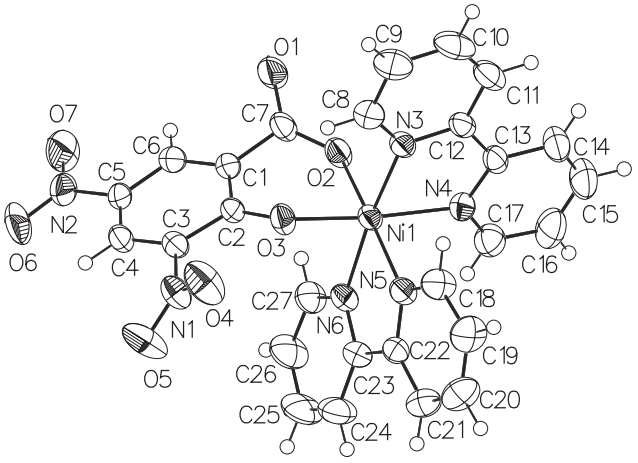
Open Access

Guixiang Xie*, Zhiwei Tang, Yan Dong and Decai Wen

Crystal structure of (bis(2,2′-bipyridine-κ^2N,N'))-(3,5-dinitrosalicylato-κ^2O,O')nickel(II), $C_{27}H_{18}N_6NiO_7$



DOI 10.1515/ncrs-2016-0270
Received September 5, 2016; accepted February 17, 2017; available online March 7, 2017

Abstract

$C_{27}H_{18}N_6NiO_7$, monoclinic, $P2_1/n$ (no. 14), $a = 8.0981(15)$ Å, $b = 21.660(3)$ Å, $c = 14.170(2)$ Å, $\beta = 95.215(4)°$, $V = 2475.1(7)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0418$, $wR_{ref}(F^2) = 0.0965$, $T = 293$ K.

CCDC no.: 1502340

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

A mixture of $Ni(OAc)_2{\cdot}2H_2O$ (0.1 mmol), 2,2′-bipyridine (0.1 mmol), 3,5-dinitrosalicylic acid (0.2 mmol) in distilled

***Corresponding author: Guixiang Xie,** College of Chemistry and Material Science, Longyan University, Longyan, Fujian, 364012, People's Republic of China, e-mail: xguix21@163.com
Zhiwei Tang, Yan Dong and Decai Wen: College of Chemistry and Material Science, Longyan University, Longyan, Fujian, 364012, People's Republic of China

Table 1: Data collection and handling.

Crystal:	Green block
Size:	0.30 × 0.20 × 0.20 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	8.5 cm^{-1}
Diffractometer, scan mode:	Weissenberg IP, ω-scans
$2\theta_{max}$, completeness:	55°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5669, 5669
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3438
$N(param)_{refined}$:	370
Programs:	SHELX [8], TeXan [9]

