Crystal structure of *E*-5-bromo-N^3-(4-methoxybenzylidene)-pyridine-3,4-diamine, $C_{13}H_{12}BrN_3O$

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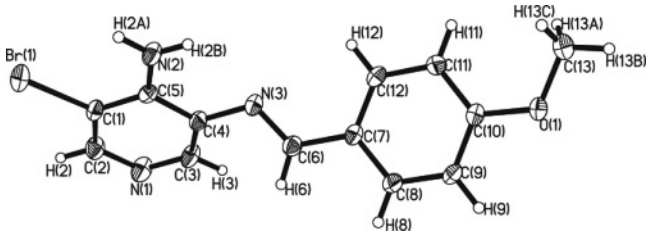

Abstract

$C_{13}H_{12}BrN_3O$, monoclinic, $P2_1/c$ (no. 14), a = 5.520(3) Å, b = 13.400(7) Å, c = 17.204(9) Å, β = 94.916(9)°, V = 1267.9 Å^3, Z = 4, $R_{gt}(F)$ = 0.0335, $wR_{ref}(F^2)$ = 0.0957, T = 296 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.17×0.21×0.26 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	32.33 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$2\theta_{max}$:	49.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6327, 2234
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1805
$N(param)_{refined}$:	164
Programs:	CrystalClear [2], SHELX [3]

Source of material

The solution of 5-bromopyridine-3,4-diamine and 4-methoxybenzaldehyde in methanol was refluxed for 3 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 7 days at ambient temperature to give yellowish block-like crystals on slowly evaporation.

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. 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 4-methoxybenzaldehyde with 5-bromopyridine-3,4-diamine is reported. The molecule of the title compound (cf. Figure), possesses an *E* configuration about the C6=N3 bond. The bond lengths have normal values. The dihedral angle between the C7-C12 benzene and N1,C1-C5 pyridine planes is 54.81(1)°. In the crystal, molecules are linked by N–H···N and N–H···O hydrogen bond into a network.

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	1.4256	1.1717	0.1993	0.051
H(3)	4*e*	0.9157	1.1361	0.3380	0.051
H(6)	4*e*	0.8883	0.9891	0.4146	0.042
H(8)	4*e*	0.7402	0.9147	0.5317	0.049
H(9)	4*e*	0.4757	0.8149	0.5921	0.046
H(11)	4*e*	0.1287	0.7472	0.3835	0.041
H(12)	4*e*	0.4019	0.8453	0.3236	0.039
H(13A)	4*e*	–0.1634	0.6924	0.4611	0.076
H(13B)	4*e*	–0.1321	0.6129	0.5280	0.076
H(13C)	4*e*	0.0368	0.6096	0.4593	0.076
H(2A)	4*e*	1.1615	0.8516	0.1619	0.046
H(2B)	4*e*	0.9683	0.8390	0.2165	0.046

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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*	1.47417(6)	0.99107(3)	0.11399(2)	0.0471(2)	0.0621(3)	0.0498(2)	0.0034(2)	0.0217(2)	–0.0003(2)
C(1)	4*e*	1.2772(5)	1.0361(2)	0.1917(2)	0.035(2)	0.038(2)	0.028(1)	0.003(1)	0.009(1)	0.004(1)
C(2)	4*e*	1.3065(6)	1.1323(2)	0.2190(2)	0.050(2)	0.035(2)	0.044(2)	–0.008(1)	0.015(2)	0.005(1)
C(3)	4*e*	1.0109(6)	1.1104(2)	0.3007(2)	0.061(2)	0.032(2)	0.038(2)	–0.000(1)	0.021(2)	–0.002(1)
C(4)	4*e*	0.9733(5)	1.0122(2)	0.2793(2)	0.037(2)	0.031(2)	0.027(1)	–0.001(1)	0.006(1)	0.003(1)
C(5)	4*e*	1.1077(5)	0.9711(2)	0.2205(2)	0.033(2)	0.027(1)	0.024(1)	0.005(1)	0.002(1)	0.003(1)
C(6)	4*e*	0.7814(5)	0.9494(2)	0.3833(2)	0.041(2)	0.033(2)	0.033(2)	–0.002(1)	0.006(1)	–0.000(1)
C(7)	4*e*	0.6018(5)	0.8902(2)	0.4206(2)	0.038(2)	0.030(1)	0.029(1)	0.002(1)	0.009(1)	0.000(1)
C(8)	4*e*	0.6188(6)	0.8805(2)	0.5018(2)	0.048(2)	0.046(2)	0.029(2)	–0.012(1)	0.006(1)	–0.006(1)
C(9)	4*e*	0.4597(5)	0.8216(2)	0.5381(2)	0.047(2)	0.046(2)	0.023(1)	–0.006(1)	0.007(1)	–0.001(1)
C(10)	4*e*	0.2753(5)	0.7721(2)	0.4942(2)	0.036(2)	0.031(1)	0.031(1)	0.002(1)	0.009(1)	0.003(1)
C(11)	4*e*	0.2521(5)	0.7806(2)	0.4132(2)	0.035(2)	0.036(2)	0.033(2)	–0.002(1)	0.002(1)	0.001(1)
C(12)	4*e*	0.4163(5)	0.8395(2)	0.3777(2)	0.040(2)	0.033(1)	0.024(1)	0.007(1)	0.005(1)	0.003(1)
C(13)	4*e*	–0.0481(6)	0.6526(3)	0.4924(2)	0.052(2)	0.051(2)	0.050(2)	–0.015(2)	0.010(2)	0.005(2)
N(1)	4*e*	1.1730(5)	1.1719(2)	0.2724(2)	0.073(2)	0.029(1)	0.050(2)	–0.007(1)	0.023(1)	–0.002(1)
N(2)	4*e*	1.0752(4)	0.8757(2)	0.1968(1)	0.049(2)	0.032(1)	0.037(1)	–0.002(1)	0.017(1)	–0.004(1)
N(3)	4*e*	0.7982(4)	0.9491(2)	0.3097(1)	0.040(1)	0.031(1)	0.031(1)	0.001(1)	0.011(1)	0.001(1)
O(1)	4*e*	0.1222(4)	0.7164(2)	0.5355(1)	0.046(1)	0.048(1)	0.035(1)	–0.014(1)	0.0090(9)	0.0062(9)

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