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Crystal structure of bis{5-methoxy-2-((*E*)-((4-((*E*)-1-(methoxyimino)ethyl)phenyl)imino)methyl)phenolato- $\kappa^2 N,O$ }nickel(II), $C_{34}H_{34}N_4NiO_6$

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Abstract

$C_{34}H_{34}N_4NiO_6$, monoclinic, $P2_1/c$ (no. 14), $a = 10.5538(5)$ Å, $b = 6.1691(2)$ Å, $c = 24.2831(9)$ Å, $\beta = 100.519(4)^\circ$, $Z = 2$, $V = 1554.43(11)$ Å³, $R_{gt}(F) = 0.0402$, $wR_{ref}(F^2) = 0.0973$, $T = 294.39(10)$ 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

All solvents and other reagents were of analytical grade. Synthesis of the ligand was prepared by a similar method reported earlier [1, 2]. A solution of nickel(II) acetate tetrahydrate (2.5 mg, 10 mmol) in ethanol (2 mL) was added dropwise to a solution of (*E*)-1-(4-(((*E*)-2-hydroxy-4-methoxybenzylidene)amino)phenyl) ethan-1-one *O*-methyl oxime (6.0 mg, 20 mmol) in ethanol (5 mL) at room

Table 1: Data collection and handling.

Crystal:	Clear light brown block
Size:	$0.37 \times 0.35 \times 0.34$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.68 mm^{-1}
Diffractometer, scan mode:	SuperNova, Dual, ω
$\theta_{\max}$, completeness:	26.0° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6253, 3050, 0.027
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2426
$N(\text{param})_{\text{refined}}$:	208
Programs:	CrysAlis ^{PRO} [15], SHELX [16], Olex2 [17]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.0000	0.5000	0.0000	0.03687(15)
O1	−0.03651(18)	0.4282(3)	0.07071(7)	0.0552(5)
O2	−0.21388(18)	0.6141(3)	0.23025(7)	0.0602(5)
O3	0.63789(18)	0.8244(3)	−0.15055(8)	0.0627(5)
N1	0.12132(18)	0.7277(3)	0.03505(8)	0.0399(5)
N2	0.5580(2)	0.7756(3)	−0.11151(9)	0.0539(6)
C1	−0.0449(2)	0.5664(4)	0.11072(10)	0.0412(6)
C2	−0.1232(2)	0.5105(4)	0.14964(10)	0.0455(6)
H2	−0.1655	0.3776	0.1468	0.055*
C3	−0.1372(2)	0.6520(4)	0.19175(9)	0.0467(6)
C4	−0.0723(3)	0.8508(4)	0.19770(11)	0.0561(7)
H4	−0.0820	0.9450	0.2266	0.067*
C5	0.0050(3)	0.9046(4)	0.16076(10)	0.0534(7)
H5	0.0487	1.0362	0.1650	0.064*
C6	0.0210(2)	0.7671(4)	0.11631(9)	0.0415(6)
C7	0.1074(2)	0.8291(4)	0.08024(10)	0.0427(6)
H7	0.1575	0.9520	0.0899	0.051*
C8	0.2197(2)	0.7956(4)	0.00488(9)	0.0390(5)
C9	0.2240(2)	0.9995(4)	−0.01731(11)	0.0474(6)
H9	0.1681	1.1064	−0.0091	0.057*
C10	0.3111(2)	1.0466(4)	−0.05188(11)	0.0491(7)
H10	0.3123	1.1852	−0.0668	0.059*
C11	0.3962(2)	0.8931(4)	−0.06468(9)	0.0386(5)
C12	0.3942(2)	0.6894(4)	−0.03966(11)	0.0498(7)
H12	0.4531	0.5843	−0.0459	0.060*
C13	0.3067(2)	0.6422(4)	−0.00593(11)	0.0499(7)
H13	0.3061	0.5048	0.0098	0.060*
C14	0.4851(2)	0.9348(4)	−0.10399(10)	0.0436(6)

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C15	0.4854(3)	1.1486(4)	−0.13276(14)	0.0760(10)
H15A	0.5619	1.1610	−0.1487	0.114*
H15B	0.4838	1.2631	−0.1061	0.114*
H15C	0.4108	1.1592	−0.1619	0.114*
C16	0.7082(3)	0.6349(5)	−0.15871(15)	0.0838(11)
H16A	0.7695	0.6682	−0.1822	0.126*
H16B	0.6499	0.5253	−0.1763	0.126*
H16C	0.7528	0.5829	−0.1232	0.126*
C17	−0.2838(3)	0.4156(5)	0.22694(13)	0.0749(9)
H17A	−0.3370	0.4026	0.1905	0.112*
H17B	−0.3372	0.4141	0.2550	0.112*
H17C	−0.2245	0.2964	0.2330	0.112*

temperature and stirred for 2 h. The solution was filtered and the filtrate was allowed to stand at quiet environment for about 4 weeks. Several yellow block crystals have been obtained. Elemental analysis: Anal. Calcd. for C₃₄H₃₄N₄NiO₆: C, 62.50%; H, 5.25%; N, 8.58%; Found: C, 62.45%; H, 5.29%; N, 8.53%.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

Complexes with oximes acting as ligands have been presented [2–4]. Various properties of oxime-type compounds along with their applications in analytical chemistry increased interest [5–7]. In particular, the oxime-type complexes are prominent in the chemistry. Such complexes are used as analytical reagents for the detection and determination of some metal ions [8]. Further more some complexes represent non-linear optical materials others show supramolecular architectures.

