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Refinement of the crystal structure of bis(*N*-*n*-butylsalicylaldiminato-κ^2*N*,*O*)nickel(II), $Ni(C_{11}H_{14}NO)_2$

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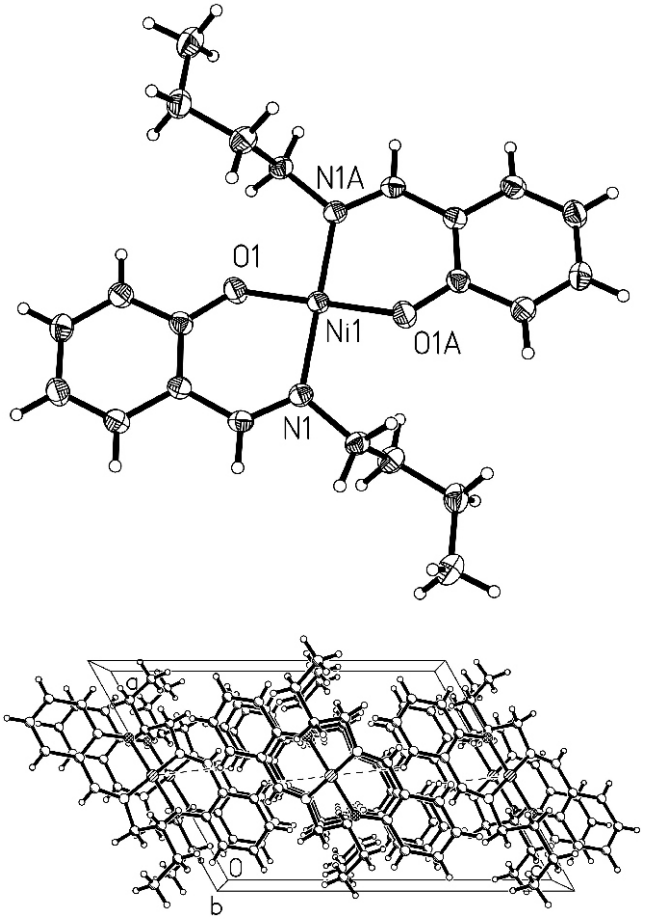

Abstract

$C_{22}H_{28}N_2NiO_2$, monoclinic, $P12_1/c1$ (no. 14), a = 10.857(1) Å, b = 7.2521(8) Å, c = 14.704(1) Å, β = 119.352(6)°, V = 1009.2 Å^3, Z = 2, $R_{gt}(F)$ = 0.033, $wR_{ref}(F^2)$ = 0.090, T = 298 K.

Source of material

Salicylaldehyde (244 mg, 2 mmol) and *n*-butylamine (146 mg, 2 mmol) were dissolved in an aqueous ethanol solution (10 mL). The mixture was stirred at room temperature for 1 h to give a clear yellow solution, which was added to a methanol solution (10 mL) of $NiCl_2 \cdot 7H_2O$ (512 mg, 2 mmol). The mixture was stirred for another 30 min at room temperature to give a brown solution and then filtered. The filtrate was kept in air for 7 days, forming dark blue block-shaped crystals. The crystals were isolated and dried in a vacuum desiccator containing anhydrous $CaCl_2$ (yield 62 %).

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All reagents and solvents were used as obtained without further purification.

Elemental analysis — found: C, 64.00 %; H, 6.92 %; N, 6.61 %; calculated for $C_{22}H_{28}N_2NiO_2$: C, 64.26 %; H, 6.86 %; N, 6.81 %.

IR data are available in the CIF file.

Discussion

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with d(C—H) = 0.93-0.97 Å and U_{iso}(H) = 1.2 U_{eq}(carrier) or 1.5 U_{eq}(methyl groups).

Discussion

Nickel is an important urease-required element. Due to their biologically relevant important behaviors, many nickel complexes with Schiff base have been extensively investigated in the past decades [1-3]. For example, the Schiff base nickel(II) complex with *N*,*O*-donor ligands possesses potent inhibitory activity against urease [4]. Due to their structural and electronic characteristics, bis(salicylaldiminato)nickel(II) complexes, possessing the substituents *R* and *X* on the imino and aromatic moieties, have been widely investigated since early 1960s [5,6]. Interestingly, these complexes may display different structural arrangements, whose stability strongly depends on the steric hindrance of the *R* group bound to the imino moiety. For example, when *R* is the linear *n*-butyl group a very small distortion from planarity is observed [7].

In the title crystal structure with the *n*-butyl group bound to the imino moiety, the Schiff base Ni(II) complex has inversion symmetry; Ni(II) lies on the center of symmetry (symmetry code: 1–*x*,2–*y*,–*z*) and is four-coordinated by two N atoms and two O atoms from two bidentate Schiff base ligands, which affords a square planar *trans*-[NiN_2O_2] coordination. The bond lengths are d(Ni1—O1) = 1.828(1) Å and d(Ni1—N1) = 1.928(1) Å. The double bond length d(N1—C7) = 1.293(2) Å is somewhat shorter than that observed in the previously reported similar complex bis(*N*-*n*-butyl-2-hydroxy-1-naphthaldiiminato)-nickel(II) (1.310(2) Å) [8].

Table 1. Data collection and handling.

