Crystal structure of *N*,*N′*-bis(2-hydroxy-4-methoxysalicylidene)-ethylenediaminatonickel(II), $Ni(C_{18}H_{18}N_2O_4)$

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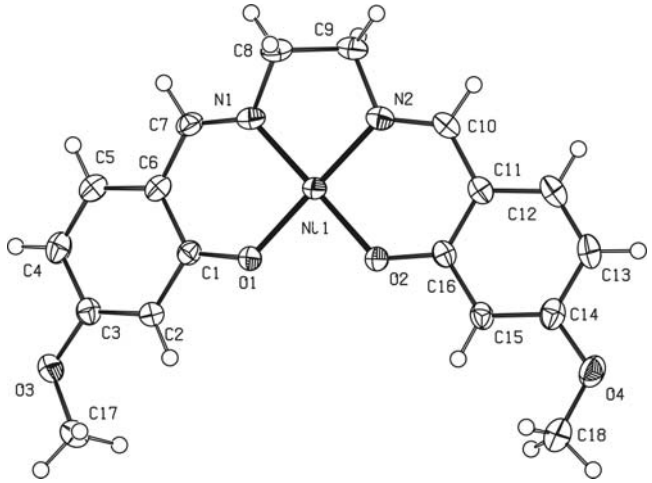

Abstract

$C_{18}H_{18}N_2NiO_4$, orthorhombic, $Pna2_1$ (no. 33), a = 25.884(1) Å, b = 6.3836(3) Å, c = 9.9193(5) Å, V = 1639.0 Å^3, Z = 4, $R_{gt}(F)$ = 0.041, $wR_{ref}(F^2)$ = 0.088, T = 173 K.

Source of material

All purchased chemicals were of reagent grade and used without further purification. *N*,*N′*-bis(2-hydroxyl-4-methoxysalicylidene)ethylenediamine (L) was prepared from the reaction of ethylenediamine and 2-hydroxy-4-methoxybezaldehyde in ethanol. A mixture of ligand L (0.328 g, 1 mmol) and $Ni(NO_3)_2 \cdot 6H_2O$ (0.29 g, 1 mmol) in ethanol (15 ml) was refluxed for four hours under stirring. The progress of the reaction was monitored by TLC using hexane/ethylacetat (1/2, *v*/*v*) as eluent. The resultant mixture was filtered and the filtrate was kept at 4 °C to evaporate slowly. The orange block-shaped single crystals suitable for X-ray crystal structure determination were obtained.

Experimental details

The hydrogen atoms were included in the refinement at geometrically idealized positions with d(C—H) = 0.95, 0.99 and 0.98 Å for aryl, methyl and methylene, respectively, and U_{iso}(H) = 1.2 U_{eq}(C). An absolute structure could not be established as the crystal was twinned and a TWIN command in conjunction with BASF (0.43) command was used to refine the structure.

Discussion

During the last few decades, there has been considerable interest in the chemistry of Schiff base compounds. Schiff bases, containing different donor atoms, also find use in analytical applications for metal coordination [1,2]. Especially derivatives of salicylaldehyde and diamine have been of greate interest. They act as tetra-dentate ligands and provide suitable coordination mode for transition metal ions, so that obtained complexes have great potential in catalysis and material chemistry [3]. Nickel complexes with tetra-dentate Schiff base ligands have been wildly investigated and several oxidation states for nickel have been reported in such complexes [4-6]. Nickel can form mono and trinuclear complexes with tetradentate Schiff base ligands [6]. we investigated the synthesis and crystal structure of several Schiff bases derived amino-thiotriazin and thiotriazol and their Ag(I) and Cu(I) complexes [1,7-10].

The nickel(II) is in the square planar environment formed by the tetra-dentate salen ligand. The phenoxy oxygen atoms O1, O2 and amine nitrogen atoms N1, N2 coordinate nickel(II) ion with the distances of d(Ni1—O1) = 1.855(2), d(Ni1—O2) = 1.847(1), d(Ni1—N1) = 1.845(2) Å and d(Ni1—N2) = 1.856(3) Å. These values indicate a slight deviation from an ideal square planar geometry, which is also reflected in the bond angles ∠N1– Ni1–N2, ∠N1–Ni1–O1, ∠N2–Ni1–O2, ∠O1–Ni1–O2, with values in the range of 85.81(9)° - 94.9(1)°. The C—O bond lengths of the coordinating oxygen atoms, d(C1—O1) = 1.302(3) Å and d(C16—O2) = 1.311(3) Å, are significantly shorter than those of the C–OCH_3 [d(C3—O3) = 1.362(4) Å and d(C14—O4) = 1.366(4) Å]. The bond lengths and bond angles around the Ni(II) ion are in accordance with the values found in other Ni(II) complexes with similar ligands [4,6].

Table 1. Data collection and handling.

Crystal:	orange block, size 0.08 × 0.12 × 0.14 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	12.10 cm^{-1}
Diffractometer, scan mode:	Nonius APEX2 CCD, φ/ω
$2\theta_{max}$:	60.2°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4477, 4477
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4183
$N(param)_{refined}$:	229
Programs:	SHELXS-97, SHELXL-97 [11]

