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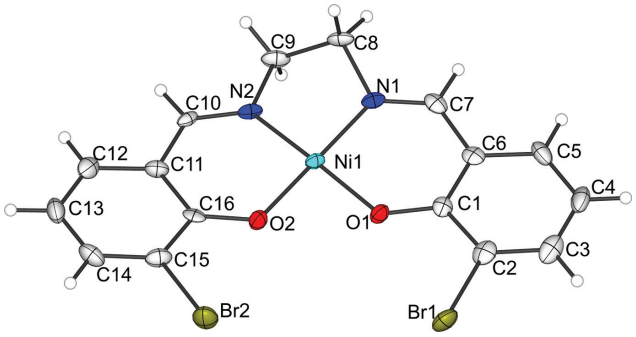
Crystal structure of bis((3-bromosalicylidene)-ethylenediaminato-κ⁴*N*,*N*′,*O*,*O*′) nickel (II), $C_{16}H_{12}Br_2NiN_2O_2$



https://doi.org/10.1515/ncrs-2020-0154
Received March 23, 2020; accepted April 7, 2020; available online April 24, 2020

Abstract

$C_{16}H_{12}Br_2NiN_2O_2$, monoclinic, $P2_1/c$ (no. 14), $a = 11.7989(9)$ Å, $b = 11.9007(7)$ Å, $c = 12.7164(17)$ Å, $\beta = 119.779(7)°$, $V = 1549.8(3)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0324$, $wR_{ref}(F^2) = 0.0687$, $T = 173(2)$ K.

CCDC no.: 1919057

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.09 × 0.09 × 0.08 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	6.42 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5819, 2723, 0.027
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2289
$N(param)_{refined}$:	208
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4], Diamond [5]

Source of material

All commercially available reagents were used as supplied. The halogenated tetradentate Schiff base ligand (3*Br*-salen) was prepared by mixing 3-bromosalicylaldehyd (0.147 g, 1.0 mmol) and ethylenediamine (0.03 g, 0.5 mmol) in 30 mL of methanol. Then, $NiCl_2{\cdot}6H_2O$ (0.237 g, 1 mmol) was added to the aforementioned red solution. The resulting red suspension was heated for another 3 h. After cooling, the red filtrate was sealed in a beaker and kept undisturbed at room temperature. The red block crystals of the title compound were afforded after one week.

Experimental details

The structure was solved by direct methods. Hydrogen atoms were placed in calculated positions and included in the refinement in the riding model approximation, with U_{iso}(H) set to $1.2U_{eq}$(C).

Comment

The study on Schiff base complexes have attracted intense attention due to their preparative accessibility and structural diversity [6–9]. Nickel and its complexes widely exist in living organisms, having allergic and metastasis effects on tissue and hormone. In this field, a large number of salen-type Schiff-base ligands have been used for the construction of Ni(II) complexes [10, 11]. However, related research about halogenated Schiff base ligands is still rare [12]. With the aim of exploring the relationship between the constitution of Schiff-base ligands and the complex structures, we have engaged in the research of isolating Ni complexes with different halogenated Schiff bases.

The title compound crystallizes in the space group $P2_1/c$, which is constructed by one crystallographically unique Ni(II) and one dibromo substituated schiff-base ligand 3Br-salen. As shown in the figure, the central Ni atom exhibits a

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	−0.15935(4)	0.06272(4)	−0.22080(4)	0.01653(12)
Br2	−0.48446(4)	−0.04963(4)	−0.63680(4)	0.01761(13)
Ni1	−0.03013(5)	−0.13163(4)	−0.47173(5)	0.00912(14)
O1	−0.0440(3)	−0.0561(2)	−0.3518(2)	0.0114(6)
O2	−0.2094(3)	−0.1326(2)	−0.5524(2)	0.0122(6)
N1	0.1493(3)	−0.1216(3)	−0.3927(3)	0.0101(7)
N2	−0.0174(3)	−0.2148(3)	−0.5884(3)	0.0099(7)
C1	0.0505(4)	−0.0219(3)	−0.2475(4)	0.0105(9)
C2	0.0197(4)	0.0360(3)	−0.1671(4)	0.0153(9)
C3	0.1148(4)	0.0700(3)	−0.0543(4)	0.0178(10)
H3	0.0910	0.1060	−0.0033	0.021*
C4	0.2458(4)	0.0517(4)	−0.0152(4)	0.0186(10)
H4	0.3096	0.0748	0.0615	0.022*
C5	0.2801(4)	−0.0009(4)	−0.0910(4)	0.0154(9)
H5	0.3680	−0.0123	−0.0654	0.018*
C6	0.1852(4)	−0.0379(3)	−0.2066(4)	0.0109(9)
C7	0.2265(4)	−0.0850(3)	−0.2855(4)	0.0123(9)
H7	0.3158	−0.0894	−0.2571	0.015*
C8	0.2030(4)	−0.1515(3)	−0.4723(4)	0.0126(9)
H8A	0.2926	−0.1769	−0.4244	0.015*
H8B	0.2007	−0.0872	−0.5200	0.015*
C9	0.1177(4)	−0.2447(3)	−0.5535(4)	0.0146(9)
H9A	0.1273	−0.2510	−0.6247	0.018*
H9B	0.1415	−0.3159	−0.5108	0.018*
C10	−0.1121(4)	−0.2495(3)	−0.6915(4)	0.0098(9)
H10	−0.0904	−0.2910	−0.7409	0.012*
C11	−0.2470(4)	−0.2284(3)	−0.7343(4)	0.0124(9)
C12	−0.3377(4)	−0.2640(3)	−0.8521(4)	0.0150(10)
H12	−0.3089	−0.3056	−0.8963	0.018*
C13	−0.4673(4)	−0.2386(4)	−0.9029(4)	0.0172(10)
H13	−0.5266	−0.2640	−0.9802	0.021*
C14	−0.5100(4)	−0.1738(3)	−0.8373(4)	0.0162(10)
H14	−0.5973	−0.1530	−0.8724	0.019*
C15	−0.4227(4)	−0.1411(3)	−0.7210(4)	0.0139(9)
C16	−0.2880(4)	−0.1667(3)	−0.6632(4)	0.0099(9)

tetra-coordinated geometry, which is defined by N_2O_2 in the equatorial plane from 3*Br*-salen. The bond lengths of Ni(1)-O(1), Ni(1)-O(2), Ni(1)-N(1) and Ni(1)-N(2) are 1.839(5), 1.843(4), 1.837(5) and 1.850(6) Å, respectively. In the crystal structure, the neutral molecules are linked by weak intermolecular C—H···O hydrogen bonds and aromatic face-to-face interactions to form a three-dimensional supermolecular network. [C8—H8(B)···O1: 3.234(5) Å; interactions $Cg1\cdots Cg2^{i}$ (symmetry code: $1-X$, $1-Y$, $-Z$) centroid-to-centroid distance = 3.8392 Å, where *Cg1* is the centroid of the C1—C6 ring and *Cg2* is the centroid of the C11—C16 ring].

Acknowledgements: This work was supported by Fund for Less Developed Regions of the National Natural Science Foundation of China (no. 31760257); Joint Basic Research Program (partial)of Yunnan Local Undergraduate Universities (2017FH001-002); The reserve academic and technical leaders of Yunnan Province (2019HB098).

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