Crystal structure of *trans*-N_3-(2-chlorobenzylidene)-5-bromopyridine-3,4-diamine, $C_{12}H_9BrClN_3$

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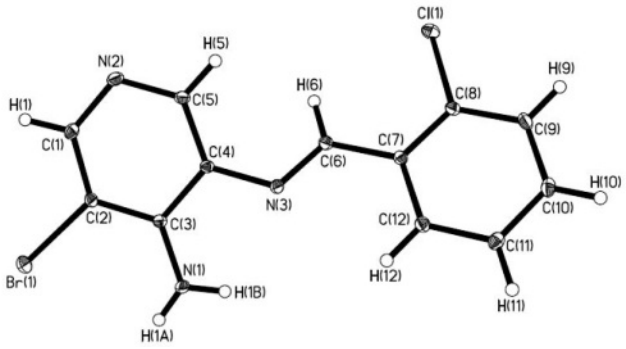

Abstract

$C_{12}H_9BrClN_3$, monoclinic, $P2_1/n$ (no. 14), a = 3.8743(5) Å, b = 22.229(2) Å, c = 13.631(1) Å, β = 98.103(6)°, V = 1162.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.0287, $wR_{ref}(F^2)$ = 0.0831, T = 113 K.

Table 1. Data collection and handling.

Crystal:	yellowish blocks, size 0.20×0.20×0.22 mm
Wavelength:	Mo K_α radiation (0.71075 Å)
μ:	37.45 cm^{-1}
Diffractometer, scan mode:	Rigaku Saturn724 CCD, ω
$2\theta_{max}$:	67.46°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15867, 4620
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3491
$N(param)_{refined}$:	161
Programs:	CRYSTAL CLEAR [2], SHELX [3]

Source of material

The solution of 5-bromopyridine-3,4-diamine and 2-chlorobenzaldehyde in methanol was refluxed for 6 h, and then the crude product was isolated by filtration and recrystallized from methanol to yield the yellowish title compound. Finally, the title compound was dissolved in a small amount of acetone and the solution was kept for 3 days at ambient temperature to give rise to yellowish block-like crystals on slowl evaporation of the solvent.

Experimental details

The imino H atom was located in a difference fourier map and refined with N–H = 0.86 Å.The remaining H atoms were positioned geometrically (C–H = 0.93-0.98 Å) and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq} (methyl groups).

Discussion

Schiff bases have played an important role in the development of coordination chemistry as they readily form stable complexes with most of the transition metals. They show interesting properties, e.g., their ability to reversibly bind oxygen, catalytic activity in hydrogenation of olefins, transfer of an amino group, photochromic properties and complexing ability towards toxic metals [1]. In this paper, the title new Schiff base compound derived from the condensation of 2-chlorobenzaldehyde with 5-bromopyridine-3,4-diamine is reported. The molecule of the title compound, Fig. 1, possesses an *E* configuration with respect to the C6=N3 bond. The bond lengths have normal values. The dihedral angle between the C7–C12 benzene and N2,C1–C5 pyridine planes is 28.2(1)°. The crystal packing is stabilized by N–H···N hydrogen bonds. An intramolecular N–H···N hydrogen bond may influence the molecular conformation. In the crystal, the molecules are linked by N–H···N hydrogen bonds into chains.

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

Atom	Site	x	y	z	U_{iso}
H(1A)	4*e*	0.490(6)	0.2827(9)	0.557(2)	0.027
H(1B)	4*e*	0.604(7)	0.3334(9)	0.509(2)	0.027
H(1)	4*e*	0.0060	0.1708	0.2815	0.017
H(5)	4*e*	0.2781	0.3267	0.1815	0.017
H(6)	4*e*	0.7058	0.3870	0.2276	0.015
H(9)	4*e*	1.0738	0.5859	0.1858	0.021
H(10)	4*e*	1.0104	0.6340	0.3342	0.020
H(11)	4*e*	0.7996	0.5807	0.4625	0.019
H(12)	4*e*	0.6419	0.4800	0.4390	0.017

