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Crystal structure of 2,2'-((((ethane-1,2-diylbis(oxy))bis(2,1-phenylene))bis(azanylylidene))bis(methanylylidene))diphenolato- $\kappa^2N;\kappa^4O$)nickel(II), $C_{28}H_{22}N_2O_4Ni$

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.26 × 0.21 × 0.19 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.91 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	27.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	13577, 2556, 0.155
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1372
$N(\text{param})_{\text{refined}}$:	160
Programs:	Bruker programs [1], SHELX [2, 3], OLEX2 [4]

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Abstract

$C_{28}H_{22}N_2NiO_4$, tetragonal, $P4_12_12$ (no. 92), $a = 10.989(4)$ Å, $c = 18.470(2)$ Å, $V = 2230.4(16)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0571$, $wR_{\text{ref}}(F^2) = 0.1382$, $T = 293(2)$ 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

N,N' -bis(salicylaldehyde)-1,4-bis(o-aminophenoxy)ethane (H_2L) used in the present work was synthesized *via* a standard

Schiff base condensation reaction of salicylaldehyde (0.977 g, 8 mmol) and bis(4-aminophenoxy)ethane (0.977 g, 4 mmol) following a literature procedure [5]. All other reagents were purchased from Shanghai Aladdin Biochem (China) and used without further purification. A mixture of $Ni(ClO_4)_2 \cdot 6H_2O$ (36.6 mg, 0.1 mmol) and H_2L (45.3 mg, 0.1 mmol) in 30 mL MeOH/dichloromethane (1:2, v/v) was stirred at ambient temperature for 30 min to afford a claybank solution. The mixture was filtered and the filtrate was shelved in a sealed bottle filled with diethyl ether atmosphere, and the well-shaped reddish crystals were obtained in a yield of 42% (based on Ni).

Experimental details

The structure is also refinable in the entiomorphic chiral space group $P4_32_12$ as an inversion twin. All of the hydrogen atoms were placed in the calculated positions and all the non-hydrogen atoms were refined anisotropically.

Discussion

Over the last few decades the design and synthesis of Salen-type schiff base complexes were studied because of their fascinating structure and potential properties [6–8]. Some schiff base complexes are similar to bio-enzyme systems and suitable for the simulation of bioscience [9, 10]. Moreover, nickel is one of the essential trace elements in organisms, which can promote absorption of iron, growth of red blood cells, synthesis of aminase and may be also a kind of structural stabilizer for DNA and RNA [11].

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
N1	0.1734(5)	0.3941(5)	0.0556(3)	0.0427(14)
Ni1	0.29264(8)	0.29264(8)	0.000000	0.0461(4)
O1	0.3216(5)	0.4203(4)	−0.0736(2)	0.0560(14)
O2	0.2028(4)	0.1515(4)	0.0721(2)	0.0505(12)
C1	0.2700(7)	0.5281(6)	−0.0765(4)	0.0506(19)
C2	0.2973(8)	0.6058(7)	−0.1349(3)	0.0565(19)
H2	0.349526	0.578063	−0.170946	0.068*
C3	0.2496(7)	0.7210(8)	−0.1409(4)	0.067(2)
H3	0.270702	0.769176	−0.180320	0.081*
C4	0.1713(8)	0.7663(7)	−0.0895(4)	0.077(3)
H4	0.140628	0.844916	−0.093410	0.092*
C5	0.1389(8)	0.6928(7)	−0.0320(4)	0.067(2)
H5	0.085288	0.722987	0.002576	0.081*
C6	0.1848(6)	0.5721(6)	−0.0238(4)	0.0464(18)
C7	0.1463(6)	0.5068(6)	0.0385(4)	0.0465(18)
H7	0.096320	0.548475	0.070724	0.056*
C8	0.1248(6)	0.3401(6)	0.1190(4)	0.0460(18)
C9	0.0677(7)	0.4010(7)	0.1768(4)	0.059(2)
H9	0.062130	0.485414	0.175876	0.071*
C10	0.0201(7)	0.3382(7)	0.2345(4)	0.064(2)
H10	−0.015731	0.380775	0.272380	0.076*
C11	0.0247(6)	0.2123(8)	0.2370(4)	0.0556(18)
H11	−0.011501	0.170824	0.275296	0.067*
C12	0.0830(6)	0.1483(7)	0.1828(4)	0.051(2)
H12	0.087686	0.063857	0.184603	0.061*
C13	0.1349(6)	0.2126(7)	0.1251(3)	0.0444(16)
C14	0.1350(8)	0.0671(6)	0.0287(4)	0.061(2)
H14A	0.078194	0.022528	0.058890	0.073*
H14B	0.189958	0.009061	0.006383	0.073*

The Ni(II) center is six-coordinated by two nitrogen and four oxygen atoms from the hexadentate Salen-type L^{2−} dianion to form two six-membered and three five-membered rings and exhibits a slightly distorted-octahedral geometry. The axial positions are occupied by two imine nitrogen (N1) atoms with the Ni–N bond lengths of 2.004(5) Å, and the N1–Ni–N1 bond angle is 172.1(3)°. The equatorial plane are defined by two phenolic oxygen atoms (O1) and two ether oxygen atoms (O2) with the Ni–O distance of 1.979(5) Å and 2.270(5) Å, and the bond angles around the Ni(II) atom vary from 75.5(2)° to 163.14(19)°.

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References

1. Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2009).
2. Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr. A* **64** (2008) 112–122.
3. Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr. C* **71** (2015) 3–8.
4. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* **42** (2009) 339–341.
5. Zhang, Q. L.; Zhang, Y. Q.; Zhu, B. X.: Synthesis and crystal structure of Co(II) and Zn(II) complexes with one-dimensional chain structure. *Chinese. J. Inorg. Chem.* **27** (2011) 923–927.
6. Mahlooji, N.; Behzad, M.; Tarahhomi, A.; Maroney, M.; Ruddbari, H. A.; Bruno, G.; Ghanbari, B.: Synthesis, crystal structures and hirshfeld surface analyses of two new Salen type nickel/sodium heteronuclear complexes. *J. Mol. Struct.* **1110** (2016) 119–127.
7. Yu, L. C.; Ming, W. C.; Yuan, C. T.: New salen-type dysprosium(III) double decker and triple-decker complexes. *Polyhedron* **102** (2015) 8–15.
8. Sui, Y.; Hu, R. H.; Luo, Z. G.; Lin, W. H.; Liu, D. S.: Synthesis, structure and properties of an Erbium(III) complex with chiral salen-type schiff base ligand. *Z. Anorg. Allg. Chem.* **641** (2015) 1566–1570.
9. Zhu, B. X.; Ruan, W. J.; Gao, F.; Hu, G. H.; Zhu, Z. A.: Study on spectral characterization of water soluble chiral salen Mn(III) complexes and the recognition of amino acids. *Acta Chim. Sinica.* **62** (2004) 58–65.
10. Mohamed, G. G.; Omar, M. M.; Ibrahim, A. A.: Biological activity studies on metal complexes of novel tridentate schiff base ligand. Spectroscopic and thermal characterization. *Eur. J. Med. Chem.* **44** (2009) 4801–4812.
11. Alexious, M.; Tsivikas, I.; Dendreinou-Samara C.; Pantazaki, A. A.; Trikalitis, P.; Lalioti, N.; Kyriakidis, D. A.; Kessissoglou, D. P.: High nuclearity nickel compounds with three, four or five metal atoms showing antibacterial activity. *J. Inorg. Biochem.* **93** (2003) 256–264.