Crystal structure of (5-bromo-2,3-diaminopyridine-*N*,*N′*-bis(3-methoxy-salicylideneiminato)-*O*,*O′*,*N*,*N′*)nickel(II) — methanol (1:1), $Ni(C_{21}H_{16}BrN_3O_4) \cdot CH_3OH$

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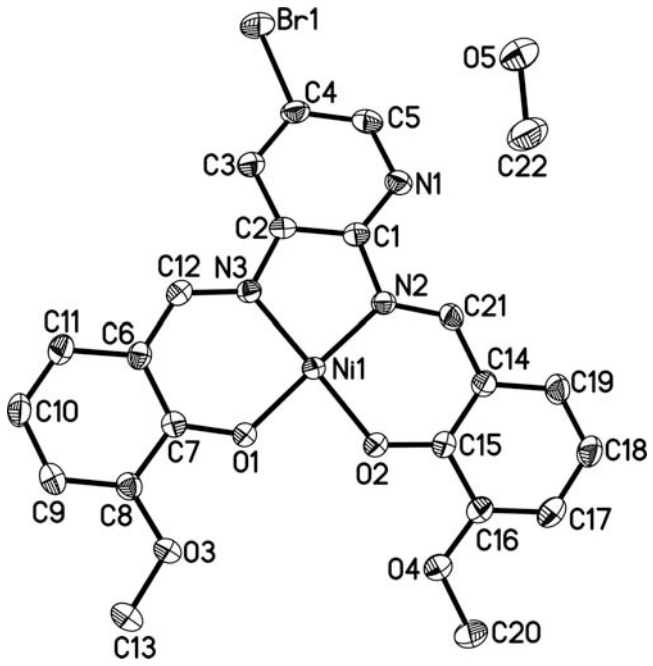

Abstract

$C_{22}H_{20}BrN_3NiO_5$, triclinic, $P\bar{1}$ (no. 2), a = 7.5761(8) Å, b = 12.030(1) Å, c = 13.255(1) Å, α = 105.873(2)°, β = 97.984(2)°, γ = 95.002(2)°, V = 1140.8 Å^3, Z = 2, $R_{gt}(F)$ = 0.040, $wR_{ref}(F^2)$ = 0.108, T = 293 K.

Source of material

The Schiff base ligand was synthesized by condensation 5-bromo-2,3-diaminopyridine and 2-hydroxy-3-methoxybenzaldehyde with the ratio 1 : 2 in ethanol. The synthesis of the title complex was carried out by reacting $Ni(ClO_4)_2 \cdot 6H_2O$ and the schiff base ligand (1:1, molar ratio) in methanol. After stirring, the process was continued for about 30 min at room temperature. The mixture was filtered and the filtrate was allowed to partially evaporate in air for sevral days to produce red-brown crystals suitable for X-ray diffraction with a yield about 60 %.

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, in which some could exhibit interesting properties [1-4].

The coordination sphere for the Ni(II) ion in the title crystal structure is a slightly distorted square planar, in which the four positions are occupied by two N atoms and two O atoms of the Schiff-base ligand. The mean deviation from the plane formed by the two N atoms, two O atoms and the Ni ion is only 0.035 Å, indicative of that these five atoms can form a perfect plane. The bond lengths of Ni—N are 1.874(3) Å and 1.929(3) Å, and Ni—O 1.861(2) Å and 1.905(2) Å, respectively, are consistant with the corresponding distances in 6,6′-dimethoxy-2,2′-(ethane-1,2-diyl-bis(nitrilomethylidyne)diphenolato)nickel(II) [5].

Table 1. Data collection and handling.

