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5,5-Dimethyl-8,9-methylenedioxy-2,3-diphenyl-5,6-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@263.net

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.048
wR factor = 0.127
Data-to-parameter ratio = 13.9

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

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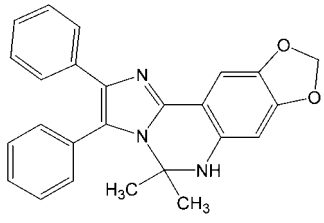
The title compound $C_{25}H_{21}N_3O_2$, (I), was synthesized by the reaction of 4,5-diphenyl-2-(2-nitro-4,5-methylenedioxyphenyl)imidazole with acetone, induced by a low-valent titanium reagent ($TiCl_4$/Zn). X-ray analysis reveals that (I) contains a pyrimidine ring in a twist-boat conformation.

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.*, 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). We report here the crystal structure of 5,5-dimethyl-8,9-methylenedioxy-2,3-diphenyl-5,6-dihydroimidazo[1,2-*c*]quinazoline, (I), synthesized by the reaction of 4,5-diphenyl-2-(2-nitro-4,5-methylenedioxyphenyl)imidazole with acetone, induced by a low-valent titanium reagent ($TiCl_4$/Zn).



(I)

In (I), atoms N1, C4, C5, C8, N2 and C9 form a pyrimidine ring, with interatomic distances of 1.437 (3) Å for N1—C9 and 1.488 (3) Å for N2—C9, which indicate that these C—N bonds are single. The pyrimidine ring adopts a twist-boat conformation; atoms C4, C5, C8 and N2 are coplanar, while atoms N1 and C9 deviate from this plane by −0.142 (2) and 0.418 (1) Å, respectively. The dihedral angle between the C12—C17 and C18—C23 phenyl rings is 75.91 (2)°. In addition, because of the existence of a conjugated system, the N1—C4 [1.383 (3) Å], N2—C8 [1.363 (3) Å] and N2—C10 [1.396 (3) Å] distances are significantly shorter than the typical Csp^2—N bond distance (1.426 Å; Lorente *et al.*, 1995). The molecules are linked by N—H···N hydrogen bonds (Table 2), to form extended chains in the *c* direction (Fig. 2).

Experimental

The title compound, (I), was prepared by the reaction of 4,5-diphenyl-2-(2-nitro-4,5-methylenedioxyphenyl)imidazole with acetone,

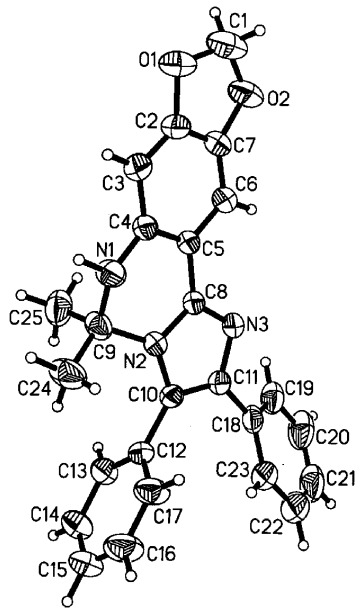


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

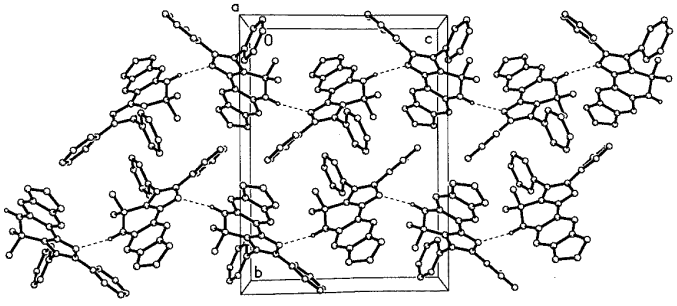


Figure 2
The molecular packing in the crystal structure of (I). Hydrogen bonds are shown as dashed lines.

induced by a low-valent titanium reagent ($TiCl_4$/Zn) (m.p. 512–513 K). Single crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{25}H_{21}N_3O_2$
M_r = 395.45
Monoclinic, $P2_1/c$
a = 9.594 (2) Å
b = 16.928 (4) Å
c = 12.865 (3) Å
β = 95.73 (2)°
V = 2079.1 (8) Å^3
Z = 4
D_x = 1.263 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 29 reflections
θ = 2.8–13.9°
μ = 0.08 mm^{-1}
T = 296 (2) K
Block, colorless
0.48 × 0.38 × 0.26 mm

Data collection

Siemens $P4$ diffractometer
ω scans
4424 measured reflections
3873 independent reflections
1824 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 25.5°
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 20$
$l = -15 \rightarrow 15$
3 standard reflections
every 97 reflections
intensity decay: 7.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.127
S = 0.82
3873 reflections
278 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0654P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0096 (12)

Table 1
Selected geometric parameters (Å, °).

N1—C4	1.383 (3)	N3—C8	1.327 (3)
N1—C9	1.437 (3)	N3—C11	1.388 (3)
N2—C8	1.363 (3)	C5—C8	1.445 (3)
N2—C10	1.396 (3)	C10—C11	1.372 (3)
N2—C9	1.488 (3)		
C4—N1—C9	120.2 (2)	N3—C8—C5	127.4 (2)
C8—N2—C10	107.08 (18)	N2—C8—C5	120.6 (2)
C8—N2—C9	120.06 (19)	N1—C9—N2	106.93 (19)
C10—N2—C9	130.88 (18)	N1—C9—C24	110.0 (2)
C8—N3—C11	104.96 (18)	N2—C9—C24	114.4 (2)
N1—C4—C5	118.8 (2)	N1—C9—C25	109.7 (2)
N1—C4—C3	120.5 (2)	N2—C9—C25	106.1 (2)
N3—C8—N2	112.0 (2)		
C9—N1—C4—C5	−31.3 (3)	C10—N2—C8—N3	−1.3 (2)
C9—N1—C4—C3	152.6 (2)	C9—N2—C8—N3	−166.9 (2)
C2—C3—C4—N1	175.6 (2)	C9—N2—C8—C5	13.3 (3)
N1—C4—C5—C6	−176.5 (2)	C4—N1—C9—N2	47.1 (3)
N1—C4—C5—C8	1.5 (3)	C4—N1—C9—C24	171.9 (2)
C11—N3—C8—N2	0.5 (2)	C10—N2—C9—N1	160.5 (2)
C11—N3—C8—C5	−179.8 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···N3^{i}	0.92 (3)	2.24 (3)	3.135 (3)	166 (2)

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

The H atom on the N atom was refined isotropically, with the N—H bond length restrained to 0.92 (3) Å; other H atoms were positioned geometrically and refined as riding [C—H = 0.93–0.97 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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References

Blackman, A., Hambley, T. W., Picker, R., Taylor, W. C. & Thirasana, N. (1987). *Tetrahedron Lett.* **28**, 5561–5564.
Billimoria, A. D. & Cava, M. P. (1994). *J. Org. Chem.* **59**, 6777–6782.
Brana, M. F., Castellano, J. M., Keilhauer, 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.
Lorente, A., Galan, C., Fonseca, I. & Sanz-Aparicio, J. (1995). *Can. J. Chem.* **73**, 1546–1555.
McMurry, J. E. (1983). *Acc. Chem. Res.* **16**, 405–411.
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., Mu, L. L. & 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., Rong, L. C., Wang, J. X., Zhuang, Q. Y., Wang, X. S. & Hu, H. W. (2003). *Tetrahedron Lett.* **44**, 3199–3201.
Siemens (1994). *XSCANS*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.