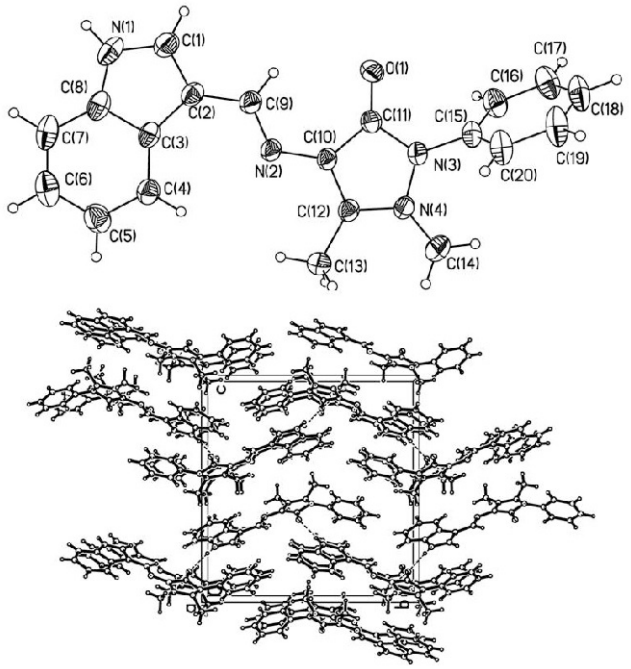
Crystal structure of 1,5-dimethyl-4-[(3-indoleyl)-methyleneamino]-2-phenyl-1*H*-pyrazol-3(2*H*)-one, $C_{20}H_{18}N_4O$

Jun-Ling Wang*, Jun-Ying Ma, Zheng-Hui Bai and Hua Lin

Chemical Engineering & Pharmaceutics College, Henan University of Science and Technology, Luoyang 471003, Henan Province, P. R. China

Received March 28, 2012, accepted June 12, 2012, available online August 09, 2012, CCDC no. 1267/3812



Abstract

$C_{20}H_{18}N_4O$, orthorhombic, $P2_12_12_1$ (no. 19), a = 6.8679(8) Å, b = 15.768(2) Å, c = 16.498(2) Å, V = 1786.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.0362, $wR_{ref}(F^2)$ = 0.0899, T = 296 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.19×0.27×0.28 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.79 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	50.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12686, 3321
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2530
$N(param)_{refined}$:	228
Programs:	SHELX [5]

Source of material

A solution of indole-3-carboxaldehyde (1.00 mmol) in ethanol (10 ml) was added to a stirred ethanol solution (10 ml) of 4-aminoantipyrine (1.15mmol) in a three-necked flask. The reaction mixture was refluxed for 4h, then the solution was cooled to room temperature. The yellow precipitated residue was removed from the solution by filtration and then washed with cold ethanol 3 times, and finally dried to obtain the title Schiff base (yield 84.0 %). Single crystals suitable for *X*-ray diffraction were obtained from an ethanol (95%) by slow evaporation at room temperature.

Discussion

Schiff bases are important organic ligands forming metal complexes and they have in general been reported to possess antimicrobial [1] and antitumour activities [2]. Indole derivatives are prepared for along time for a variety of biological activities such as CNS depressant, anticancerous, antibiotic, antihistaminic, anticonvulsants and many others [3]. A novel indole Schiff base was synthesized by the reaction of indole-3-carboxaldehyde and 4-aminoantipyrine. The title compound is built up by only the Schiff base molecules, with all bond lengths and bond angles in normal ranges [4] (Figure, top). The distance d(C9–N2) = 1.279(2) Å reveals the typical C=N Schiff base bond and d(C10–N2) = 1.393(2) Å visualizes a single bond. The molecule adopts a trans configuration about C=N, and the whole molecule is nearly coplanar with a dihedral angle of 6.269° between the two aromatic rings. The N–H···O hydrogen bonds connect the molecules to generate an one-dimensional supramolecular structure.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4*a*	–0.0619	0.6713	0.2499	0.087
H(4)	4*a*	0.5805	0.6417	0.1224	0.074
H(5)	4*a*	0.7409	0.5132	0.1321	0.097
H(6)	4*a*	0.6059	0.4012	0.2020	0.106
H(7)	4*a*	0.3033	0.4123	0.2603	0.092
H(9)	4*a*	0.1171	0.7967	0.1725	0.067
H(13A)	4*a*	0.7498	0.8401	–0.0162	0.099
H(13B)	4*a*	0.7218	0.7862	0.0628	0.099
H(13C)	4*a*	0.8495	0.8686	0.0648	0.099
H(14A)	4*a*	0.7770	0.9984	–0.0251	0.125
H(14B)	4*a*	0.6507	1.0797	–0.0103	0.125
H(14C)	4*a*	0.5755	1.0085	–0.0687	0.125
H(16)	4*a*	0.0773	1.0964	0.0164	0.091
H(17)	4*a*	0.0075	1.2375	0.0436	0.106
H(18)	4*a*	0.2147	1.3163	0.1223	0.103
H(19)	4*a*	0.4895	1.2552	0.1745	0.107
H(20)	4*a*	0.5587	1.1144	0.1492	0.089
H(1A)	4*a*	0.0060	0.5268	0.2836	0.093