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

Atom	x	y	z	U_{iso}*/U_{eq}
Ni1	0.63824(5)	0.369630(15)	0.82529(2)	0.03136(10)
N1	0.4773(3)	0.32126(12)	0.51319(17)	0.0447(6)
N2	0.7725(4)	0.49713(13)	0.40091(18)	0.0518(7)
N3	0.4443(3)	0.40647(10)	0.89026(15)	0.0345(5)
N4	0.7371(3)	0.37073(10)	0.96577(15)	0.0360(5)
N5	0.5721(3)	0.27725(10)	0.84337(15)	0.0351(5)
N6	0.8285(3)	0.32325(10)	0.76747(15)	0.0358(5)
O1	0.6898(4)	0.54809(10)	0.73997(15)	0.0802(9)
O2	0.7257(3)	0.45594(9)	0.80703(12)	0.0416(5)
O3	0.5161(2)	0.37271(9)	0.69458(12)	0.0383(5)
O4	0.3583(3)	0.30630(12)	0.55192(17)	0.0703(8)
O5	0.5333(3)	0.28864(11)	0.45306(18)	0.0724(8)
O6	0.7607(4)	0.47301(12)	0.32309(15)	0.0781(9)
O7	0.8426(4)	0.54636(12)	0.41698(17)	0.0773(8)
C1	0.6580(4)	0.46127(12)	0.64086(18)	0.0357(7)
C2	0.5725(3)	0.40326(12)	0.62811(17)	0.0317(6)
C3	0.5564(4)	0.38101(12)	0.53290(18)	0.0338(6)
C4	0.6198(4)	0.41060(13)	0.45965(19)	0.0374(7)
H4A	0.6086	0.3938	0.3990	0.045*
C5	0.7002(4)	0.46530(13)	0.47659(18)	0.0377(7)
C6	0.7174(4)	0.49082(13)	0.56618(19)	0.0399(7)
H6A	0.7702	0.5287	0.5757	0.048*
C7	0.6922(4)	0.49141(13)	0.7361(2)	0.0436(8)
C8	0.3002(4)	0.42438(13)	0.8469(2)	0.0444(8)
H8A	0.2819	0.4191	0.7817	0.053*
C9	0.1765(4)	0.45034(15)	0.8943(2)	0.0557(9)
H9A	0.0764	0.4623	0.8621	0.067*
C10	0.2057(5)	0.45804(15)	0.9905(3)	0.0585(9)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H10A	0.1246	0.4755	1.0247	0.070*
C11	0.3529(4)	0.44020(14)	1.0359(2)	0.0494(8)
H11A	0.3732	0.4452	1.1011	0.059*
C12	0.4724(4)	0.41454(12)	0.98418(19)	0.0356(7)
C13	0.6366(4)	0.39508(12)	1.02631(19)	0.0369(7)
C14	0.6877(5)	0.40219(15)	1.1212(2)	0.0551(9)
H14A	0.6178	0.4199	1.1622	0.066*
C15	0.8427(6)	0.38273(18)	1.1540(2)	0.0682(11)
H15A	0.8795	0.3875	1.2177	0.082*
C16	0.9438(5)	0.35632(17)	1.0935(2)	0.0629(10)
H16A	1.0489	0.3421	1.1149	0.075*
C17	0.8849(4)	0.35148(14)	0.9996(2)	0.0484(8)
H17A	0.9531	0.3337	0.9578	0.058*
C18	0.4495(4)	0.25710(14)	0.8913(2)	0.0472(8)
H18A	0.3721	0.2855	0.9093	0.057*
C19	0.4315(5)	0.19650(15)	0.9156(2)	0.0564(9)
H19A	0.3457	0.1845	0.9509	0.068*
C20	0.5403(5)	0.15414(15)	0.8874(2)	0.0558(9)
H20A	0.5293	0.1127	0.9029	0.067*
C21	0.6657(4)	0.17290(13)	0.8362(2)	0.0463(8)
H21A	0.7398	0.1444	0.8150	0.056*
C22	0.6808(4)	0.23533(12)	0.81622(18)	0.0339(6)
C23	0.8154(4)	0.26097(13)	0.7653(2)	0.0379(7)
C24	0.9238(5)	0.22531(16)	0.7204(2)	0.0604(10)
H24A	0.9147	0.1825	0.7202	0.072*
C25	1.0465(5)	0.25398(18)	0.6756(3)	0.0723(12)
H25A	1.1199	0.2306	0.6436	0.087*
C26	1.0603(5)	0.31717(16)	0.6781(3)	0.0618(10)
H26A	1.1440	0.3371	0.6491	0.074*
C27	0.9486(4)	0.34994(15)	0.7241(2)	0.0462(8)
H27A	0.9567	0.3928	0.7252	0.055*

water (10 mL) was filled in a 20 mL Teflon-lined stainless steel reactor. The solution was heated to 453 K for 3 days, then slowly cooled to room temperature to get green block-like crystals.

Experimental details

H atoms were positioned geometrically and were included in the refinement in the riding-model approximation [C—H = 0.93 Å and $U_{iso}(H) = 1.2\,U_{eq}(C)$].