In the title complex, Ni1 is four-coordinated by two O atoms and two N atoms from two different ligands. The Ni1–N1 bond lengths are both 1.9845(18) and the Ni1–O1 bond lengths are both 1.8801(16). The angles of N1–Ni1–O1 and N1–Ni1–O1ⁱ are 90.06(7) and 89.94(7), respectively. All geometric parameters are in the typical ranges.

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References

1. Zhao, J. X.; Zhao, L.; Li, P. P.; Wang, F.; An, Q. Q.: Crystal structure of (*E*)-1-(4-(((*E*)-5-bromo-2-hydroxy-

- benzylidene)amino)phenyl)ethan-1-one *O*-methyl oxime, C₁₆H₁₅BrN₂O₂. Z. Kristallogr. NCS **232** (2017) 731–732.
2. Zhao, L.; Zhao, J. X.; An, Q. Q.; Wang, F.: Crystal structure of (*E*)-1-(4-(((*E*)-3,5-dibromo-2-hydroxy-benzylidene)amino)phenyl)ethan-1-one *O*-methyl oxime C₁₇H₁₆Br₂N₂O₂. Z. Kristallogr. NCS **231** (2016) 1053–1054.
3. Gayathri, D.; Velmurugan, D.; Ravikumar, K.; Saravanakumar, D.; Kandaswamy, M.: 4-Methyl-2-[(4-nitrophenylimino)methyl]phenol. Acta Crystallogr. **E63** (2007) o2324–o2326.
4. Zhao, J. X.; Zhao, L.; Li, P. P.; Zhu, Z. B.: Crystal structure of bis{5-methoxy-2-[(*E*)-((4-((*E*)-1-(methoxy imino)ethyl)phenyl)imino)methyl]phenolato-κ²N,O)}copper(II), C₃₄H₃₄CuN₄O₆. Z. Kristallogr. NCS **232** (2017) 863–865.
5. Li, P. P.; Zhao, L.; Zhao, J. X.; Zhu, Z. B.; Wang, F.; An, Q. Q.: Synthesis and crystal structure of bis{1-(((4-(1-(hydroxyimino)ethyl)phenyl)imino)methyl)naphthalene-2-olato-κ²O,N)}copper(II), C₃₈H₃₀CuN₄O₄. Z. Kristallogr. NCS **232** (2017) 889–890.
6. Izabela, P. M.; Wanda, R. P.; Maciej, K.; Maria, K.; Zbigniew, H.: Different supramolecular architectures in self-assembled praseodymium(III) and europium(III) complexes with rare coordination pattern of salicylaldimine ligand. Polyhedron **97** (2015) 167–174.
7. Zhao, L.; An, Q. Q.; Zhao, J. X.; Li, P. P.; Wang, F.: Crystal structure of bis(2-[(*E*)-((4-((*E*)-1-(ethoxyimino)ethyl)phenyl)imino)methyl)-5-methoxy-phenolato-κ²O,N)}copper(II), C₃₆H₃₈CuN₄O₆. Z. Kristallogr. NCS **232** (2016) 595–597.
8. Sun, Y. X.; Li, C. Y.; Yang, C. J.; Zhao, Y. Y.; Guo, J. Q.; Yu, B.: Two Cu(II) complexes with Schiff base ligands: synthesis, crystal structure, spectroscopic properties, and substituent effect. Chin. J. Inorg. Chem. **32** (2016) 327–335.
9. Sun, Y. X.; Zhao, Y. Y.; Li, C. Y.; Yu, B.; Guo, J. Q.; Li, J.: Supramolecular cobalt(II) and copper(II) complexes with Schiff base ligand: syntheses, characterizations and crystal structures. Chin. J. Inorg. Chem. **32** (2016) 913–920.
10. Dong, W. K.; Zhang, X. Y.; Sun, Y. X.; Dong, X. Y.; Li, G.; Wang, J.: A 2D supramolecular copper(II) complex with an asymmetric Salamo-type ligand: synthesis, crystal structure, and fluorescent property. Synth. React. Inorg. Met.-Org. Nano-Met. Chem. **45** (2015) 956–962.
11. Sun, Y. X.; Lu, R. E.; Li, X. R.; Zhao, Y. Y.; Li, C. Y.: A Schiff base ligand containing oxime group and its Cu(II) complex: syntheses and supramolecular structures. Chin. J. Inorg. Chem. **31** (2015) 1055–1062.
12. Wang, P.; Zhao, L.: Synthesis, structure and spectroscopic properties of the trinuclear cobalt(II) and nickel(II) complexes based on 2-hydroxynaphthaldehyde and bis(aminooxy)alkane. Spectrochim. Acta **A135** (2015) 342–350.
13. Zhao, L.; Dong, X. T.; Sun, Y. X.; Cheng, Q.; Dong, X. Y.; Wang, L.: Synthesis, crystal structure and thermal property of a 1D chain-like copper(II) complex. Chin. J. Inorg. Chem. **28** (2012) 2413–2418.
14. Sun, Y. X.; Xu, L.; Zhao, T. H.; Liu, S. H.; Liu, G. H.; Dong, X. T.: Synthesis and crystal structure of a 3D supramolecular copper(II) complex with 1-(3-phenyl)ethanone oxime. Synth. React. Inorg. Metal-Org. Nano-Met. Chem. **43** (2013) 509–513.

15. Agilent Technologies: CrysAlis^{PRO} Software system, version 1.171.37.35, Agilent Technologies UK Ltd, Oxford, UK (2011).
16. Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr. A* **64** (2008) 112–122.
17. 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.