
C10—N2—C7—N1	−159.28 (15)	C7—N2—C10—C17	8.3 (3)
C8—N2—C7—C23	149.38 (16)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···N3^{i}	0.883 (18)	2.260 (19)	3.133 (2)	169.5 (17)

Symmetry code: (i) $x, \frac{3}{2} - y, z - \frac{1}{2}$.

The H atom on nitrogen was refined isotropically, with the N—H bond length restrained to 0.88 (2) Å; other H atoms were positioned geometrically and refined as riding [C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2U_{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*.

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