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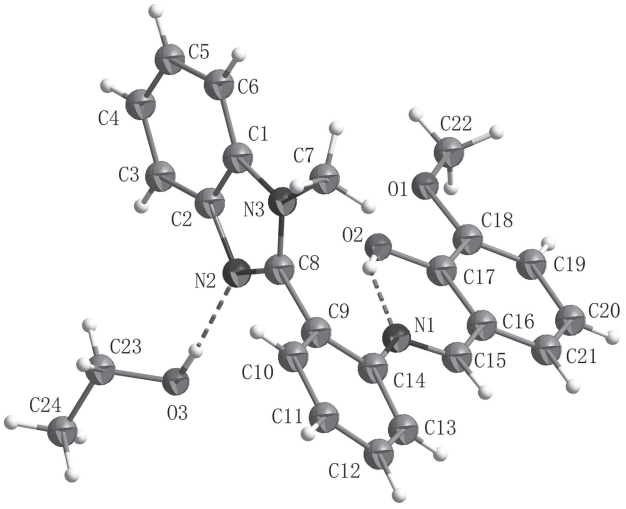
Crystal structure of 2-methoxy-6-(((2-(1-methyl-1*H*-benzo[*d*] imidazol-2-yl)phenyl)imino) methyl)phenol — ethanol (1/1), $C_{24}H_{25}N_3O_3$



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Abstract

$C_{24}H_{25}N_3O_3$, monoclinic, (no. 14), $a = 10.2246(7)$ Å, $b = 21.2529(7)$ Å, $c = 10.6924(6)$ Å, $\beta = 116.990(8)°$, $V = 2070.4(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0499$, $wR_{ref}(F^2) = 0.1155$, $T = 102$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was synthesized by the condensation of 2-(1-methyl-1*H*-benzo[*d*]imidazol-2-yl)aniline and 2-hydroxy-3-methoxybenzaldehyde (*o*-vanillin) (molar ratio 1:1)

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Table 1: Data collection and handling.

Crystal:	Orange, rhombic, size 0.08×0.20×0.25 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	0.87 cm^{-1}
Diffractometer, scan mode:	Xcalibur, Eos, Gemini, ω scans
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9252, 4065
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2925
$N(param)_{refined}$:	276
Programs:	CrysAlis [7], SHELX [8], Diamond [9]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2)	4*e*	0.5236	0.1438	0.9786	0.040
H(3A)	4*e*	0.3106	0.0951	1.1493	0.047
H(15)	4*e*	0.6834	0.0129	0.9464	0.025
H(21)	4*e*	0.6266	0.0236	0.6984	0.030
H(13)	4*e*	0.7302	−0.0474	1.1125	0.028
H(19)	4*e*	0.4159	0.1757	0.4777	0.032
H(12)	4*e*	0.8383	−0.0960	1.3285	0.030
H(10)	4*e*	0.7772	0.0592	1.5078	0.029
H(11)	4*e*	0.8636	−0.0426	1.5269	0.032
H(4)	4*e*	0.3058	0.3347	1.2393	0.033
H(7A)	4*e*	0.9133	0.2258	1.4142	0.038
H(7B)	4*e*	0.9193	0.1545	1.3793	0.038
H(7C)	4*e*	0.9140	0.1738	1.5185	0.038
H(3)	4*e*	0.2676	0.2305	1.1689	0.030
H(20)	4*e*	0.5250	0.0785	0.4901	0.033
H(5)	4*e*	0.5406	0.3747	1.3604	0.032
H(6)	4*e*	0.7451	0.3119	1.4164	0.031
H(23A)	4*e*	0.1234	0.1402	1.1672	0.042
H(23B)	4*e*	0.2163	0.0945	1.2917	0.042
H(22A)	4*e*	0.2420	0.2463	0.4801	0.067
H(22B)	4*e*	0.3949	0.2787	0.5266	0.067
H(22C)	4*e*	0.2805	0.3082	0.5695	0.067
H(24A)	4*e*	0.0520	0.0138	1.1805	0.077
H(24B)	4*e*	−0.0395	0.0583	1.0537	0.077
H(24C)	4*e*	−0.0323	0.0713	1.2010	0.077

Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(2)	4*e*	0.4833(2)	0.16694(6)	0.9102(1)	0.0348(8)	0.0275(8)	0.0220(7)	0.0088(6)	0.0160(7)	0.0043(6)
N(1)	4*e*	0.6221(2)	0.06954(7)	1.0500(2)	0.0216(9)	0.0199(8)	0.0209(8)	0.0000(7)	0.0102(7)	0.0005(7)
O(3)	4*e*	0.2320(2)	0.07645(6)	1.1189(2)	0.0257(8)	0.0304(8)	0.0417(9)	−0.0036(7)	0.0179(7)	−0.0085(7)
N(3)	4*e*	0.7204(2)	0.18225(7)	1.3486(2)	0.0189(8)	0.0211(8)	0.0210(8)	−0.0005(7)	0.0094(7)	0.0003(7)
O(1)	4*e*	0.3790(2)	0.23309(6)	0.6791(1)	0.049(1)	0.0325(8)	0.0289(8)	0.0171(7)	0.0194(7)	0.0120(7)
C(15)	4*e*	0.6339(2)	0.05023(9)	0.9413(2)	0.019(1)	0.018(1)	0.027(1)	−0.0021(8)	0.0106(8)	−0.0011(8)
C(17)	4*e*	0.5002(2)	0.14322(9)	0.8020(2)	0.022(1)	0.025(1)	0.021(1)	−0.0016(9)	0.0106(9)	−0.0016(8)
C(18)	4*e*	0.4434(2)	0.17714(9)	0.6758(2)	0.023(1)	0.023(1)	0.028(1)	0.0046(9)	0.0115(9)	0.0040(9)
N(2)	4*e*	0.4925(2)	0.14420(7)	1.2281(2)	0.0213(9)	0.0213(8)	0.0232(8)	0.0009(7)	0.0094(7)	0.0000(7)
C(14)	4*e*	0.6856(2)	0.03560(9)	1.1779(2)	0.0161(9)	0.022(1)	0.023(1)	−0.0015(8)	0.0095(8)	0.0015(8)
C(21)	4*e*	0.5803(2)	0.06192(9)	0.6929(2)	0.024(1)	0.025(1)	0.030(1)	0.0006(9)	0.0141(9)	−0.0023(9)
C(13)	4*e*	0.7388(2)	−0.02584(9)	1.1915(2)	0.021(1)	0.022(1)	0.026(1)	−0.0014(9)	0.0105(9)	−0.0012(8)
C(9)	4*e*	0.6966(2)	0.06705(8)	1.2974(2)	0.017(1)	0.022(1)	0.024(1)	−0.0016(8)	0.0106(8)	0.0008(8)
C(19)	4*e*	0.4539(2)	0.15303(9)	0.5611(2)	0.024(1)	0.034(1)	0.022(1)	−0.0006(9)	0.0109(9)	0.0040(9)
C(16)	4*e*	0.5715(2)	0.08560(8)	0.8113(2)	0.018(1)	0.022(1)	0.021(1)	−0.0031(8)	0.0086(8)	−0.0005(8)
C(1)	4*e*	0.6259(2)	0.23255(9)	1.3277(2)	0.025(1)	0.022(1)	0.020(1)	0.0031(9)	0.0129(8)	0.0026(8)
C(8)	4*e*	0.6346(2)	0.13108(8)	1.2877(2)	0.023(1)	0.022(1)	0.0179(9)	−0.0018(9)	0.0111(8)	0.0010(8)
C(12)	4*e*	0.8041(2)	−0.05490(9)	1.3209(2)	0.023(1)	0.020(1)	0.033(1)	0.0016(8)	0.0136(9)	0.0048(9)
C(10)	4*e*	0.7660(2)	0.03772(9)	1.4278(2)	0.024(1)	0.028(1)	0.022(1)	0.0006(9)	0.0116(9)	0.0007(8)
C(11)	4*e*	0.8186(2)	−0.02311(9)	1.4396(2)	0.025(1)	0.027(1)	0.027(1)	0.0033(9)	0.0112(9)	0.0091(9)
C(4)	4*e*	0.3858(2)	0.30843(9)	1.2603(2)	0.033(1)	0.028(1)	0.029(1)	0.009(1)	0.019(1)	0.0055(9)
C(7)	4*e*	0.8804(2)	0.18426(9)	1.4214(2)	0.023(1)	0.025(1)	0.029(1)	0.0000(9)	0.0118(9)	0.0009(9)
C(3)	4*e*	0.3622(2)	0.24634(9)	1.2184(2)	0.024(1)	0.027(1)	0.024(1)	0.0006(9)	0.0091(9)	0.0026(9)
C(20)	4*e*	0.5208(2)	0.09489(9)	0.5688(2)	0.027(1)	0.036(1)	0.021(1)	−0.003(1)	0.0134(9)	−0.0021(9)
C(2)	4*e*	0.4844(2)	0.20810(9)	1.2526(2)	0.023(1)	0.021(1)	0.020(1)	−0.0003(8)	0.0109(8)	0.0015(8)
C(5)	4*e*	0.5283(2)	0.33261(9)	1.3340(2)	0.034(1)	0.022(1)	0.027(1)	0.0018(9)	0.0156(9)	0.0012(9)
C(6)	4*e*	0.6507(2)	0.29568(9)	1.3684(2)	0.029(1)	0.024(1)	0.027(1)	−0.0023(9)	0.0151(9)	0.0002(9)
C(23)	4*e*	0.1535(2)	0.0967(1)	1.1913(2)	0.032(1)	0.040(1)	0.038(1)	−0.002(1)	0.020(1)	−0.005(1)
C(22)	4*e*	0.3192(3)	0.2696(1)	0.5536(2)	0.057(2)	0.041(1)	0.035(1)	0.019(1)	0.019(1)	0.016(1)
C(24)	4*e*	0.0218(3)	0.0565(1)	1.1532(3)	0.038(1)	0.072(2)	0.050(2)	−0.019(1)	0.026(1)	−0.018(1)