Discussion

A lot of attention has been paid to salicylate complexes because of their structural features and biological applications. Complexs with salicylato and *N*-donor ligands, were found to display diverse structure types [1–3]. A large number of hydrogen-bonded supramolecular architectures based on bipyridine and carboxylato ligands have been reported [4]. Though the functional groups such as NO_2 are able to form robust and strong hydrogen bond, the syntheses of metal-organic supramolecular assemblies employing organic ligands with NO_2 groups are limited [5]. Up to now, only 3,5-dinitrosalicylato complexes have been reported [6, 7].

In the title crystal structure the Ni(II) atom exhibits an octahedral coordination geometry, defined by four N atoms of two 2,2′-bipy ligands and two O atoms from one 3,5-$(NO_2)_2$sal ligand. The Ni-N(4) and Ni-N(6) distances are equivalent, while the Ni—N(3) bond (2.053 Å) is significantly shorter than Ni—N(3) (2.093 Å). The bipyridine rings of 2,2′-bipy containing N3 and N4 are approximately coplanar, exhibiting a dihedral angle of 2.0(1)°, while the bipyridine rings of the other 2,2′-bipy containing N5 and N6 are non-coplanar with a dihedral angle of 11.4(2)°. The 3,5-$(NO_2)_2$sal ligand is coordinated to Ni(II) atom *via* one oxygen atom of the carboxylate group (O2) and one oxygen atom from the phenolate group (O3). The carboxylate group of the 3,5-$(NO_2)_2$sal ligand is rotated relatively to the aromatic ring with a dihedral angle of 36.8(2)°. The adjacent mononuclear units are further connected to each other by C—H···O hydrogen bonds formed between the NO_2 groups of 3,5-$(NO_2)_2$sal ligands and phenyl hydrogens of 2,2′-bipy.

Acknowledgements: We thank the Foundation of Education Department of Fujian Province, PR China (No. JB12220, JB12214, JK2012048).

References

1. Khalaji, A. D.; Ghoran, S. H.; Rohlicek, J.; Dusek, M.: Characterization and crystal structure of a 17-membered macrocyclic Schiff base compound MeO-sal-pn-bn. J. Struct. Chem. **56** (2015) 259–265.
2. Vieira, B. J.; Coutinho, J. T.; Dias, J. C.; Nunes, J. C.; Santos, I. C.; Pereira, L. C.; Waerenborgh, J. C.: Crystal structure and spin crossover behavior of the [Fe(5-Cl-qsal)$_2$][Ni(dmit)$_2$]·2CH_3CN complex. Polyhedron **85** (2015) 643–651.
3. Varughese, S.; Pedireddi, V. R.: Hydrogen bond mediated open-frame networks in coordination polymers: supramolecular assemblies of Pr (III) and 3, 5-dinitro-4-methylbenzoic acid with aza-donor compounds. Chem. Commun. **14** (2005) 1824–1826.
4. Ye, B. H.; Tong, M. L.; Chen, X. M.: Metal-organic molecular architectures with 2,2′-bipyridyl-like and carboxylate ligands. Coord. Chem. Rev. **249** (2005) 545–565.
5. Pedireddi, V. R.; Varughese, S.: Solvent-dependent coordination polymers: cobalt complexes of 3,5-dinitrobenzoic acid and 3,5-dinitro-4-methylbenzoic acid with 4,4′-bipyrdine. Inorg. Chem. **43** (2004) 450–457.
6. He, X.; Bi, M. H.; Ye, K. Q.; Fang, Q. R.; Zhang, P.; Xu, J. N.; Wang, Y.: Self-assembly of a 1D helical manganese coordination polymer and a tetranuclear lanthanum complex with 1, 10-phenanthroline and 3, 5-dinitrosalicylato dianion. Inorg. Chem. Commun. **9** (2006) 1165–1168.

7. Wen, D. C.; Liu, S. X.; Ribas, J.: Syntheses, structures and magnetic property of two copper complexes with cyclic dimer and 2D herringbone-like network built from helical motif. Inorg. Chem. Commun. **10** (2007) 661–665.
8. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
9. TeXan: Texray Structural Analysis Package Molecular Structure Corp. The Woodlands, TX, (1998).