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*a*	0.0681	0.5619	0.3618	0.030
H(4)	4*a*	–0.0671	0.8561	0.2686	0.038
H(5)	4*a*	–0.0212	1.1208	0.1642	0.036
H(7)	4*a*	0.0581	1.2933	0.1085	0.032
H(8A)	4*a*	0.1666	1.4521	0.1312	0.037
H(8B)	4*a*	0.1250	1.4585	0.0108	0.037
H(9A)	4*a*	0.1764	1.2680	–0.1298	0.035
H(9B)	4*a*	0.2182	1.4114	–0.0531	0.035
H(10)	4*a*	0.2780	1.1436	–0.0887	0.031
H(12)	4*a*	0.3509	0.9159	–0.1053	0.040
H(13)	4*a*	0.3975	0.6244	–0.0409	0.040
H(15)	4*a*	0.2812	0.4506	0.2105	0.030
H(17A)	4*a*	–0.0326	0.2893	0.5066	0.045
H(17B)	4*a*	0.0159	0.4417	0.5272	0.045
H(17C)	4*a*	0.0135	0.2901	0.3989	0.045
H(18A)	4*a*	0.3849	0.1120	0.2527	0.046
H(18B)	4*a*	0.3256	0.1488	0.2136	0.046
H(18C)	4*a*	0.3538	0.3049	0.3162	0.046

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)	4*a*	0.17344(1)	0.96303(5)	0.13401(5)	0.0223(1)	0.0190(1)	0.0231(1)	–0.0003(1)	0.0007(2)	0.0022(2)
O(1)	4*a*	0.12885(8)	0.8066(3)	0.2403(2)	0.021(1)	0.025(1)	0.028(1)	0.0018(8)	0.0026(8)	0.0041(8)
O(2)	4*a*	0.22053(8)	0.7512(3)	0.1656(2)	0.0208(9)	0.0250(9)	0.032(1)	0.0017(7)	0.0031(8)	0.0051(8)
O(3)	4*a*	–0.03295(9)	0.5371(4)	0.3855(2)	0.026(1)	0.035(1)	0.036(1)	–0.0009(9)	0.0058(9)	0.006(1)
O(4)	4*a*	0.37381(7)	0.3463(3)	0.1258(3)	0.0232(9)	0.037(1)	0.044(1)	0.0067(8)	–0.001(1)	0.000(1)
N(1)	4*a*	0.1269(1)	1.1785(3)	0.1093(2)	0.031(1)	0.0184(9)	0.028(1)	0.0001(9)	–0.002(1)	0.0024(9)
N(2)	4*a*	0.2160(1)	1.1102(4)	0.0170(2)	0.030(1)	0.023(1)	0.022(1)	–0.0024(9)	–0.002(1)	0.0020(9)
C(1)	4*a*	0.0790(1)	0.8298(4)	0.2490(3)	0.023(1)	0.022(1)	0.022(1)	0.002(1)	–0.001(1)	–0.002(1)
C(2)	4*a*	0.0504(1)	0.6740(4)	0.3187(3)	0.025(1)	0.023(1)	0.026(1)	0.001(1)	0.002(1)	0.002(1)
C(3)	4*a*	–0.0029(1)	0.6843(5)	0.3243(3)	0.025(2)	0.027(1)	0.025(1)	–0.001(1)	0.002(1)	–0.002(1)
C(4)	4*a*	–0.0304(1)	0.8514(5)	0.2651(3)	0.022(1)	0.035(2)	0.038(2)	0.004(1)	–0.002(1)	–0.003(1)
C(5)	4*a*	–0.0030(1)	1.0064(5)	0.2027(3)	0.029(2)	0.028(1)	0.033(2)	0.005(1)	–0.003(1)	0.001(1)
C(6)	4*a*	0.0515(1)	1.0028(5)	0.1933(3)	0.025(1)	0.024(1)	0.027(1)	0.004(1)	–0.004(1)	–0.002(1)
C(7)	4*a*	0.0779(1)	1.1739(4)	0.1331(4)	0.032(1)	0.020(1)	0.028(1)	0.0050(9)	0.001(2)	0.000(2)
C(8)	4*a*	0.1510(1)	1.3701(5)	0.0569(3)	0.037(2)	0.019(1)	0.036(2)	–0.001(1)	–0.003(1)	0.003(1)
C(9)	4*a*	0.1920(1)	1.3000(5)	–0.0410(3)	0.039(2)	0.022(1)	0.027(1)	–0.004(1)	–0.001(1)	0.004(1)
C(10)	4*a*	0.2612(1)	1.0543(5)	–0.0260(3)	0.027(1)	0.029(1)	0.022(1)	–0.007(1)	0.001(1)	0.003(1)
C(11)	4*a*	0.2874(1)	0.8674(5)	0.0144(3)	0.025(1)	0.029(1)	0.024(1)	–0.004(1)	0.002(1)	–0.001(1)
C(12)	4*a*	0.3367(1)	0.8229(6)	–0.0404(3)	0.028(2)	0.042(2)	0.029(2)	–0.009(1)	0.006(1)	0.002(1)
C(13)	4*a*	0.3644(1)	0.6506(6)	–0.0030(3)	0.022(1)	0.045(2)	0.033(2)	–0.002(1)	0.006(1)	–0.003(1)
C(14)	4*a*	0.3431(1)	0.5124(5)	0.0924(3)	0.021(1)	0.030(1)	0.036(2)	–0.002(1)	–0.002(1)	–0.008(1)
C(15)	4*a*	0.2951(1)	0.5472(4)	0.1471(4)	0.019(1)	0.027(1)	0.029(2)	–0.0036(9)	0.002(1)	–0.001(1)
C(16)	4*a*	0.2660(1)	0.7259(4)	0.1096(3)	0.020(1)	0.026(1)	0.022(2)	–0.0045(9)	–0.000(1)	–0.003(1)
C(17)	4*a*	–0.0070(2)	0.3769(6)	0.4605(4)	0.040(2)	0.035(2)	0.037(2)	0.002(2)	0.010(2)	0.009(2)
C(18)	4*a*	0.3584(1)	0.2181(6)	0.2356(4)	0.032(2)	0.040(2)	0.043(2)	0.010(1)	–0.004(2)	–0.000(2)

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