Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-[(5-Chloro-2-hydroxyphenyl)(phenyl)-methyl]-2-(2-pyridyl)-1*H*-benzimidazole

Pan-Ming Jian,[a] Yi-Zhi Li,[b] Xiao-Li Wu,[a] Yong-Xiang Ma,[a]* Da-Hua Pan[a] and Bao-Hua Chen[a]

[a]National Laboratory of Applied Organic Chemistry, Lanzhou University, Lanzhou 730000, People's Republic of China, and [b]Coordination Chemistry Institute, State Key Laboratory of Coordination Chemistry, Nanjing University, Nanjing 210093, People's Republic of China

Correspondence e-mail: llyyjz@nju.edu.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.034
wR factor = 0.092
Data-to-parameter ratio = 11.6

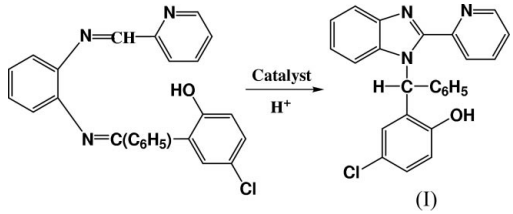
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 14 April 2003
Accepted 6 May 2003
Online 16 May 2003

The title compound, $C_{25}H_{18}ClN_3O$, results from the intramolecular reaction of a difunctional Schiff base in the presence of acid as a catalyst. There is a chiral C atom in the molecule, but the crystal structure is a racemic mixture. There is one strong intermolecular O—H···N hydrogen bond and three weak C—H···N interactions (two intra- and one intermolecular), leading to the formation of a helical chain of molecules.

Comment

The preparation of benzimidazoles has attracted some attention due to their varied physiological characteristics, such as anticancer agents, fungicides, antichagasic drugs, inhibitors, and plant-growth regulators (Zhou & Hassner, 2001; Matsuno *et al.*, 2000; Kucukbay *et al.*, 2001; Purygin *et al.*, 2000; Bag *et al.*, 1996).



(I)

In general, benzimidazole derivatives are obtained by the reaction of an *o*-phenylenediamine with a carboxylic acid, ester, amide, nitrile *etc.*, or by the palladium-catalysed carbonylation, coupling and cyclization of haloaromatics and *o*-phenylenediamines. Recently, Alajarin *et al.* (1999) described a [4 + 2] intramolecular cycloaddition of ketimines with imines to form benzimidazo[1,2-*b*]isoquinolines, but it is a tedious procedure requiring expensive reagents. This paper reports a novel method for the synthesis of 1,2-benzimidazoles in good yields, by hydrogen-transfer cyclization between azomethine groups in the presence of acidic catalysts under mild conditions.

There is a chiral C atom in the molecule of the title compound, (II); however, it crystallizes as a racemic mixture in the centrosymmetric space group *P* 21/*c*. The dihedral angle between the benzimidazole ring system and the pyridyl ring is 24.29 (8)°. The bond angles H13—C13—N2, H13—C13—C20 and H13—C13—C14 are 103.2 (8), 110.0 (8) and 105.7 (8)°, respectively. There is one strong intermolecular O—H···N hydrogen bond and three weak C—H···N interactions (two intra- and one intermolecular), leading to the formation of a helical chain of molecules (Fig. 3).

Experimental

The title compound, (II), was prepared as follows: in the presence of *p*-$CH_3C_6H_4SO_3H$, an intramolecular hydrogen-transfer cyclization of 1-*N*-(phenyl-5-chloro-2-hydroxylphenyl)methylene-2-*N*-(pyridin-2-

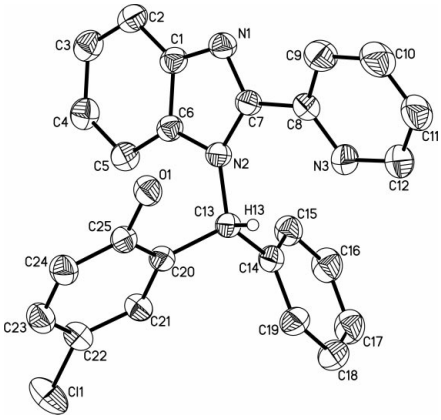


Figure 1
The molecular structure of title compound, with ellipsoids drawn at the 50% probability level.

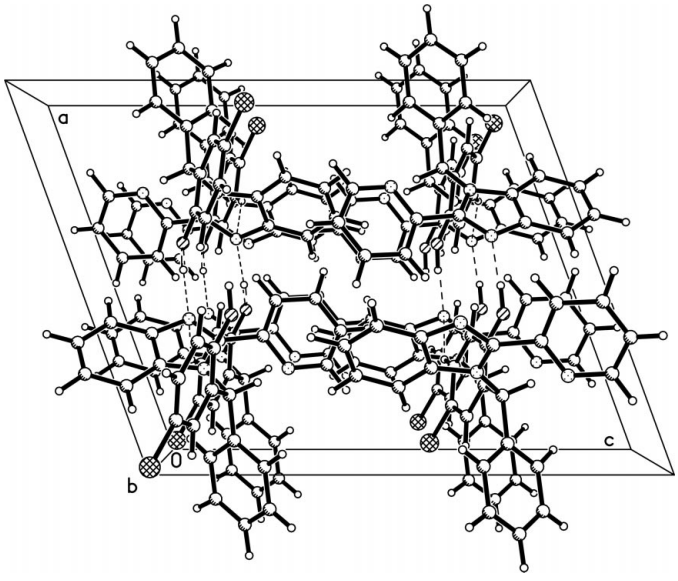


Figure 2
The packing of the title compound, viewed down the *b* axis.

