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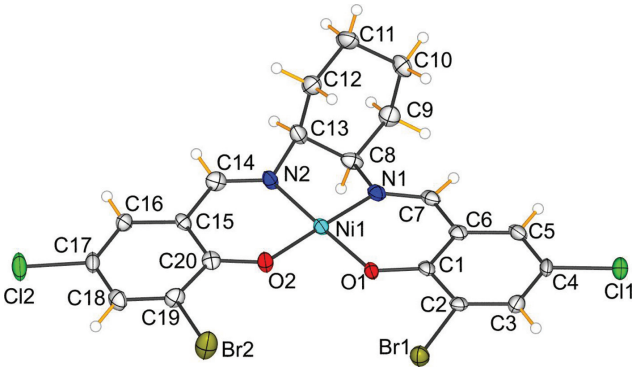
Crystal structure of 6,6′-((cyclohexane-1,2-diylbis(azanylylidene))bis(methanylylidene))bis(2-bromo-4-chlorophenolato-κ^4*N*,*N*′,*O*,*O*′)nickel(II), $C_{20}H_{16}Br_2Cl_2NiN_2O_2$



https://doi.org/10.1515/ncrs-2020-0061
Received January 31, 2020; accepted March 10, 2020; available online March 25, 2020

Abstract

$C_{20}H_{16}Br_2Cl_2NiN_2O_2$, monoclinic, $P2_1/c$ (no. 14), $a = 12.6946(7)$ Å, $b = 11.9262(7)$ Å, $c = 14.9398(9)$ Å, $\beta = 115.1400°$, $V = 2047.6(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0386$, $wR_{ref}(F^2) = 0.0834$, $T = 273(2)$ K.

CCDC no.: 1989370

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

All hydrogen atoms were placed in calculated positions and refined using a riding model.

Experimental details

All commercially available reagents were used as supplied. The title compound was synthesized according

Table 1: Data collection and handling.

Crystal:	Green block
Size:	0.12 × 0.12 × 0.16 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	5.1 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-III, φ and ω
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9563, 4135, 0.037
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3137
$N(param)_{refined}$:	262
Programs:	CrysAlis^{PRO} [1], SHELX [2], Olex2 [3], Diamond [4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.41428(4)	0.63749(4)	0.10110(3)	0.02423(12)
Br2	0.17101(4)	0.95819(4)	0.13096(4)	0.03514(15)
C1	0.5922(3)	0.7900(3)	0.1116(3)	0.0178(9)
C2	0.5621(3)	0.6755(3)	0.1065(3)	0.0182(9)
C3	0.6324(4)	0.5907(4)	0.1027(3)	0.0214(10)
H3	0.6087	0.5164	0.0996	0.026*
C4	0.7396(4)	0.6164(4)	0.1035(3)	0.0213(10)
C5	0.7732(4)	0.7256(4)	0.1064(3)	0.0222(10)
H5	0.8444	0.7422	0.1057	0.027*
C6	0.7010(4)	0.8135(4)	0.1105(3)	0.0186(9)
C7	0.7367(4)	0.9271(4)	0.1058(3)	0.0196(9)
H7	0.8054	0.9376	0.0988	0.023*
C8	0.7183(4)	1.1297(3)	0.0941(3)	0.0220(10)
H8	0.6703	1.1518	0.0255	0.026*
C9	0.8462(4)	1.1435(4)	0.1145(3)	0.0263(10)
H9A	0.8652	1.0888	0.0756	0.032*
H9B	0.8571	1.2175	0.0928	0.032*
C10	0.9293(4)	1.1294(4)	0.2220(3)	0.0264(10)
H10A	1.0078	1.1462	0.2307	0.032*
H10B	0.9276	1.0522	0.2417	0.032*
C11	0.8964(4)	1.2071(4)	0.2873(4)	0.0309(11)
H11A	0.9067	1.2845	0.2727	0.037*
H11B	0.9474	1.1931	0.3562	0.037*
C12	0.7696(4)	1.1884(4)	0.2700(3)	0.0264(10)
H12A	0.7608	1.1129	0.2900	0.032*
H12B	0.7497	1.2406	0.3102	0.032*
C13	0.6878(4)	1.2051(4)	0.1620(3)	0.0231(10)
H13	0.6891	1.2838	0.1436	0.028*
C14	0.4993(4)	1.2480(4)	0.1513(3)	0.0214(9)

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Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H14	0.5261	1.3216	0.1629	0.026*
C15	0.3831(3)	1.2267(3)	0.1413(3)	0.0166(9)
C16	0.3124(4)	1.3196(3)	0.1369(3)	0.0197(9)
H16	0.3413	1.3922	0.1427	0.024*
C17	0.1995(4)	1.3008(3)	0.1237(3)	0.0216(10)
C18	0.1571(4)	1.1929(4)	0.1192(3)	0.0228(10)
H18	0.0811	1.1814	0.1111	0.027*
C19	0.2277(4)	1.1034(3)	0.1268(3)	0.0216(10)
C20	0.3428(4)	1.1140(3)	0.1352(3)	0.0174(9)
Cl1	0.82807(10)	0.50800(10)	0.09645(9)	0.0324(3)
Cl2	0.10612(10)	1.41293(10)	0.11323(10)	0.0368(3)
N1	0.6809(3)	1.0156(3)	0.1106(2)	0.0187(8)
N2	0.5686(3)	1.1730(3)	0.1453(3)	0.0189(8)
Ni1	0.54326(4)	1.01994(4)	0.12688(4)	0.01671(14)
O1	0.5200(2)	0.8663(2)	0.1153(2)	0.0175(6)
O2	0.4034(2)	1.0254(2)	0.1377(2)	0.0205(6)

to classical organic synthesis of Schiff-bases, 3-bromo-5-chlorosalicylaldehyde 0.471 g (1.0 mmol) and (±)-1.2-cyclohexanediamine 0.5 mL (0.5 mmol) were dissolved in a mixture containing 20 mL ethanol and 10 mL methanol, the yellow suspension was then stirred at 60 °C for 30 min. Then, $Ni(NO_3)_2 \cdot 6H_2O$ (0.300 g, 0.5 mmol) was added to the above solution and the resulting yellow mixture was further stirred for another 30 min and filtered. The green black crystals of the title compound were obtained after 1 week by slow evaporation.

Comment

Nickel(II) containing Schiff-base complexes have received much attention owing to their superior biological and catalytic properties. In the last several years, related research is becoming one of the most developing fields in coordination chemistry [5, 6]. Whereas, the literatures about halogenated salen-type Schiff-bases and Nickel(II) based Schiff-base complex is rather rare [7–9]. As a part of our current research interest on the exploration of the regulating effect of Schiff base ligands in transition metal complexes, we report a new Nickel(II) complex based on a halogenated Schiff base ligand.

Single-crystal X-ray diffraction reveals that the title compound crystallizes in the monoclinic space group $P2_1/c$, which unit cell contains two pairs of racemic neutral Ni(II) monomers. The dihedral angle calculated between the planes of two aryl rings is 6.87(10)°. Thus the whole molecule exhibits an almost coplanar configuration. The central Ni^{II} exhibits a tetra-coordinated environment, which is defined by N_2O_2 in the equatorial plane from halogenated ligand. The bond lengths of Ni—O(N) are in the range of 1.851(2)∼1.864(4) Å and bond angles of O(N)—Ni-O vary from 85.53(3) to 177.71(2)°, which is compare favorably with the corresponding values observed in salen-type Ni(II) analogous [9–11].

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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