in ethanol. A yellow powder was obtained by evaporation of solution in a yield of 95%. The orange single-crystal was prepared by recrystallization from *n*-hexane and dichloromethane. IR (KBr, cm^{-1}): 3433 s, 3003w, 2970w, 1614vs, 1569*m*, 1527*m*, 1461vs, 1385*m*, 1329*m*, 1257vs, 786*m*, 771*m*, 747*m*, 739*m*. ^{1}HNMR (600 MHz, DMSO, p.p.m.): 12.24 (s, 1H), 9.02 (s, 1H), 7.71–7.57 (m, 5H), 7.51–7.46 (m, 1H), 7.31–7.21 (m, 2H), 7.18 (m, 1H), 7.04 (m, 1H), 6.85 (m, 1H), 3.68 (s, 3H), 3.56 (s, 3H). Elemental analysis: found – C, 71.65%; H, 6.57%; N, 10.23% (VarioEL), calcd. for $C_{24}H_{25}N_3O_3$ (403.47 g/mol) – C, 71.44%; H, 6.25%; N, 10.41%.

Experimental details

All the H atoms of carbon were included using a riding-model, with C—H = 0.93–0.97 Å, and their $U_{iso} = 1.2U_{eq}$ or $1.5U_{eq}$. The H atoms of oxygen were also included using a riding-mod, with O—H = 0.82 Å and their $U_{iso} = 1.5U_{eq}$.

Discussion

Bidentate, tridentate or polydentate Schiff-base ligands with steric hindrance effect can be used to protect the catalytic activity of the metal center in the early transition metal olefin polymerization catalysts [1, 2]. The Schiff-base compounds with benzimidazole moiety [3, 4] and their metal complexes [5, 6] also have been reported on their broad biological activities. The title compound is a polydentate Schiff-base with benzimidazole and steric hindrance synthesized in a convenient approach. It was expected to be used as ligand to prepare metal complexes, which has the potential application in olefin polymerization catalyst.

The bond length of C15—N1 in the title molecule is 1.288(2) Å, showing a characteristic double C=N bond. The molecule consists of three planar moieties. In the benzimidazole ring, the bond distances of N2—C2, N2—C8, N3—C1 and N3—C8 are in the range of 1.325(2) to 1.393(2) Å. The benzimidazole ring (*Cg*(1)) and six-membered C9—C10—C11—C12—C13—C14 ring (*Cg*(2)) nearly perpendicular to each other with a dihedral angle of 78.7(1)°. The two six-membered *Cg*(2) and C16—C17—C18—C19—C20—C21 ring (*Cg*(3)) are almost parallel with a dihedral angle of 11.5(1)°. There is an intramolecular hydrogen bond between the hydroxyl O2 and imino N1. The title compound contains a solvent ethanol molecule,

which is linked by the O—H···N hydrogen bond between the imidazolyl N2 and hydroxyl O3 of the ethanol molecule. In the crystal packing structure, there are some intermolecular forces including C—H···O hydrogen bonds and π-π stacking forces. The distance between the six-membered ring centroids of *Cg*(3) and *Cg*(2) (1−*x*, −*y*, 2−*z*) is 3.8075(12) Å.

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