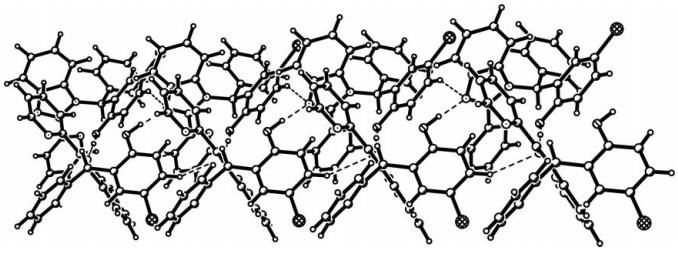


Figure 3
The crystal packing, showing the intermolecular interactions leading to the formation of two helical chains.

yl)methylene-1,2-phenylenedimine was carried out between the two C═N bonds of the asymmetrical difunctional Schiff base (I) to give cyclozation product (II). The hydrogen on the C atom of the aldimine transferred to the C atom of the ketoimine and formed a chiral center. The specific rotation of the product is zero, as it is racemic. A single crystal of the title compound was obtained by slow diffusion (1:1 MeOH–MeCN) over a period of one month.

Crystal data

$C_{25}H_{18}ClN_3O$	D_x = 1.330 Mg m^{-3}
M_r = 411.87	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25 reflections
a = 13.515 (2) Å	θ = 2.1–23.2°
b = 9.597 (2) Å	μ = 0.21 mm^{-1}
c = 16.875 (2) Å	T = 293 (2) K
β = 110.01 (1)°	Block, colorless
V = 2056.6 (6) Å^3	0.3 × 0.2 × 0.2 mm
Z = 4	

Data collection

Bruker *P*4 diffractometer	R_{int} = 0.045
ω scans	θ_{max} = 26.0°
Absorption correction: ψ scan (*XPREP* in *SHELXTL*; Bruker, 2000)	$h = -16 \rightarrow 16$
	$k = -11 \rightarrow 11$
T_{min} = 0.95, T_{max} = 0.96	$l = -19 \rightarrow 19$
10228 measured reflections	3 standard reflections
3982 independent reflections	every 97 reflections
3129 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	All H-atom parameters refined
$R[F^2 > 2\sigma(F^2)]$ = 0.034	$w = 1/[\sigma^2(F_o^2) + (0.02P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.092	$(\Delta/\sigma)_{max} < 0.001$
S = 1.01	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
3982 reflections	$\Delta\rho_{min}$ = −0.15 e Å^{-3}
343 parameters	

Table 1
Selected geometric parameters (Å, °).

N2—C13	1.4802 (16)	C20—C13	1.5251 (18)
C14—C13	1.5109 (19)	C13—H13	0.952 (13)
N2—C13—C14	112.56 (11)	N2—C13—H13	103.2 (8)
N2—C13—C20	110.27 (10)	C14—C13—H13	105.7 (8)
C14—C13—C20	114.47 (11)	C20—C13—H13	110.0 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1′···N1^{i}	0.99 (2)	1.77 (2)	2.7520 (16)	174.8 (17)
C13—H13···N3	0.952 (13)	2.317 (13)	2.9422 (19)	122.6 (9)
C15—H15···N2	1.005 (15)	2.513 (15)	2.8643 (19)	100.0 (10)
C23—H23···N1^{ii}	0.977 (19)	2.568 (19)	3.464 (2)	152.5 (14)

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

All H atoms were found in difference maps and refined isotropically. C—H bond lengths are in the range 0.952 (13)–1.058 (15) Å. The O—H bond length is 0.99 (2) Å.

Data collection: *XSCANS* (Bruker, 2000); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

This work was supported by the Natural Science Foundation (QT program) of the PRC and the Natural Science Foundation of Gansu Province of China (YS991-A25-001).

References

Alajarin, M., Vidal, A. Tovar, F. & Conesa, C. (1999). *Tetrahedron Lett.* **40**, 6127–6130.

Bag, S. K., Chakraborty, S. B. & Chaudhuri, S. R. (1996). *J. Ind. Chem. Soc.* **73**, 113–118.

Bruker (2000). XSCANS and *SHELXTL.* Bruker AXS Inc., Madison, Wisconsin, USA.

Kucukbay, H., Durmaz, R., Guven, M. & Gunal, S. (2001). *Arznein. Forsch.* **51**, 420–424.

Matsuno, T., Kato, M., Sasahara, H., Watanabe, T., Inaba, M., Takahashi, M., Yaguchi, S., Yoshioka, K., Sakato, M. & Kawashima, S. (2000). *Chem. Pharm. Bull.* **48**, 1778–1781.

Purygin, P. P., Kuz'mina, V. E., Sergeeva, L. I., Pan' kov, S. V., Belyakova, N. A. & Zarubin, Yu. P. (2000). *Pharm. Chem. J.* 34, 53–55.

Zhou, C. & Hassner, A. (2001). *Carbohydr. Res.* **333**, 313–326.