Crystal:	red-brown block, size 0.19 × 0.21 × 0.23 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	26.40 cm^{-1}
Diffractometer, scan mode:	Bruker APEX II CCD, φ/ω
$2\theta_{max}$:	50.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5614, 3964
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3017
$N(param)_{refined}$:	293
Programs:	SHELXS-97 [6], SHELXL-97 [7], XP [8]

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

Atom	Site	x	y	z	U_{iso}
H(5)	2*i*	0.3038	0.8057	0.2390	0.106
H(3)	2*i*	0.3084	1.2634	0.7394	0.059
H(5A)	2*i*	0.1176	1.5086	0.6106	0.062
H(9)	2*i*	0.4536	0.6113	0.5403	0.063
H(10)	2*i*	0.5600	0.7455	0.7080	0.067
H(11)	2*i*	0.5148	0.9380	0.7396	0.061
H(12)	2*i*	0.3823	1.0900	0.6809	0.049
H(13A)	2*i*	0.4226	0.5243	0.3467	0.095
H(13B)	2*i*	0.2396	0.4897	0.2673	0.095
H(13C)	2*i*	0.2443	0.4948	0.3871	0.095
H(17)	2*i*	–0.3004	0.8838	–0.0654	0.072
H(18)	2*i*	–0.3192	1.0831	–0.0358	0.073
H(19)	2*i*	–0.1992	1.2121	0.1278	0.066
H(20A)	2*i*	–0.3411	0.6953	–0.0436	0.145
H(20B)	2*i*	–0.1850	0.6200	–0.0318	0.145
H(20C)	2*i*	–0.1643	0.7183	–0.0878	0.145
H(21)	2*i*	–0.0372	1.2550	0.3111	0.052
H(22A)	2*i*	0.2666	0.7021	0.0428	0.137
H(22B)	2*i*	0.1772	0.6601	0.1281	0.137
H(22C)	2*i*	0.3726	0.6349	0.1122	0.137