* Correspondence author (e-mail: wjl425666@126.com)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*a*	0.0498(3)	0.6412(1)	0.2363(1)	0.076(2)	0.063(1)	0.080(2)	–0.012(1)	0.019(1)	0.001(1)
C(2)	4*a*	0.2031(3)	0.6735(1)	0.1932(1)	0.062(1)	0.051(1)	0.055(1)	–0.012(1)	0.006(1)	–0.0018(9)
C(3)	4*a*	0.3421(3)	0.6050(1)	0.1857(1)	0.068(1)	0.045(1)	0.047(1)	–0.014(1)	–0.0042(9)	–0.0022(8)
C(4)	4*a*	0.5240(3)	0.5965(1)	0.1500(1)	0.065(1)	0.053(1)	0.068(1)	–0.007(1)	–0.002(1)	0.001(1)
C(5)	4*a*	0.6194(4)	0.5198(1)	0.1562(2)	0.077(2)	0.065(1)	0.100(2)	0.003(1)	–0.008(1)	–0.007(1)
C(6)	4*a*	0.5372(5)	0.4518(2)	0.1980(2)	0.109(2)	0.054(1)	0.102(2)	0.004(2)	–0.031(2)	0.000(1)
C(7)	4*a*	0.3584(4)	0.4580(1)	0.2331(1)	0.114(2)	0.050(1)	0.066(1)	–0.019(1)	–0.023(2)	0.008(1)
C(8)	4*a*	0.2608(4)	0.5351(1)	0.2266(1)	0.085(2)	0.053(1)	0.051(1)	–0.020(1)	–0.010(1)	0.0009(9)
C(9)	4*a*	0.2193(3)	0.7590(1)	0.1644(1)	0.062(1)	0.048(1)	0.058(1)	–0.0057(9)	0.009(1)	–0.0051(9)
C(10)	4*a*	0.3931(3)	0.8667(1)	0.0990(1)	0.056(1)	0.0369(9)	0.050(1)	–0.0057(8)	0.0054(9)	–0.0071(8)
C(11)	4*a*	0.2648(3)	0.9380(1)	0.1049(1)	0.058(1)	0.042(1)	0.059(1)	–0.0058(9)	0.006(1)	–0.0123(9)
C(12)	4*a*	0.5607(3)	0.8936(1)	0.0627(1)	0.057(1)	0.044(1)	0.050(1)	–0.0064(9)	0.0043(9)	–0.0051(8)
C(13)	4*a*	0.7360(3)	0.8426(1)	0.0417(1)	0.062(1)	0.059(1)	0.076(1)	0.002(1)	0.016(1)	–0.001(1)
C(14)	4*a*	0.6467(4)	1.0197(2)	–0.0198(1)	0.092(2)	0.072(1)	0.086(2)	–0.008(1)	0.027(1)	0.019(1)
C(15)	4*a*	0.3239(3)	1.0918(1)	0.0811(1)	0.077(1)	0.039(1)	0.052(1)	0.000(1)	–0.003(1)	0.0017(9)
C(16)	4*a*	0.1605(3)	1.1282(1)	0.0489(1)	0.080(2)	0.062(1)	0.085(2)	0.006(1)	–0.017(1)	–0.011(1)
C(17)	4*a*	0.1193(4)	1.2128(1)	0.0649(2)	0.110(2)	0.064(1)	0.091(2)	0.026(1)	–0.017(2)	0.002(1)
C(18)	4*a*	0.2422(4)	1.2596(1)	0.1117(1)	0.142(2)	0.042(1)	0.072(1)	0.012(1)	–0.007(2)	0.003(1)
C(19)	4*a*	0.4052(4)	1.2231(1)	0.1428(1)	0.139(2)	0.046(1)	0.084(2)	–0.003(1)	–0.034(2)	–0.005(1)
C(20)	4*a*	0.4470(4)	1.1389(1)	0.1277(1)	0.105(2)	0.047(1)	0.069(1)	0.002(1)	–0.027(1)	0.000(1)
N(1)	4*a*	0.0837(3)	0.5587(1)	0.2565(1)	0.092(2)	0.070(1)	0.073(1)	–0.031(1)	0.012(1)	0.012(1)
N(2)	4*a*	0.3728(2)	0.78398(9)	0.12751(9)	0.0604(9)	0.0393(8)	0.0541(8)	–0.0073(7)	0.0100(8)	–0.0052(7)
N(3)	4*a*	0.3615(2)	1.00410(9)	0.0664(1)	0.064(1)	0.0397(8)	0.067(1)	–0.0011(7)	0.0086(9)	–0.0031(7)
N(4)	4*a*	0.5513(2)	0.97813(9)	0.04775(9)	0.064(1)	0.0437(9)	0.061(1)	–0.0064(8)	0.0136(8)	0.0015(7)
O(1)	4*a*	0.1022(2)	0.94616(8)	0.13736(9)	0.0619(9)	0.0525(8)	0.097(1)	–0.0012(7)	0.0218(8)	–0.0170(7)

Acknowledgments. The authors thank Henan University of Science and Technology for supporting this work.

References

1. Cheng, Q. F; Xu, X.Y; Wang, Q. F.: Synthesis and antibacterial activities of novel parazole Schiff bases and metal complexes.Chin. J. Org. Chem. **29** (2009) 1387-1391.
2. Zhong, X.; Yi, J.; Sun, J.; Wei, H. L.; Liu, W. S.; Yu, K. B.: Synthesis and crystal structure of some transition metal com plexes with a novel bis-Schiffbase ligand and their antitumor ac tivities. Eur. J. Med. Chem. **41** (2006) 1090-1092.
3. Deepa, S; Anjani, K..T.; Sweta, S.; Gauri, S.; Pushpa, M.; Harish, C. Anil, K. M.: Synthesis, characterization and biological activity of Schiff base analogues of indole-3-carboxaldehyde. Eur. J. Med. Chem. **43** (2008) 160-165.
4. Zhang, X. L.: 4-Chloro-2-[(*E*)-(2-chlorophenyl)iminomethyl]phenol. Acta Crystallogr. **E65** (2009) o512.
5. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.