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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}
Br(1)	4*e*	0.17191(5)	0.170047(7)	0.49150(1)	0.0262(1)	0.01687(9)	0.01907(9)	–0.00412(6)	0.00330(7)	0.00447(6)
Cl(1)	4*e*	0.9679(1)	0.46929(2)	0.11479(3)	0.0334(3)	0.0263(2)	0.0131(2)	–0.0073(2)	0.0094(2)	–0.0009(1)
N(1)	4*e*	0.4964(5)	0.30129(6)	0.5058(1)	0.0292(8)	0.0126(6)	0.0101(6)	–0.0051(5)	–0.0016(5)	0.0008(4)
N(2)	4*e*	0.1216(4)	0.24569(6)	0.2153(1)	0.0209(7)	0.0144(6)	0.0116(6)	–0.0001(5)	–0.0002(5)	–0.0026(5)
N(3)	4*e*	0.5194(4)	0.37940(6)	0.3513(1)	0.0148(6)	0.0104(5)	0.0118(5)	–0.0001(4)	0.0005(5)	0.0005(4)
C(1)	4*e*	0.1044(4)	0.20970(7)	0.2934(1)	0.0161(8)	0.0109(6)	0.0164(7)	0.0002(5)	0.0008(6)	–0.0024(5)
C(2)	4*e*	0.2216(4)	0.22595(6)	0.3902(1)	0.0136(7)	0.0104(6)	0.0131(6)	0.0002(5)	0.0022(5)	0.0016(5)
C(3)	4*e*	0.3741(4)	0.28247(6)	0.4131(1)	0.0130(7)	0.0105(6)	0.0106(6)	0.0015(5)	0.0013(5)	0.0002(5)
C(4)	4*e*	0.3942(4)	0.32052(6)	0.3304(1)	0.0133(7)	0.0104(6)	0.0098(6)	0.0013(5)	0.0010(5)	0.0005(4)
C(5)	4*e*	0.2645(5)	0.30018(7)	0.2356(1)	0.0182(8)	0.0140(6)	0.0100(6)	0.0003(5)	0.0020(5)	–0.0003(5)
C(6)	4*e*	0.6647(4)	0.40752(6)	0.2860(1)	0.0152(8)	0.0117(6)	0.0119(6)	0.0014(5)	0.0024(5)	0.0000(5)
C(7)	4*e*	0.7693(4)	0.47098(6)	0.2996(1)	0.0126(7)	0.0118(6)	0.0117(6)	0.0007(5)	0.0012(5)	0.0015(5)
C(8)	4*e*	0.9020(4)	0.50365(7)	0.2254(1)	0.0163(8)	0.0157(6)	0.0112(6)	–0.0008(5)	0.0010(5)	0.0019(5)
C(9)	4*e*	0.9888(5)	0.56431(7)	0.2376(1)	0.0180(8)	0.0166(7)	0.0171(7)	–0.0046(6)	–0.0004(6)	0.0063(6)
C(10)	4*e*	0.9502(4)	0.59275(7)	0.3255(1)	0.0163(8)	0.0115(6)	0.0217(8)	–0.0008(5)	–0.0029(6)	0.0018(5)
C(11)	4*e*	0.8231(4)	0.56120(7)	0.4018(1)	0.0158(8)	0.0129(6)	0.0177(7)	0.0022(5)	–0.0001(6)	–0.0012(5)
C(12)	4*e*	0.7320(4)	0.50119(7)	0.3877(1)	0.0151(7)	0.0130(6)	0.0139(6)	0.0010(5)	0.0034(5)	0.0009(5)

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References

1. Kocyigit, O.; Kursunlu, A. N.; Guler, E.: J. Hazard. Mater. **183** (2010) 334-340.
2. Rigaku: Crystal Structure Analysis Package. Rigaku Corporation, Tokyo, Japan 2009.
3. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.