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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}
Ni(1)	2*i*	0.13772(6)	1.01109(4)	0.39771(3)	0.0388(3)	0.0324(3)	0.0340(3)	0.0050(2)	0.0015(2)	0.0067(2)
Br(1)	2*i*	0.26707(8)	1.51355(4)	0.83239(4)	0.1241(5)	0.0520(3)	0.0559(3)	0.0190(3)	–0.0023(3)	–0.0111(2)
O(1)	2*i*	0.2100(3)	0.8679(2)	0.4005(2)	0.052(1)	0.037(1)	0.038(1)	0.008(1)	–0.002(1)	0.009(1)
O(2)	2*i*	0.0253(3)	0.9318(2)	0.2552(2)	0.056(2)	0.040(1)	0.039(1)	0.008(1)	–0.005(1)	0.008(1)
O(3)	2*i*	0.2686(4)	0.6499(2)	0.3634(2)	0.070(2)	0.036(1)	0.052(2)	0.015(1)	0.001(1)	0.008(1)
O(4)	2*i*	–0.1354(4)	0.7795(2)	0.0696(2)	0.097(2)	0.048(2)	0.051(2)	0.002(2)	–0.019(2)	0.007(1)
O(5)	2*i*	0.3587(4)	0.7965(3)	0.1888(2)	0.076(2)	0.072(2)	0.052(2)	0.007(2)	0.009(2)	0.000(2)
N(1)	2*i*	0.0892(4)	1.3546(3)	0.5005(2)	0.051(2)	0.038(2)	0.052(2)	0.010(1)	0.004(2)	0.009(2)
N(2)	2*i*	0.0713(4)	1.1554(2)	0.3901(2)	0.039(2)	0.035(2)	0.038(2)	0.005(1)	0.005(1)	0.008(1)
N(3)	2*i*	0.2417(3)	1.0874(2)	0.5445(2)	0.034(1)	0.035(2)	0.037(2)	0.004(1)	0.005(1)	0.007(1)
C(1)	2*i*	0.1240(4)	1.2447(3)	0.4909(3)	0.035(2)	0.039(2)	0.042(2)	0.003(2)	0.007(2)	0.007(2)
C(2)	2*i*	0.2117(4)	1.2071(3)	0.5772(3)	0.037(2)	0.033(2)	0.039(2)	0.000(2)	0.008(2)	0.004(2)
C(3)	2*i*	0.2556(5)	1.2858(3)	0.6817(3)	0.054(2)	0.043(2)	0.046(2)	0.008(2)	0.002(2)	0.006(2)
C(4)	2*i*	0.2144(5)	1.3986(3)	0.6919(3)	0.058(2)	0.040(2)	0.041(2)	0.006(2)	0.007(2)	–0.002(2)
C(5)	2*i*	0.1368(5)	1.4313(3)	0.6011(3)	0.055(2)	0.037(2)	0.060(3)	0.009(2)	0.012(2)	0.005(2)
C(6)	2*i*	0.3697(4)	0.9231(3)	0.5884(3)	0.034(2)	0.047(2)	0.040(2)	0.004(2)	0.004(2)	0.016(2)
C(7)	2*i*	0.3036(4)	0.8410(3)	0.4836(3)	0.034(2)	0.040(2)	0.041(2)	0.005(2)	0.007(2)	0.014(2)
C(8)	2*i*	0.3381(5)	0.7223(3)	0.4669(3)	0.041(2)	0.043(2)	0.048(2)	0.005(2)	0.005(2)	0.016(2)
C(9)	2*i*	0.4331(5)	0.6886(4)	0.5517(3)	0.052(2)	0.051(2)	0.062(3)	0.016(2)	0.009(2)	0.025(2)
C(10)	2*i*	0.4983(5)	0.7703(4)	0.6541(3)	0.056(2)	0.065(3)	0.051(2)	0.016(2)	–0.003(2)	0.026(2)
C(11)	2*i*	0.4705(5)	0.8849(4)	0.6734(3)	0.045(2)	0.060(3)	0.045(2)	0.009(2)	–0.001(2)	0.016(2)
C(12)	2*i*	0.3359(4)	1.0414(3)	0.6129(3)	0.039(2)	0.046(2)	0.035(2)	0.000(2)	0.004(2)	0.009(2)
C(13)	2*i*	0.2960(6)	0.5298(3)	0.3391(3)	0.078(3)	0.040(2)	0.072(3)	0.017(2)	0.010(2)	0.014(2)
C(14)	2*i*	–0.0862(5)	1.0952(3)	0.2005(3)	0.042(2)	0.047(2)	0.039(2)	0.013(2)	0.008(2)	0.011(2)
C(15)	2*i*	–0.0659(4)	0.9747(3)	0.1831(3)	0.040(2)	0.046(2)	0.038(2)	0.005(2)	0.005(2)	0.015(2)
C(16)	2*i*	–0.1526(5)	0.8952(3)	0.0803(3)	0.050(2)	0.049(2)	0.043(2)	0.002(2)	–0.000(2)	0.010(2)
C(17)	2*i*	–0.2472(5)	0.9364(4)	–0.0004(3)	0.056(2)	0.072(3)	0.043(2)	0.002(2)	–0.007(2)	0.011(2)
C(18)	2*i*	–0.2606(5)	1.0564(4)	0.0176(3)	0.065(3)	0.069(3)	0.048(2)	0.020(2)	–0.006(2)	0.020(2)
C(19)	2*i*	–0.1860(5)	1.1336(4)	0.1154(3)	0.060(2)	0.058(3)	0.051(2)	0.022(2)	0.005(2)	0.022(2)
C(20)	2*i*	–0.2130(8)	0.6963(4)	–0.0320(4)	0.144(5)	0.053(3)	0.063(3)	–0.014(3)	–0.034(3)	0.001(2)
C(21)	2*i*	–0.0178(5)	1.1781(3)	0.3035(3)	0.047(2)	0.041(2)	0.045(2)	0.017(2)	0.008(2)	0.014(2)
C(22)	2*i*	0.2882(8)	0.6899(4)	0.1118(4)	0.121(4)	0.072(3)	0.060(3)	0.021(3)	–0.013(3)	–0.004(3)

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