Crystal:	dark blue block, size 0.18 × 0.22 × 0.28 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.80 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	53°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10480, 2086
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1926
$N(param)_{refined}$:	125
Program:	SHELXTL [9]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(3)	4*e*	0.1245	1.0083	0.0313	0.057
H(4)	4*e*	0.0872	0.8902	0.1600	0.058
H(5)	4*e*	0.2574	0.7097	0.2923	0.061
H(6)	4*e*	0.4651	0.6483	0.2928	0.056
H(7)	4*e*	0.6110	0.6600	0.2134	0.047
H(8A)	4*e*	0.7790	0.6685	0.1852	0.048
H(8B)	4*e*	0.7939	0.8314	0.1210	0.048
H(9A)	4*e*	0.6454	0.6566	–0.0351	0.060
H(9B)	4*e*	0.6601	0.4900	0.0375	0.060
H(10A)	4*e*	0.8195	0.4691	–0.0288	0.065
H(10B)	4*e*	0.8928	0.6506	0.0307	0.065
H(11A)	4*e*	0.9725	0.5012	0.1892	0.109
H(11B)	4*e*	1.0245	0.3954	0.1213	0.109
H(11C)	4*e*	0.8960	0.3198	0.1309	0.109

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*b*	½	1	0	0.0295(2)	0.0389(2)	0.0320(2)	0.00103(8)	0.0127(1)	0.00396(8)
O(1)	4*e*	0.3368(1)	0.9688(2)	0.0055(1)	0.0334(6)	0.0714(8)	0.0461(7)	0.0058(5)	0.0180(5)	0.0215(5)
N(1)	4*e*	0.5976(1)	0.8122(2)	0.10356(8)	0.0338(6)	0.0383(6)	0.0353(6)	0.0019(4)	0.0142(5)	0.0007(5)
C(1)	4*e*	0.4232(2)	0.7908(2)	0.1619(1)	0.0396(7)	0.0349(7)	0.0386(7)	–0.0008(5)	0.0184(6)	0.0010(5)
C(2)	4*e*	0.3205(1)	0.9001(2)	0.0816(1)	0.0361(7)	0.0388(7)	0.0409(7)	–0.0032(5)	0.0178(6)	0.0027(6)
C(3)	4*e*	0.1939(2)	0.9358(3)	0.0834(1)	0.0357(7)	0.0532(9)	0.0495(8)	0.0015(7)	0.0180(7)	0.0085(8)
C(4)	4*e*	0.1716(2)	0.8652(2)	0.1607(1)	0.0417(8)	0.0539(9)	0.0543(9)	–0.0040(7)	0.0279(7)	–0.0030(7)
C(5)	4*e*	0.2732(2)	0.7568(2)	0.2401(1)	0.0577(9)	0.0543(9)	0.0506(9)	–0.0019(8)	0.0342(8)	0.0031(7)
C(6)	4*e*	0.3968(2)	0.7208(2)	0.2400(1)	0.0535(9)	0.0440(8)	0.0440(8)	0.0049(7)	0.0254(7)	0.0072(6)
C(7)	4*e*	0.5530(2)	0.7458(2)	0.1638(1)	0.0413(7)	0.0363(7)	0.0376(7)	0.0050(5)	0.0167(6)	0.0044(5)
C(8)	4*e*	0.7322(1)	0.7327(2)	0.1189(1)	0.0346(7)	0.0431(8)	0.0396(7)	0.0057(6)	0.0155(6)	0.0037(6)
C(9)	4*e*	0.7075(2)	0.5990(2)	0.0316(1)	0.0464(8)	0.0490(9)	0.0520(9)	0.0003(7)	0.0222(7)	–0.0066(7)
C(10)	4*e*	0.8438(2)	0.5407(3)	0.0335(2)	0.059(1)	0.0534(9)	0.061(1)	–0.0042(8)	0.039(1)	–0.0071(9)
C(11)	4*e*	0.9432(2)	0.4292(4)	0.1272(2)	0.060(1)	0.085(2)	0.086(2)	0.021(1)	0.046(1)	0.011(1)

References

1. Kolotilov, S. V.; Cador, O.; Golhen, S.; Shvets, O.; Ilyin, V. G.; Pavlishchuk, V. V.; Ouahab, L.: Synthesis, structure, sorption and magnetic properties of Ni(II) and Cu(II) complexes with thiosemicarbazone of 2-hydroxybenzaldehyde, bridged by 4,4′-bipyridine. Inorg. Chim. Acta. **360** (2007) 1883-1889.
2. Li, Y. G.; Shi, D. H.; Zhu, H. L.; Yan, H.; Weng, S. N.: Transition metal complexes (*M* = Cu, Ni and Mn) of Schiff-base ligands: Syntheses,crystal structures, and inhibitory bioactivities against urease and xanthine oxidase. Inorg. Chim. Acta **360** (2007) 2881-2889.
3. Akitsu, T.; Einaga, Y.: Synthesis, crystal structures and electronic properties of Schiff base nickel(II) complexes: Towards solvatochromism induced by a photochromic solute. Polyhedron **24** (2005) 1869-1877.
4. Cui, Y. M.; Yan, W. X.; Cai, Y. J.; Chen, W.; Zhu, H. L.: Synthesis, Molecular docking, and inhibitory activity of a Ni Schiff-base complex against urease. J. Coord. Chem. **63** (2010) 3706-3713.
5. Holm, R. H.; Everett Jr., R. H.; Chakravorty, A. in: *Progress in Inorganic Chemistry* (Ed. S. J. Lippard), Vol. 7, p. 83, Wiley, New York, 1966.
6. Holm, R. H.; O'Connor, M. J.: In *Progress in Inorganic Chemistry* (Ed. S. J. Lippard), vol. 14, p. 241, Wiley, New York, 1971.
7. Frasson, E.; Panattoni, C.; Sacconi, L.: Bis(*N*-*n*-Butylsalicylaldiminato)nickel(II). Gazz. Chim. Ital. **92** (1962) 1470.
8. Elerman, Y.; Elmali, A.; Svoboda, I.; Fuess, H.: Bis(*N*-*n*-butyl-2-hydroxy-1-naphthaldiminato)nickel(II). Acta Crystallogr. **C54** (1998) 1076-1078.
9. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.