Jianchang Xiao, Cun Zhou, Hui Tan, Meifen Huang and Qiong Wu*

Crystal structure of bis((3-chlorosalicylidene)-ethylenediaminato- $\kappa^4\text{N},\text{N}',\text{O},\text{O}'$)nickel (II), $\text{C}_{16}\text{H}_{12}\text{Cl}_2\text{NiN}_2\text{O}_2$

Table 1: Data collection and handling.

Crystal:	Green block
Size:	0.18 × 0.16 × 0.14 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.66 mm ⁻¹
Diffractometer, scan mode:	Xcalibur, φ and ω
θ_{max} , completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	15534, 2635, 0.107
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1760
$N(\text{param})_{\text{refined}}$:	208
Programs:	CrysAlis ^{PRO} [1], SHELX [2], Olex2 [3], Diamond [4]

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Abstract

$\text{C}_{16}\text{H}_{12}\text{Cl}_2\text{NiN}_2\text{O}_2$, orthorhombic, *Pbca* (no. 61), $a = 12.5166(12)$ Å, $b = 12.5881(14)$ Å, $c = 19.0086(19)$ Å, $V = 2995.0(5)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0435$, $wR_2 = 0.0826$, $T = 296.15$.

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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.

***Corresponding author: Qiong Wu**, Department of Chemical Science and Technology, Kunming University, Yunnan, Kunming 65200, P.R. China, e-mail: wuqiongkm@163.com. <https://orcid.org/0000-0001-7931-6750>

Jianchang Xiao, Hui Tan and Meifen Huang: Department of Chemical Science and Technology, Kunming University, Yunnan, Kunming 65200, P.R. China

Cun Zhou: The High School Affiliated to Minzu University of China Kunming Wuhua, Experimental School, 6# Chenjiaying Road, Wuhua District, Kunming, Yunnan 650102, P.R. China

Experimental details

All commercially available reagents were used as supplied. The halogenated tetradentate Schiff base ligand (3-Cl-salen) was prepared by mixing 3-chlorosalicylaldehyde (0.147 g, 1.0 mmol) and ethylenediamine (0.03 g, 0.5 mmol) in 30 mL of methanol. Then, $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (0.300 g, 0.5 mmol) was added to this yellow solution. The resulting dark red suspension was heated for another 2 h. After cooling, the brownish 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.

Comment

Nickel(II) containing Schiff-base complexes have received much attention owing to their superior biological and catalytic properties. In the last years, related research is one of the most developing fields in coordination chemistry [5, 6]. But halogenated salen-type Schiff-base complexes are 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 orthorhombic space group *Pbca*. As it is a centrosymmetric space group the unit cell contains four pairs of racemic neutral Ni(II) mononuclear complexes (see the Figure). 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}

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Ni1	0.63061(3)	0.53407(4)	0.49571(2)	0.02082(15)
Cl1	0.59054(8)	0.54196(8)	0.24945(5)	0.0323(3)
Cl2	0.44928(8)	0.83946(9)	0.41387(5)	0.0383(3)
O1	0.64041(18)	0.5244(2)	0.39892(12)	0.0247(6)
O2	0.55203(19)	0.6564(2)	0.47918(12)	0.0245(6)
N1	0.7154(2)	0.4165(2)	0.51177(15)	0.0221(8)
N2	0.6122(2)	0.5398(2)	0.59170(15)	0.0225(7)
C1	0.6837(3)	0.4488(3)	0.36249(19)	0.0222(9)
C2	0.6708(3)	0.4457(3)	0.28871(19)	0.0244(10)
C3	0.7157(3)	0.3698(3)	0.2467(2)	0.0319(10)
H3	0.7055	0.3719	0.1983	0.038*
C4	0.7767(3)	0.2891(3)	0.2768(2)	0.0365(11)
H4	0.8083	0.2380	0.2483	0.044*
C5	0.7901(3)	0.2851(3)	0.3475(2)	0.0283(10)
H5	0.8296	0.2301	0.3673	0.034*
C6	0.7446(3)	0.3635(3)	0.39144(19)	0.0221(9)
C7	0.7565(3)	0.3536(3)	0.46518(19)	0.0251(10)
H7	0.7973	0.2971	0.4816	0.030*
C8	0.7401(3)	0.3994(3)	0.58674(18)	0.0317(10)
H8A	0.8040	0.4380	0.6000	0.038*
H8B	0.7512	0.3245	0.5961	0.038*
C9	0.6451(3)	0.4399(3)	0.62697(18)	0.0270(10)
H9A	0.5875	0.3884	0.6259	0.032*
H9B	0.6642	0.4535	0.6756	0.032*
C10	0.5672(3)	0.6129(3)	0.62790(19)	0.0244(10)
H10	0.5643	0.6038	0.6764	0.029*
C11	0.5210(3)	0.7077(3)	0.5993(2)	0.0244(9)
C12	0.4766(3)	0.7826(3)	0.6460(2)	0.0306(10)
H12	0.4805	0.7705	0.6942	0.037*
C13	0.4281(3)	0.8726(3)	0.6218(2)	0.0346(11)
H13	0.4007	0.9223	0.6532	0.042*
C14	0.4200(3)	0.8892(3)	0.5500(2)	0.0320(10)
H14	0.3862	0.9498	0.5332	0.038*
C15	0.4617(3)	0.8163(3)	0.5034(2)	0.0286(9)
C16	0.5140(3)	0.7220(3)	0.5262(2)	0.0227(9)

exhibits a tetra-coordinated environment, which is defined by N₂O₂ in the equatorial plane from the 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 comparable with the corresponding values observed in analogous salen-type Ni(II) complexes [9–11].

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