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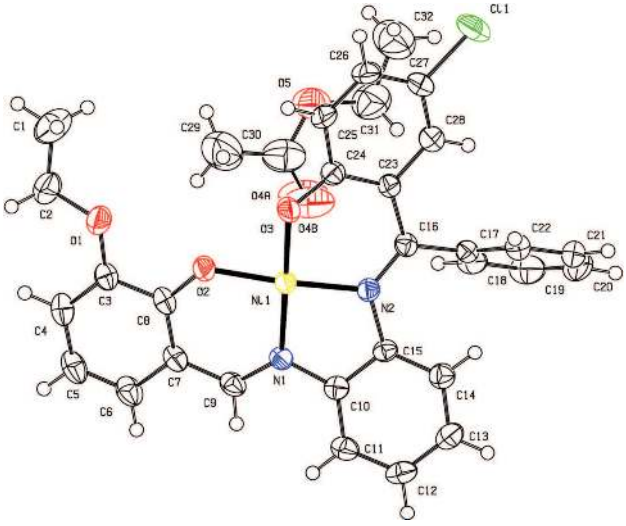
Crystal structure of [4-chloro-2-(((2-((3-ethoxy-2-oxidobenzylidene)amino)phenyl)imino)(phenyl)methyl)phenolato-κ^4*N*,*N*′,*O*,*O*′}nickel(II) - ethyl acetate (1/1), $C_{32}H_{29}ClN_2NiO_5$



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Abstract

$C_{32}H_{29}ClN_2NiO_5$, triclinic, $P\bar{1}$ (no. 2), $a = 9.7657(5)$ Å, $b = 10.1262(6)$ Å, $c = 16.0745(9)$ Å, $\alpha = 83.025(2)°$, $\beta = 77.437(2)°$, $\gamma = 66.240(2)°$, $V = 1419.02(14)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0403$, $wR_{ref}(F^2) = 0.0908$, $T = 293(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

(*E*)-2-(((2-aminophenyl)imino)(phenyl)methyl)-4-chlorophenol (mono-imine) was synthesized by the method described [4]. 5-Chloro-2-hydroxybenzophenone (0.233 g, 1 mmol), 1,2-diaminobenzene (0.108 g, 1 mmol), piperidine (0.085 g, 1 mmol), and triethylorthoformate (0.148 g, 1 mmol) were refluxed for 2 h in ethanol (10 mL), then the solution was cooled and the mono-imine was obtained by filtration. Unsymmetrical tetradentate Schiff base ligand (*UTSBL*) was prepared by the obtained mono-imine and 3-ethyoxyl-2-hydroxybenzaldehyde. The mono-imine (0.323 g, 1 mmol) and 3-methoxy-2-hydroxybenzaldehyde (0.168 g, 1 mmol) were added to the solution of ethanol (10 mL) and refluxed for 2 h. The precipitate thus formed was collected by filtration. *UTSBL* (0.471 g, 1 mmol) and nickel acetate tetrahydrate (0.249 g, 1 mmol) were refluxed in methanol and ethyl acetate for about 1 h. After filtration, the solution was evaporated slowly at room temperature to obtain green prismatic crystals.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.32 × 0.21 × 0.11 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.82 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ_{max}, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	32913, 4987, 0.053
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4029
$N(param)_{refined}$:	372
Programs:	Bruker programs [1], SHELX [2]

Experimental details

The H atoms were positioned geometrically with d(C—H) = 0.93–0.98 Å and refined as riding with $U_{iso}(H) = 1.2\ U_{eq}$(carrier) or 1.5 U_{eq}(methyl).

Discussion

The complexes of symmetrical or unsymmetrical sal(salphen)tetradentate (N_2O_2) Schiff bases raise certain concerns for their intriguing applications in various fields [4–8]. There have been reported similar symmetrical crystal strcutures of Ni(II) [9, 10]. Herein, we report the crystal structure of a similar Ni(II) complex.

The dihedral angles between the aryl rings (C3—C4—C5—C6—C7—C8 and C10—C11—C12—C13—C14—C15), (C3—C4—C5—C6—C7—C8 and C23—C24—C25—C26—C27—C28),

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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}
Ni1	0.30792(4)	0.37454(4)	0.64762(2)	0.02772(11)
Cl1	−0.23263(13)	0.32020(12)	1.01227(6)	0.0815(3)
O2	0.42601(19)	0.47224(19)	0.65928(11)	0.0341(4)
O3	0.1748(2)	0.4804(2)	0.73702(12)	0.0391(5)
O1	0.5733(2)	0.5936(2)	0.71920(12)	0.0437(5)
N1	0.4329(2)	0.2829(2)	0.55049(13)	0.0279(5)
N2	0.2014(2)	0.2561(2)	0.64774(13)	0.0275(5)
O5	0.3524(3)	0.1789(3)	0.93543(15)	0.0694(7)
C23	0.0529(3)	0.3147(3)	0.78929(16)	0.0302(6)
C8	0.5644(3)	0.4484(3)	0.61874(16)	0.0302(6)
C16	0.1132(3)	0.2266(3)	0.71513(16)	0.0278(6)
C24	0.0796(3)	0.4414(3)	0.79322(16)	0.0316(6)
C15	0.2452(3)	0.1860(3)	0.56815(15)	0.0276(6)
C10	0.3713(3)	0.2028(3)	0.51498(16)	0.0274(6)
C12	0.3538(3)	0.0722(3)	0.40630(17)	0.0392(7)
H12	0.390948	0.032008	0.352885	0.047*
C17	0.0767(3)	0.0955(3)	0.71997(16)	0.0306(6)
C3	0.6490(3)	0.5137(3)	0.64882(17)	0.0333(6)
C11	0.4249(3)	0.1459(3)	0.43476(16)	0.0349(6)
H11	0.509197	0.157475	0.400047	0.042*
C25	−0.0006(3)	0.5311(3)	0.86344(17)	0.0378(7)
H25	0.013076	0.616352	0.865176	0.045*
C28	−0.0430(3)	0.2795(3)	0.85994(17)	0.0414(7)
H28	−0.056965	0.193763	0.860712	0.050*
C13	0.2263(3)	0.0582(3)	0.45769(17)	0.0397(7)
H13	0.176948	0.009949	0.437961	0.048*
C7	0.6362(3)	0.3630(3)	0.54644(17)	0.0325(6)
C14	0.1717(3)	0.1146(3)	0.53733(17)	0.0349(6)
H14	0.085347	0.105167	0.570836	0.042*
C22	−0.0700(3)	0.1067(3)	0.72112(17)	0.0393(7)
H22	−0.148540	0.197215	0.719599	0.047*
C27	−0.1152(3)	0.3679(3)	0.92664(18)	0.0446(8)
C9	0.5633(3)	0.2896(3)	0.51506(17)	0.0327(6)
H9	0.612731	0.241759	0.464511	0.039*
C2	0.6604(4)	0.6333(4)	0.7645(2)	0.0498(8)
H2A	0.698921	0.700806	0.729961	0.060*
H2B	0.746346	0.548553	0.777926	0.060*
C6	0.7849(3)	0.3484(3)	0.50412(19)	0.0418(7)
H6	0.830256	0.294362	0.455475	0.050*
C18	0.1913(3)	−0.0394(3)	0.72479(17)	0.0396(7)
H18	0.289069	−0.047430	0.726238	0.047*
C26	−0.0971(3)	0.4965(3)	0.92850(17)	0.0415(7)
H26	−0.150030	0.558086	0.973448	0.050*
C21	−0.0994(4)	−0.0165(4)	0.72453(19)	0.0514(9)
H21	−0.197897	−0.009101	0.725783	0.062*
C4	0.7939(3)	0.4944(3)	0.60712(19)	0.0405(7)
H4	0.848343	0.536289	0.627721	0.049*
C31	0.3058(5)	0.0584(4)	0.9518(3)	0.0772(12)
H31A	0.394328	−0.031728	0.949911	0.093*
H31B	0.249589	0.057986	0.909079	0.093*
C5	0.8609(3)	0.4126(3)	0.5342(2)	0.0443(8)
H5	0.958390	0.402279	0.506106	0.053*
C19	0.1621(4)	−0.1623(3)	0.72748(19)	0.0541(9)
H19	0.239839	−0.253042	0.730215	0.065*
C20	0.0167(5)	−0.1497(4)	0.7261(2)	0.0611(10)
H20	−0.002722	−0.232395	0.726165	0.073*
C1	0.5594(4)	0.7013(4)	0.8442(2)	0.0716(11)
H1A	0.472789	0.782797	0.830338	0.107*
H1B	0.614432	0.732633	0.874790	0.107*
H1C	0.525632	0.632228	0.879123	0.107*
O4	0.4719(4)	0.1022(4)	0.8062(2)	0.1100(11)
C29	0.4823(6)	0.3156(5)	0.8531(3)	0.0885(13)
C32	0.2080(6)	0.0744(5)	1.0378(3)	0.0997(15)
H32A	0.121950	0.164885	1.039380	0.150*
H32B	0.265538	0.071984	1.079774	0.150*
H32C	0.173125	−0.003231	1.049850	0.150*
C30	0.4392(5)	0.1886(5)	0.8603(3)	0.0750(12)

(C3—C4—C5—C6—C7—C8 and C17—C18—C19—C20—C21—C22) are 15.19°, 37.47°, 56.80°, respectively. The dihedral angle between the two chelating ring system (Ni—N1—O2—C7—C8—C9 and Ni—N2—O3—C16—C23—C24) is 23.53° which indicated some distortion around the metal center. The packing of crystal structure is *via* van der Waals forces.

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